UNIFORM FEDERAL POLICY –
QUALITY ASSURANCE PROJECT PLAN

FORMER NEW YORK ORDNANCE WORKS
BALDWINSVILLE, NEW YORK
FUDS Property Number C02NY0290

Contract No: W912WJ-19-D-0001
Task Order W912WJ19F0056

December 2020

FINAL

Prepared for:

United States Army Corps of Engineers
New England District
696 Virginia Road
Concord, Massachusetts 01742

THIS PAGE WAS INTENTIONALLY LEFT BLANK

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF APPENDICES

LIST OF ACRONYMS AND ABBREVIATIONS

°F	Degrees Fahrenheit
%	Percent
%R	Percent Recovery
µg/L	microgram per liter
2,4,5-T	2,4,5-trichlorophenoxyacetic acid
AES	Atomic Emission Spectrophotometry
AGC	Army Geospatial Center
Alion	Alion Science and Technology
AOC	Area of Concern
AP	Ammonium Picrate
APP	Accident Prevention Plan
ASC	Analytical Services Coordinator
AST	Aboveground Storage Tank
AVS	Acid Volatile Sulfide
bgs	below ground surface
Bluestone	Bluestone Environmental Group, Inc.
BTAG	Biological Technical Assistance Group
BTV	Background Threshold Value
CCV	Continuing Calibration Verification
CEC	Cation Exchange Capacity
CENAE	USACE New England District
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain-of-Custody
COR	Contracting Officer's Representative
CRP	Community Relations Plan
CSM	Conceptual Site Model
CVAA	Cold Vapor Atomic Absorption
CX	Environmental and Munitions Center of Expertise
D&M	Dames & Moore
DDT	4,4'-dichlorodiphenyltrichloroethane
DERP	Defense Environmental Restoration Program
DL	Detection Limit
DNCB	Dinitrochlorobenzene
DNP	Dinitrophenol
DO	Dissolved Oxygen
DoD	Department of Defense
DoE	Department of Energy
DPT	Direct Push Technology
DQI	Data Quality Indicator

LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)

DQO	Data Quality Objective
DU	Decision Unit
DV	Data Validation
ECD	Electron Capture Detector
Eco-SSL	Ecological Soil Screening Levels
EDD	Electronic Data Deliverable
EM	Electromagnetic Induction
EPA	Environmental Protection Agency
ESD	Empire State Development
FOIL	Freedom of Information Law
ft	foot/feet
ft/day	Feet per day
ft^2	square foot/feet
FTL	Field Team Leader
FUDS	Formerly Used Defense Site
GC/MS	Gas Chromatograph/Mass Spectroscopy
GPR	Ground Penetrating Radar
GPS	Global Positioning System
GSA	General Services Administration
GW	Groundwater
HHRA	Human Health Risk Assessment
HPLC	High Performance Liquid Chromatograph
HSDB	Hazardous Substances Data Bank
HTRW	Hazardous, Toxic, and Radioactive Waste
ICAL	Initial Calibration
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectrometry
ICS	Interference Check Solutions
ICV	Initial Calibration Verification
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation Derived Waste
INPR	Inventory Project Report
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology and Regulatory Council
Koc	Carbon-Water Partition Coefficient
LCS	Laboratory Control Sample
LCS/LCSD	Laboratory Control Sample/Laboratory Control Spike Duplicate
LDI	Location Definition Information
LDR	Linear Dynamic Range
LLCCV	Low-level Calibration Check Standard

LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)

LOD	Limit of Detection
LOQ	Limit of Quantitation
M&E	Metcalf & Eddy
MC	Munitions Constituents
MCL	Maximum Contaminant Level
MEC	munitions and explosives of concern
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MMRP	Military Munitions Response Program
MPC	Measurement Performance Criteria
MRS	Munitions Response Sites
MS/ MSD	Matrix Spike /Matrix Spike Duplicate
msl	Mean sea level
NJDEP	New Jersey Department of Environmental Protection
NOAA	National Oceanic and Atmospheric Administration
NWI	National Wetland Inventory
NYOW	New York Ordnance Works
NYS	New York State
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYSUDC	New York State Urban Development Corporation
OBG	OBG Technical Services
ORP	Oxidation Reduction Potential
OSWER	Office of Solid Waste and Emergency Response
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PCB	Polychlorinated Biphenyls
PDT	Project Delivery Team
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
ppb	parts per billion
ppm	parts per million
PQLG	Project Quantitation Limit Goals
PQO	Project Quality Objective
PVC	Poly-Vinyl Chloride
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control

LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)

RC	Radisson Community
RI	Remedial Investigation
RLSO	Redline strikeout
RPD	Relative Percent Difference
RRT	Relative Retention Times
RSD	Relative Standard Deviation
RSL	Regional Screening Level
RT	Retention Time
RTC	Response to Comment
SD	Sample Duplicate
SEDD	Staged Electronic Data Deliverables
SEM	Simultaneously Extracted Metals
SI	Site Inspection
SIM	Selected Ion Monitoring
SO	Soil
SOP	Standard Operating Procedure
SRC	Syracuse Research Corporation
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOC	Semi-volatile Organic Compound
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TM	Task Manager
TOC	Total Organic Carbon
UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
US EPA	United States Environmental Protection Agency
USACHPPM	United States Army Center for Health Promotion and Preventive Medicine
USFWS	United States Fish and Wildlife Service
USGS	United States Geologic Survey
VOC	Volatile Organic Compound
WMA	Wildlife Management Area
WP	Work Plan

QAPP Crosswalk
Identifying Information

Optimized UFP-QAPP Worksheets		2106-G-05 QAPP Guidance Section	
1 & 2	Title and Approval Page	2.2.1	Title, Version, and Approval/Sign-Off
3 & 5	Project Organization and QAPP Distribution	2.2.3	Distribution List
		2.2.4	Project Organization and Schedule
4, 7 & 8	Personnel Qualifications and Sign-off Sheet	2.2.1	Title, Version, and Approval/Sign-Off
		2.2.7	Special Training Requirements and Certification
6	Communication Pathways	2.2.4	Project Organization and Schedule
9	Project Planning Session Summary	2.2.5	Project Background, Overview, and Intended Use of Data
10	Conceptual Site Model	2.2.5	Project Background, Overview, and Intended Use of Data
11	Project/Data Quality Objectives	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria
12	Measurement Performance Criteria	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria
13	Secondary Data Uses and Limitations	Chapter 3	QAPP Elements for Evaluating Existing Data
14 & 16	Project Tasks & Schedule	2.2.4	Project Organization and Schedule
15	Project Action Limits and Laboratory-Specific Detection / Quantitation Limits	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria
17	Sampling Design and Rationale	2.3.1	Sample Collection Procedure, Experimental Design, and Sampling Tasks
18	Sampling Locations and Methods	2.3.1	Sample Collection Procedure, Experimental Design, and Sampling Tasks
		2.3.2	Sampling Procedures and Requirements
19 & 30	Sample Containers, Preservation, and Hold Times	2.3.2	Sampling Procedures and Requirements
20	Field QC	2.3.5	Quality Control Requirements
21	Field SOPs	2.3.2	Sampling Procedures and Requirements
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables

QAPP Crosswalk, Continued

Optimized UFP-QAPP Worksheets		2106-G-05 QAPP Guidance Section	
23	Analytical SOPs	2.3.4	Analytical Methods Requirements and Task Description
24	Analytical Instrument Calibration	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables
26 & 27	Sample Handling, Custody, and Disposal	2.3.3	Sample Handling, Custody Procedures, and Documentation
28	Analytical Quality Control and Corrective Action	2.3.5	Quality Control Requirements
29	Project Documents and Records	2.2.8	Documentation and Records Requirements
31, 32 & 33	Assessments and Corrective Action	2.4	Assessments and Data Review
		2.5.5	Reports to Management
34	Data Verification and Validation Inputs	2.5.1	Data Verification and Validation Targets and Methods
35	Data Verification Procedures	2.5.1	Data Verification and Validation Targets and Methods
36	Data Validation Procedures	2.5.1	Data Verification and Validation Targets and Methods
37	Data Usability Assessment	2.5.2	Quantitative and Qualitative Evaluations of Usability
		2.5.3	Potential Limitations on Data Interpretation
		2.5.4	Reconciliation with Project Requirements

QAPP Worksheet #1 & 2: Title and Approval Page
(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

1. Project Identifying Information:
    a. Site/Project Name: Former New York Ordnance Works (NYOW) – Remedial Investigation (RI), Feasibility Study, Proposed Plan and Decision Document
    b. Site Location/Number: Baldwinsville, NY - CO2NY0290 [Formerly Used Defense Site (FUDS) Project Number]
    c. Contract Number: W912WJ-19-D-0001/Delivery Order Number: W912WJ19F0056

2. Lead Organization: U.S. Army Corps of Engineers, New England District (CENAE)
    a. CENAE Project Manager

    _____ _____

    Signature Date
    Erin Kirby, PG, LEP, Project Manager

    b. CENAE Quality Manager

    _____ _____

    Signature Date
    Yixian Zhang, Project Chemist

3. State Regulatory Agencies
    a. New York State Department of Environmental Conservation (NYSDEC)
    b. New York State Department of Health (NYSDOH)

4. Other Stakeholders
    U.S. Environmental Protection Agency (US EPA)
    Town of Lysander
    Village of Baldwinsville
    Radisson Community Association
    Empire State Development
    Radisson Greens Golf Course
    DiMarco Group
    Marinus Homes
    Private residential landowners on Darting Bird Lane

QAPP Worksheet #1 & 2: Title and Approval Page, Continued

5. Plans and reports from previous investigations relevant to this project:

Army Geospatial Center (AGC). 2019. NYOW – Baldwinsville, New York, Historical Photographic Analysis – Draft Report. November 2019.

Alion Science and Technology Corporation (Alion), 2008. Final Site Inspection Report for NYOW, DERP FUDS Project Number C02NY029003. Prepared for the US Army Engineering and Support Center in Huntsville and USACE Baltimore District. Contract Number W912DY-04-D-0017, Task Order #00170001.

Dames & Moore (D&M), 1981. Evaluation of Possible Hazards of Former NYOW, Radisson New Community, Town of Lysander, New York. Prepared for the Urban Development Corporation and the New York State Department of Health.

Metcalf & Eddy (M&E), 1990. Defense Environmental Restoration Program, Contamination Evaluation at the Former NYOW, Lysander, New York. Volumes I and Volume II. Prepared for the Department of the Army, Kansas City District, Corps of Engineers. Final Report. [FUDS Document No. C02NY029001_ 01.09_0002_a]

NYSDEC, 1990. Site Characterization Study, Three Rivers Facility, New York State Pesticide Storage Sites Project, Contract No. D001889. Prepared by Blasland & Bouck Engineers, P.C.

NYSDEC, 1991. Memorandum dated 14 May 1991 to New York State Urban Development Corporation regarding status of NYSDEC work at NYOW. [FUDS Document No. C02NY029003_01.01_0005_a]

NYSDEC, 1993. Operation and Maintenance Plan for the Three Rivers Pesticide Storage Site, Lysander, Onondaga County, Site No. 7-34-018.

NYSDEC, 2018. Habitat Management Plan for Three Rivers Wildlife Management Area 2018-2027. Prepared by the Division of Fish and Wildlife, Bureau of Wildlife, March 2018. https://www.dec.ny.gov/docs/wildlife_pdf/yfithreeriversshmp.pdf

Radisson, 2017. Factsheet: Empire State Development: Radisson Community Project. https://esd.ny.gov/radisson-community-project.

Reconstruction Finance Corporation, undated. Chemical Manufacturing Plant (Ammonium Picrate), Baldwinsville, New York, Known as NYOW.

United States Army Corps of Engineers (USACE), 1981. Memorandum for Commander, Kansas City District, Subject: Report on Records Search of NYOW. 13 March 1981. [FUDS Document No. C02NY029001_01.06_0006_p]

USACE, 1985. Determination and Findings of Fact, NYOW, Lysander, New York, Project No. C02NY002900.

USACE, 1991. Memorandum for Commander, North Atlantic Division, Subject: DERP-FUDS Inventory Project Report for Site No. C02NY029000, NYOW, Lysander, NY. Prepared by the Department of

QAPP Worksheet #1 & 2: Title and Approval Page, Continued

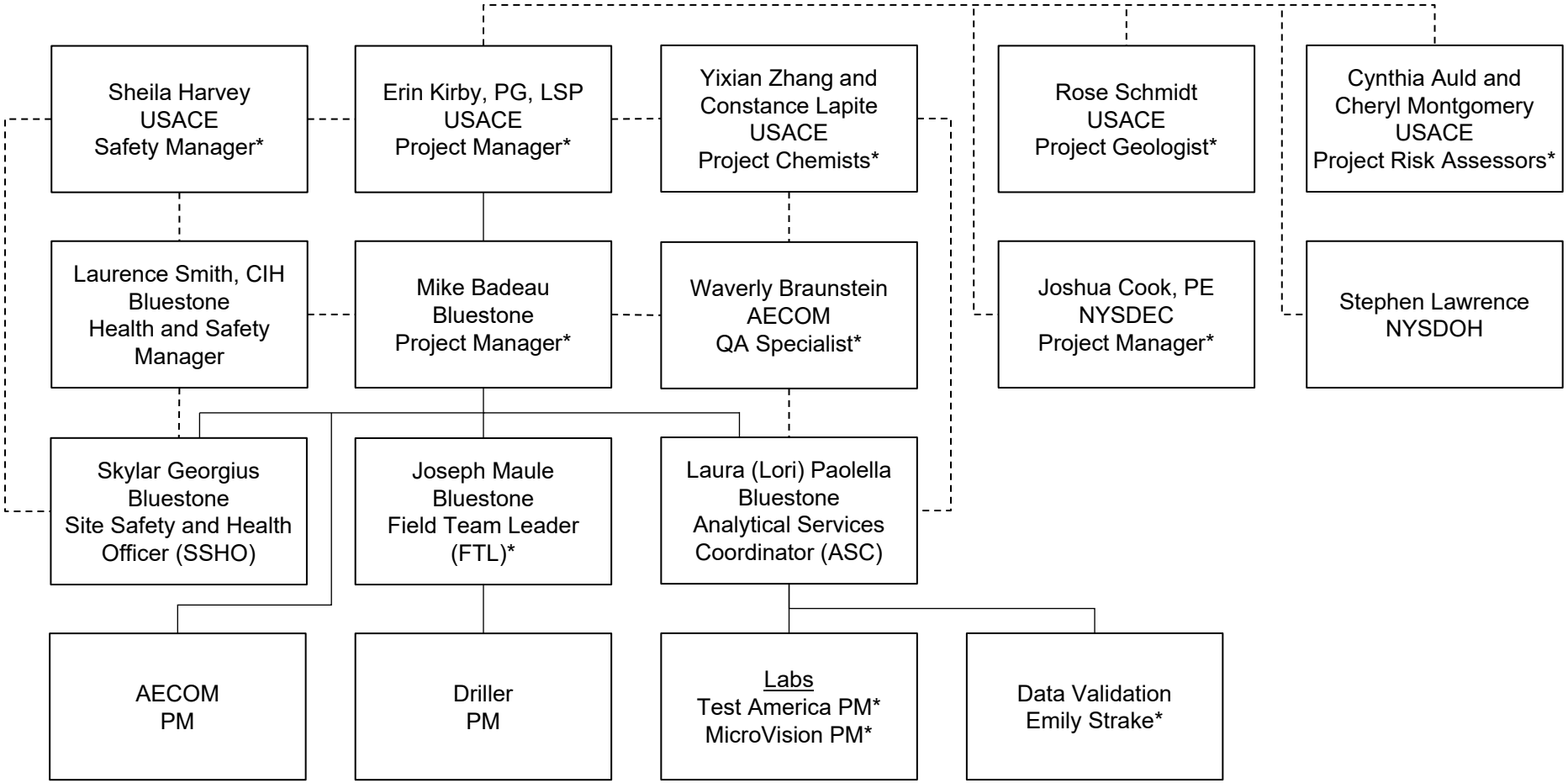
the Army, New York District, Corps of Engineers, New York, NY. 18 October 1991. [Enclosure to FUDS Document No. C02NY029002_01.08_0009_a]

USACE, 1999a. Archives Search Report, Findings for the former NYOW, Lysander Township, New York, Project Number C02NY029003. Prepared by USACE Rock Island District, September 1999. [FUDS Document No. C02NY029001_01.02_0001_a]

USACE, 2006. Fact Sheet on NYOW, Lysander, NY. Prepared by USACE New York District. [FUDS Hazardous, Toxic, and Radioactive Waste (HTRW) Document No. C02NY029001_08.11_0500_p]

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution
(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)

*QAPP recipient Lines of authority ——— Lines of Communication --------



QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-Off Sheet
(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)

ORGANIZATION: Bluestone Environmental Group, Inc.

Name	Project Title/Role	Education/Experience	Specialized Training/ Certifications	Signature/Date
Michael Badeau, PE	Project Manager (PM)	B.S. Environmental Systems Engineering/ 15 Years	OSHA 40-hr HAZWOPER and 8-hr refresher, Licensed Professional Engineer (PE) , First Aid/CPR	
Christen Sardano, LSP	Quality Control Manager	B.S., Geology and Anthropology/ 26 Years	OSHA 40-hr HAZWOPER and 8-hr refresher, MA Licensed Site Professional	
Aaron Myers, PMP	Task Manager (TM)	B.A. Environmental Science/ 10 Years	OSHA 40-hr HAZWOPER and 8-hr refresher, Site Safety & Health Officer (SSHO), First Aid/CPR	
Skylar Georgius	Site Safety and Health Officer (SSHO)	B.A. Geology/ 5 Years	OSHA 40-hr HAZWOPER and 8-hr refresher, SSHO, First Aid/CPR	
Emily Strake	Human Health Risk Assessor	MBA, B.S. Chemistry/ 20 Years	OSHA 40-hr HAZWOPER and 8-hr refresher Board Certified Environmental Professional (CEP) in Assessment	

QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-Off Sheet, Continued

Name	Project Title/Role	Education/Experience	Specialized Training/ Certifications	Signature/Date
Joseph Maule, PG	Project Geologist and Field Team Lead (FTL)	M.S. Geology, B.S. Geology/ 20 Years	Licensed Professional Geologist (PG), OSHA 40-hr HAZWOPER and 8-hr refresher	
TBD	Field Sampler	TBD	OSHA 40-hr HAZWOPER and 8-hr refresher	
Anne MacMillan, REP, PMP	Staff Scientist/Engineer	M.S. Environmental Engineering Science, B.S. Land Surface Process and Environmental Change/ 10 Years	OSHA 40-hr HAZWOPER and 8-hr refresher, First Aid/CPR	
Laurence Smith, CIH	Program Health and Safety Manager	M.S. Environmental Health/ 30 Years	Certified Industrial Hygienist (CIH), Certified Safety Professional (CSP)	
Laura (Lori) Paolella, PhD	Project Chemist and Analytical Services Coordinator (ASC)	PhD Chemistry, B.A. Chemistry and Biology/ 11 Years		
Emily Strake, CEP	Data Validator	MBA, B.S. Chemistry/ 20 Years	OSHA 40-hr HAZWOPER and 8-hr refresher Board Certified Environmental Professional (CEP) in Assessment	
Florence Sevold	Database Manager and Ecological Risk Assessor	B.S. Toxicology/ 30 Years		

QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-Off Sheet, Continued

ORGANIZATION: AECOM

Name	Project Title/Role	Education/Experience	Specialized Training/ Certifications	Signature/Date
Waverly Braunstein	Quality Assurance (QA) Specialist	B.A. Chemistry/ 35 Years		

ORGANIZATION: Test America Denver, Seattle, Savanah, and Corpus Christi

Name	Project Title/Role	Education/Experience	Specialized Training/ Certifications	Signature/Date
Patrick McEntee	Project Manager	B.A. Geology/ 31 Years		
Roxanne Sullivan	QA Manager	B.S. Chemistry/ 35 Years		

ORGANIZATION: MicroVision Labs, Inc.

Name	Project Title/Role	Education/Experience	Specialized Training/ Certifications	Signature/Date
John Knowles	President	B.S. Animal Science/ 34 Years	Northeastern University – Scanning Electron Microscopy University of Massachusetts – Material Science Carl Zeiss Microscopy SEM Course	

QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-Off Sheet, Continued

Name	Project Title/Role	Education/Experience	Specialized Training/ Certifications	Signature/Date
			McCrone Research Institute – Applied Polarized Light Microscopy, Advanced Asbestos Identification	

QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

Communication Driver	Organization and Project Role	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
Regulatory Agency Interface	USACE Project Manager (PM)	Erin Kirby, PG	978-318-8147	The USACE PM will be the direct liaison with the regulatory agencies.
Manage Field Tasks	Bluestone Task Manager	Aaron Myers, PMP	610-457-1354	TM will be the liaison to the FTL concerning investigation activities. TM will daily communicate with the project team and FTL. TM will communicate implementation issues to FTL.
QAPP Changes: • In the field • Prior to field work • During project execution	Bluestone FTL	Joseph Maule, PG	610-952-3637	FTL will notify the PM immediately and will promptly complete a Field Change Notification (FCN) form and/or corrected worksheets(s).
	Bluestone PM	Michael Badeau, PE	610-306-2966	The Bluestone PM will notify USACE PM and ASC of delays or changes to field work. Bluestone will prepare QAPP Addenda or revisions in consultation with USACE.
Field Corrective Actions	USACE PM	Erin Kirby, PG	978-318-8147	The PM determines the need for corrective actions. Corrective actions may also be identified by the field team.
	Bluestone TM	Aaron Myers, PMP	610-457-1354	The PM determines the need for corrective actions. Corrective actions may also be identified and initiated by the field team.
	Quality Assurance Coordinator (QAC), auditor, TM, FTL, and Field Team	Joseph Maule, PG	610-952-3637	PM, TM, FTL, and, per QA manual requirement, field team may identify corrective actions. FTL will initiate corrective action(s) on identified field issue(s) immediately or within the recommended timeframe. FTL will oversee implementation of corrective action(s) and notify auditor, Bluestone PM and TM by email. FTL will complete the corrective action report form.
Field Progress Reports	Bluestone FTL	Joseph Maule, PG	610-952-3637	FTL will complete on a daily basis and submit to Bluestone PM and FTL. PM or TM will forward to USACE upon request.

QAPP Worksheet #6: Communication Pathways, Continued

Communication Driver	Organization and Project Role	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
Scheduling of Analytical Services	Bluestone FTL	Joseph Maule, PG	610-952-3637	FTL will submit request to ASC before the timeframe below.
	Bluestone ASC	Laura (Lori) Paolella, PhD	484-348-4720	ASC will coordinate with laboratories 3 weeks prior to sampling.
Facilitate Database Setup and Data Management Planning	Bluestone FTL	Joseph Maule, PG	610-952-3637	FTL will provide sample and analytical information prior to sample collection including information on sample and analytical reporting groups, and types of report tables required for project.
Facilitate Data Management	Bluestone FTL	Joseph Maule, PG	610-952-3637	FTL will provide electronic survey data, sample ID, locations and analyses. FTL will transmit completed sample tracking information to data manager by the completion of each sampling case.
Incomplete Electronic Data Deliverables (EDDs) or Other EDD Issues	Bluestone Data Manager, FTL, and Data Coordinator	Florence Sevold	484-341-7380	Personnel will request resubmittal of corrected EDD by email.
Data Verification Issues, e.g., incomplete records	Bluestone FTL or Sample Manager, and Data Coordinator	Joseph Maule, PG	610-952-3637	Data Coordinator will send an email to the FTL when an issue is found. FTL will address questions or any discrepancies.
Procurement of Analytical Services	Bluestone FTL, TM, ASC	Laura (Lori) Paolella, PhD	484-348-4720	FTL or TM will prepare a laboratory request. ASC will prepare an analytical SOW and submit for project chemist review. FTL will initiate laboratory kick-off call with subcontract laboratories and email agenda.
Analytical Services Support	Bluestone ASC	Laura (Lori) Paolella, PhD	484-348-4720	ASC will act as liaison for laboratories and with subcontract laboratory(s).
Laboratory Quality Control Variances and Analytical Corrective Actions	Bluestone Laboratory QC Officer	Laura (Lori) Paolella, PhD	484-348-4720	Lab QC Officer will communicate daily with the laboratory staff and regularly with the QAC/FTL or designee. Provide oversight and direction on technical issues as needed.

QAPP Worksheet #6: Communication Pathways, Continued

Communication Driver	Organization and Project Role	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
Notification of Analytical Issues and Sample Receipt Variances	Bluestone ASC	Laura (Lori) Paolella, PhD	484-348-4720	ASC will notify FTL of any sample collection/shipment issues. Notify laboratory or subcontract laboratories to initiate corrective action.
Data Validation Issues, e.g., non-compliance with procedures; data review corrective actions	Validator	Emily Strake, CEP	484-341-0380	Validator will contact Bluestone project chemist with any laboratory non-compliance issues discovered during validation. Chemist will notify data assessor
	QA Specialist	Waverly Braunstein	978-502-5363	QA Specialist will submit a list of questions or issues to USACE or the subcontract laboratory as appropriate for correction or other appropriate response.
Reporting of Issues Relating to Analytical Data Quality (including ability to meet reporting limits, and usability of data)	ASC	Laura (Lori) Paolella, PhD	484-348-4720	ASC will communicate to PM as appropriate.
	QA Specialist	Waverly Braunstein	978-502-5363	QA Specialist will communicate to PM as appropriate. Document situation and effect in a data quality report prepared prior to evaluation of remedial design report.
Release of Analytical Data	ASC	Laura (Lori) Paolella, PhD	484-348-4720	ASC will receive and review data packages before data is used; and initiate data validation (DV) of subcontract laboratory data.
Site Health and Safety Issues Stop Work Due to Safety Issues	SSHO	Skylar Georgius	301-366-9519	The SSHO will conduct Daily Health and Safety Meetings, make decisions regarding health and safety issues and upgrading personal protective equipment. SSHO will communicate to PM, TM, Health and Safety Manager, and field staff as appropriate, per the Accident Prevention Plan (APP) and Site Safety & Health Plan (SSHP).

QAPP Worksheet #6: Communication Pathways, Continued

Communication Driver	Organization and Project Role	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
Regulatory Involvement	NYSDEC	Joshua Cook, PE	315-426-7411	Status updates, during site visits
	NYSDOH	Stephen Lawrence	518-402-7860	

QAPP Worksheet #9a: Project Planning Session Summary – 13 August 2019
(UFP-QAPP Manual Section 2.5.1 and Figures 9-12)
(EPA 2106-G-05 Section 2.2.5)

Date of Planning Session: 13 August 2019
Location: Syracuse, NY
Scoping Session Purpose: Coordination and Kick-Off Meeting

Participants:

Name	Organization	Title/ Role	E-mail	Phone
Erin Kirby, PG	USACE	Project Manager	Erin.Kirby@usace.army.mil	978-318-8147
Cheryl Montgomery	USACE	Ecological Risk Assessor	Cheryl.R.Montgomery@usace.army.mil	978-318-8644
Rose Schmidt, PG	USACE	Geologist	Rosemary.A.Schmidt@usace.army.mil	978-318-8345
Cindy Auld	USACE	Human Health Risk Assessor	Cynthia.A.Auld@usace.army.mil	978-318-8042
Yixian Zhang	USACE	Chemist	Yixian.Zhang@usace.army.mil	978-318-8730
Simone Shields	Bluestone	Project Manager	sshields@bluestoneenviro.com	215-817-5143
Anne MacMillan, REP, PMP	Bluestone	Assistant Project Manager	annemacmillan@bluestoneenviro.com	610-999-4593
Skylar Georgius	Bluestone	FTL	sgeorgius@bluestoneenviro.com	301-366-9519
Audra Balson, PG	Bluestone	Geologist	abalson@bluestoneenviro.com	717.940.8808
Cindy Woods	Bluestone	Human Health Risk Assessor	cwoods@bluestoneenviro.com	978-290-6169
Tod DeLong	Bluestone	Ecological Risk Assessor	tdelong@bluestoneenviro.com	484-341-7398
Florence Sevold	Bluestone	FUDSChem Point of Contact (POC)	fsevold@bluestoneenviro.com	484-341-7380
Lee dePersia, PE	Bluestone	Program Manager	ldepersia@bluestoneenviro.com	610-647-9500
Laura Paolella, PhD	Bluestone	Chemist	lpaolella@bluestoneenviro.com	484-348-4720
Eleanor Vivaudou, PE	AECOM	PE and NY QEP	Eleanor.Vivaudou@aecom.com	978-905-2324
Joshua Millard, PG	AECOM	Geologist	Joshua.Millard@aecom.com	978-905-2324
Constance Lapite	AECOM	Sr. Chemist	Constance.Lapite@aecom.com	978-905-3131

QAPP Worksheet #9a: Project Planning Session Summary – 13 August 2019, Continued

Notes/Comments: Complete meeting minutes were submitted to USACE on 20 August 2019, with Final Meeting Minutes being submitted to USACE on 29 August 2019. Topics covered included: Introductions, Project Communication, Invoice, and Scope of Work.

Consensus Decisions and Action Items:

Action	Responsible Party	Due Date
Ms. Kirby recommended assessing what changes are needed, present to Project Delivery Team (PDT) and she will work on making the contract changes.	Bluestone and USACE	Ongoing/no changes needed as of 13 Sep 2019
Ms. Shields will create and send out Outlook invites to the project team for deadlines related to review of deliverables. A rolling two-month period will be kept updated for future project deadlines and deliverables	Bluestone	Ongoing/up-to-date
Bluestone will update the ecological risk and human health risk assessment (HHRA) flow charts based on discussion at the meeting and Site visit the following day.	Bluestone	To be completed with Client Draft RI Work Plan (WP) due 13 Sep 2019 Note: The Work Plan was subsequently incorporated into the QAPP. A separate Work Plan document will not be produced.
Photos from the Site visit will be sent to the CENAE team, for those not able to attend the Site visit.	Bluestone	In progress
CENAE requested that: • Representative receptors have breakout categories • Change Dermal to Direct contact • Dr. Cheryl Montgomery and Mr. Tod DeLong to verify exposure pathways	Bluestone Eco Risk Assessor	To be completed with Client Draft RI WP due 13 Sep 2019
Bluestone will clarify in RI WP that abbreviated work Site hours may be required near the residences.	Bluestone	To be completed with Client Draft RI WP due 13 Sep 2019
Bluestone will address concerns related to picric acid in the Accident Prevention Plan (APP).	Bluestone	To be completed with Draft APP/SSHP due 25 Sep 2019

QAPP Worksheet #9a: Project Planning Session Summary – 13 August 2019, Continued

Action	Responsible Party	Due Date
• CENAE will follow up with potential subject matter experts for further information on picric acid, as well. • CENAE to assess if areas outside of the Areas of Concern (AOCs) are covered under the rights of entry for background sampling.		

QAPP Worksheet #9b: Project Planning Session Summary – 23 August 2019

RI WP Changes Meeting
23 August 2019; 9:00am – 10:00am

Attendees:

US Army Corps of Engineers, New England District (CENAE), Project Delivery Team (PDT):

Ms. Erin Kirby – Engineering Tech Lead (ETL)/Contracting Officer's Representative (COR)
Dr. Cheryl Montgomery – Ecological Risk Assessor (CEERD-EL)
Ms. Ellen (Michelle) Bourne (CEERD-EL)
Ms. Rose Schmidt – Geologist
Ms. Yixian Zhang – Chemist

Bluestone Environmental Group, Inc. (Bluestone):
Ms. Simone Shields – Project Manager
Ms. Anne MacMillan – Assistant Project Manager
Ms. Skylar Georgius – Field Team Leader
Ms. Cindy Woods – Human Health Risk Assessor
Mr. Fred Tenbus – Geologist/Technical Lead
Ms. Florence Sevold – FUDSChem Point of Contact (POC)
Ms. Laura (Lori) Paolella – Project Chemist

AECOM – Subcontractor:

Ms. Constance Lapite – Sr. Chemist

1. **Acid Tanks and FUDS Eligibility**
    a. **Site Wide**
    - There are two acid tanks at AOC 2 and three acid tanks at AOC 1
    - Ms. Erin Kirby summarized her previous correspondence with Mr. Josh Cook of NYSDEC.
        - Since the parcels comprising the Site were transferred to private entities prior to NYSDEC ownership, the remaining structures are not eligible for FUDS Building Demolition/Debris Removal (BD/DR). Helpful text for the sequence of events, references, and explanation of FUDS eligibility is as follows:
            - Per the Inventory Project Report (INPR) (dated October 1991, on 23 January 1946, NYOW was transferred to the custody of the Reconstruction Finance Corporation, a predecessor to the General Service Administration (GSA). Subsequently, on 05 December 1947, the GSA conveyed the title for the 2,300-acres of the Site that is the former Ammonium Picrate Area and the current Radisson Community residential area to the Fosham Corporation of Baldwinsville, New York. On 10 July 1952, the Fosham Corporation conveyed the title to the First National City Bank. On 22 October 1968, the First National City Bank conveyed the title to the 1001 East Genese Corporation of Syracuse New York. On 09 June 1969, the 1001 East Genese Corporation sold the 2,300-acres representing the former Ammonium Picrate Area to the New York State Urban Development Corporation (NYSUDC).

QAPP Worksheet #9b: Project Planning Session Summary – 23 August 2019

The NYSDC has developed the former Ammonium Picrate Area into the residential/light industrial community now known as the Radisson Community.

- The INPR (dated 1991) explicitly states that most of the Department of Defense (DoD) structures have been demolished, but "some of them were probably deemed too expensive and complex to remove". These structures include the water tower, six bunkers, acid plant, five acid tanks, pump house, and assorted foundations. The INPR further states that the abandoned structures do present a safety hazard; however, the hazard appears to be the result of the absence of maintenance over an extended period of time. Since this Site was privately owned subsequent to DoD usage, namely Fosham Corporation, First National City Bank, and the 1001 East Genese Corporation, and the title transfer document does not obligate the Government to conduct site restoration, the above mentioned structures are not eligible for demolition and removal under the Defense Environmental Restoration Program (DERP). Additionally, in accordance with FUDS ER 200-3-1, projects where a hazard is a result of neglect by an owner/grantee subsequent to DoD use, regardless of whether the deed or disposal document required the owner/grantee to maintain the property improvements in accordance with FUDS ER-200-3-1 are, by definition, ineligible under FUDS.

- Also, the five concrete aboveground acid tanks located at AOCs 1 and 2 were remediated by NYSDEC in 1991; therefore, the acid tanks not FUDS-eligible. Helpful text for the sequence of events, references, and explanation of FUDS eligibility is as follows:
  - In accordance with the FUDS ER-200-3-1, Section 3-1.5.3.10, properties where environmental restoration activities have already initiated are ineligible under the FUDS program. In a letter dated 19 January 1990 to the USACE, M&E concluded that the tanks posed an imminent hazard to the public. In February 1991, NYSDEC notified the USACE of the need for an emergency response action (Gerrard, 1993; NYDEC, 1991). NYSDEC concluded that they had the ability to conduct the clean-up more speedily than the USACE, they undertook the emergency response action (Gerrard, 1993). The emergency response included removal and disposal of approximately 15-20 tons of debris per tank and corrosive water, i.e., pH of less than 2, in the tanks (Gerrard, 1993; NYDEC, 1991). NYSDEC neutralized approximately 78,000-gallons of water and disposed of the water at a local publicly owned treatment works (Gerrard, 1993; NYDEC, 1991). NYSDEC then placed six includes (28 cubic yards) of limestone at the bottom of each tank and installed drainage holes in the tank sides (Gerrard, 1993; NYDEC, 1991). Consequently, when NYSDEC removed the five acid tanks rendering them ineligible under FUDS.
  - Currently the acid tanks are no longer present at AOCs 1 and 2; therefore, NYSDEC, NYSUDC, or another entity must have removed the five concrete tank structures although there are no records in the administrative record to document the removal and disposal of the five acid tanks.
- Subsequently residual impacts from the acid tanks are also not FUDS-eligible.

QAPP Worksheet #9b: Project Planning Session Summary – 23 August 2019

b. AOC 2

- AOC had two acid tanks
- Dr. Cheryl Montgomery and Ms. Rose Schmidt asked if any of the other structures at AOC 2 are still present. It was discussed and determined that the other structures are no longer present either.
- Since the two acid tanks at AOC 2 have been remediated by NYSDEC and the historic DoD improvements associated with this AOC have been removed, AOC 2 is not FUDS-eligible in accordance with FUDS ER-200-3-1.

c. AOC 1

- AOC 1 had three acid tanks. The tanks were numbered on the figures from the WP (Figure 2-3), but will be more clearly labeled for clarity moving forward including referring to them as "former".
- Ms. Cindy Woods described the path that the group took to get to the ramp structures in relation to where the tanks would be. Discussion of what the tanks looked like and if anyone saw the tanks during the site walk was discussed while the PDT looked at pictures taken during the site visit. It was determined that no one remembers seeing the tanks. As well, the INPR, dated August 7, 1991 (pages 2-3) states that NYSDEC removed the tanks.
- CENAE PDT recommended that the figure be revised to show the historical features more clearly, change to 11x17, and include an inset table to describe the numbering of the historical features.
- Ms. Erin Kirby confirmed that the currently analytical plan for AOC 1 will not change based on the acid tank discussion. The data will show if NYSDEC remediated completely or if there was any mobilization of metals from the acid tanks. Once AOC 1 data is received, revisions can be made to the path forward.
- Text will also be added to AOC 1 to explain discussion and removal of acid tanks from project.

2. Geology

- Rose Schmidt stated that based on the glacial till of the area, derived from the underlying sedimentary limestone bedrock, it would be high in calcium carbonate and would likely provide a buffer to any acids.
- Ms. Rose Schmidt also asked for clarification on experience with glacial geology of the Bluestone team.
  - Mr. Fred Tenbus (Technical Lead) and Ms. Skylar Georgius (Field Team Leader) do not have large amounts of glacial geology experience.
  - Ms. Audra Balson (Sr. Geologist) and Mr. Josh Millard (AECOM Sr. Geologist) do have glacial geology experience.
- Ms. Rose Schmidt stated her concern about glacial geology experience while going through the WP documents.
  - Specifically, Section 2.5.3 – Physical settings section at the beginning of the report.
  - It was identified that this section was written under the last contract and was likely written by Ms. Kim McGeehan (Bluestone)

QAPP Worksheet #9b: Project Planning Session Summary – 23 August 2019

  - Ms. Rose Schmidt was concerned that geology reviewer did not catch the incorrect statements about drumlin and how deposition occurred. She recommended that the text be reviewed by someone with more glacial geology background.
- Ms. Rose Schmidt also stated that the description of groundwater in the background section was not ideal and recommended revision.

3. RTCs to Environmental and Munitions Mandatory Center of Expertise (CX) comments:

- Ms. Erin Kirby asked if any of Bluestone's response to comments (RTCs) deviated from CENAE PDT's original RTCs.
  - Ms. Cindy Woods mentioned that some of the HH RTCs had deviated because of the change in HHRA approach from Ms. Cindy Auld
  - Ms. Skylar Georgius stated that most of the RTCs explained further what CENAE PDT had stated, ex: how many Incremental Sampling Methodology (ISM) samples would be needed.
- Ms. Cindy Woods will follow up with Ms. Cindy Auld and Ms. Erin Kirby regarding the CX RTCs in the next few days.
- Bluestone will provide the revised RI WP and QAPP in both .pdf and a redline strikeout (RLSO) version in MS Word.

Action Items:

- Bluestone to prepare an RLSO Word version of the RI WP and QAPP with acid tank revisions and removal of AOC 2. Bluestone will also prepare a clean PDF copy of the RI WP and QAPP. Bluestone will also send word files to show the RLSO and new information. Both the Word version and the PDF will have an estimated submittal date of 30 September 2019.
- Ms. Cindy Auld and Ms. Cindy Woods will discuss CX RTCs and resolve any issues. [UPDATE 30Sep19]: Ms. Cindy Auld and Ms. Cindy Woods held a conference call on 24 Sep 2019. Revised RTCs relative to the HH Approach will be submitted on 30 Sep 2019 along with the RI WP and QAPP RLSO versions.
- Geology comments will be addressed in next round of the RI WP, QAPP and RTCs.
- Community Relations Plan (CRP) due date will be finalized after discussions over the next few days regarding HH and geology. [UPDATE 30Sep19]: The final CRP was submitted as scheduled on 27 Sep 2019.
- Ms. Simone Shields will send Ms. Erin Kirby geologist resumes, as requested. [UPDATE 30Sep19]: Resumes were sent to Ms. Erin Kirby on 24 Sep 2019.

QAPP Worksheet #9c: Project Planning Session Summary – 09 December 2019

On 09 December 2019, during a meeting between CENAE (Ms. Erin Kirby) and Bluestone (Ms. Simone Shields), it was determined that AOC 5 Former Boiler Plant Area was to be added to the QAPP. This was based on information presented in the AGC NYOW Historical Photographic Analysis Draft Report, dated November 2019 and approval by the USACE FUDS Program Manager.

QAPP Worksheet #10: Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

As defined in Engineer Manual 200-1-12 - Environmental Quality, Conceptual Site Models (USACE, 2012), a Conceptual Site Model (CSM) describes sources of possible DoD-impacts, as well as complete, potentially complete, or incomplete human and ecological exposure pathways; current, determined, or reasonably anticipated future use of property; and, human and ecological potential receptors (USACE, 2012). A CSM is an iterative planning and communication tool that provides a structure to summarize and display information and to identify additional information needed to develop technically sound decisions.

10.1 DESCRIPTION AND CURRENT USE

As shown on **Figure 10-1**, the former NYOW is located in Onondaga County, New York, near the Finger Lakes, about one-mile northeast of the Village of Baldwinsville and approximately 13 miles north of Syracuse, New York. Portions of the Site are currently owned by the Radisson Community, the State of New York, and private owners. Part of the first phase of this study will be determining and verifying current and potential future land use. Future use is generally expected to remain the same as current use.

10.1.1 Site History

Prior to construction of the NYOW facility, the Site was primarily agricultural and forested (Army Geospatial Center [AGC], 2019). In 1942, the Site was acquired from private owners in various parcels to establish a facility for the manufacture of ammonium picrate during World War II. The facility was designed and built by Lummus Company and included 146 structures. Construction began on 06 April 1942 and was completed in May 1943. The Site was operated by National Aniline Defense Corporation, a Division of Allied Chemical and Dye Corporation under the supervision of the Chief of Ordnance of the War Department (USACE, 1985).

Operations began on 22 January 1942 and were ceased on 15 March 1944 (USACE, 1981). The facility was designed to produce 60,000 pounds of ammonium picrate per day, seven days a week. Ammonium picrate is a stable, high explosive compound used in the production of armor piercing shells. The plant included an administration area, ammonium picrate area, acid area, and magazine storage/bunker area (**Figure 10-2**) (Reconstruction Finance Corporation, undated). Historical Site features are shown on **Figures 10-3 through 10-6**. The facility encompassed approximately 2,100 acres of the 6,795-acre property; the remainder was farmland. The property was declared surplus on 30 August 1945 (USACE, 1981).

On 23 January 1946, the 6,795-acre property was transferred to the custody of the Reconstruction Finance Corporation (USACE, 1991). Reconstruction Finance Corporation was a predecessor to the General Service Administration (GSA). Subsequently, 4,495 acres in the northern and western parts of the Site were classified as farmland and assigned to the Farm Credit Administration for disposal. By 1947, all tracts of land designated as farmland had been sold, including 2,600 acres to the NYSDEC for use as the Three Rivers State Wildlife Management Area (WMA) and 1,895 acres subdivided among 98 individual private owners (USACE, 2006) (**Figure 10-7**).

The remaining 2,300 acres were classified as industrial property. In 1948 and 1950, 500 acres of the industrial property were sold to Fosham Corporation, a merger of a rail company (L.B. Foster) and Hamilton Corporation of Cincinnati. A later purchase by Fosham (approximately 1,600 acres) was

QAPP Worksheet #10: Conceptual Site Model, Continued

identified in the deed as contaminated; the deed required the purchaser to decontaminate the property and to provide proof of decontamination to the government. Fosham Corporation notified the GSA in October 1950 by letter that "all decontamination work necessary under the terms of this agreement has been performed by this company and, in particular, the drying houses and the lower portion of the ramps in each A.P. line have been burned." A letter dated 14 December 1950 from the New York Regional Office of GSA noted that the premises were examined by a representative of the Ordnance Ammunition Center, US Army Joliet, Illinois on 24 November 1950 and "no evidence of contamination was found" (USACE, 1981).

On 10 July 1952, the Fosham Corporation conveyed the title to the First National City Bank. On 22 October 1968, the First National City Bank conveyed the title to the 1001 East Genesee Corporation of Syracuse New York. On 09 June 1969, the 1001 East Genesee Corporation sold the 2,300-acres representing the former Ammonium Picrate Area to the NYSUDC; this area is now known as the Radisson Community (**Figure 10-7**). The source of the "Parcel Land Use" shown on **Figure 10-7** is the Onondaga County Parcel File (http://www.fsihost.com/onondaga/metadata/Parcels.pdf), last updated in 2019. Land Use is defined as "Property's land use description." There is no recreational use category; therefore, golf courses and recreational use land are shown as commercial land use.

10.1.2 Overview of Areas of Concern

AOC 1 – Ammonium Picrate Area

The Former Ammonium Picrate Area is located approximately one mile north of the Former Administration Area, which was located on Route 31 at Chestnut Ridge Road (**Figure 10-3**). The current land use for this area is housing and undeveloped land. Homes are not built on areas of potential impacts.

This area was used for the manufacture of ammonium picrate and included six 13,500 square foot (ft^2) manufacturing buildings, six 10,676 ft^2 dryer houses, three aboveground 12 foot (ft) diameter by 12 ft high brick liquid storage tanks, and a 4,612 ft^2 laboratory building (Reconstruction Finance Corporation, undated). Ammonium picrate is produced by mixing picric acid and ammonia (Alion, 2008). Picric acid is formed from dinitrochlorobenzene (DNCB) (substituted for phenol), sulfuric acid, and nitric acid (Olsen and Goldstein, 1924).

Air photo analysis (AGC, 2019) also shows an open storage area located to the northeast of the main production area, and a borrow area to the west (**Figure 10-3**). Air photos also show later backfilling, post-DoD use, of the former borrow pit.

AOC 3 – Former Landfill Area

The Former Landfill Area is located under the current Radisson Golf Course club house parking lot (**Figure 10-4**). This area was used for the disposal of household and office trash, paint cans, stainless-steel "spiders" (e.g., equipment used to mix the ammonium picrate), and other potential manufacturing items (D&M, 1981). Based on historical documentation there were two distinct phases of dumping at the landfill in 1944 during the operation of the DoD and then in 1949 after the DoD transferred the property and the Fosham Corporation owned the area (AGC, 2019). D&M (1981) indicated that local residents used the landfill as a trash dump after the security fences were removed from the Site in 1949. Three prior studies

QAPP Worksheet #10: Conceptual Site Model, Continued

have been completed at this AOC; the following section summarizes the results. There is no record that the Former Landfill was capped; although, D&M (1981) indicated that the landfill was covered with soil prior to removal of the security fences. A parking lot serving the golf course and club house currently covers the landfill.

A former open storage area, located southwest of the former landfill on the southern side of Potter Road in an area that is currently part of the golf course fairway, has been identified (see **Figure 10-4**). The open storage area is visible on two aerial photos dated February and August 1944 which suggests DoD use (AGC, 2019). Between 1944 and 1949, the open storage area ceased activity and while some surface debris was still present, the area is mostly cleared and vegetation is reestablishing itself (AGC, 2019). No other documentation of the history or use of this storage area was found.

AOC 4 – Former Bunker Area

The Former Bunker Area is located within the Three Rivers WMA (**Figure 10-5**). According to the Archives Search Report (USACE, 1999a): "…boxed AP (ammonium picrate) was taken by truck from the AP production lines and stored in the Storage Magazine Area prior to shipment." The fourteen original bunkers each measured approximately 30 ft by 60 ft and were constructed with concrete walls on three sides and front wall with a barn door for access (NYSDEC, 1993). The ceilings were constructed of wooden rafters. Earth was built up around the outside walls on the three concrete sides. An apron allowed access to each of the bunkers. Current use of this area is recreational with remaining bunkers for WMA maintenance equipment and vehicle storage.

AOC 5 – Former Power Plant Area

The Former Power Plant Area is located north of the Administration Area (**Figure 10-6**). The AOC included a large aboveground storage tank (AST), a boiler house, coal storage, and an electric substation. There were also two drainage ditches that ran across the area (AGC, 2019). The large AST contained "Fire Water" (Reconstruction Finance Company, undated). Additional site features are shown in **Figure 10-6**. Features include:

- An abandoned rail line and spur;
- Three below ground coal aggregate bins in line with the rail spur: a dual bin (bins A and B) located between the substation and the boiler house, and a single bin (bin C) located west of the boiler house;
- A second water AST and pipeline between the large AST and the boiler house, as well as a short water distribution pipeline located north of the Site;
- Additional drainage ditches;
- A possible dump; and,
- Former roads.

The boiler house was a two-story building with a total area of 8,366 ft^2 (Reconstruction Finance Company, undated). No additional written historical information could be found. The former Power Plant Area was not investigated in studies by D&M (1981), M&E (1990) or Alion (2006). Current use of this AOC is as undeveloped land.

QAPP Worksheet #10: Conceptual Site Model, Continued

10.1.3 Post-DoD Use and FUDS Eligibility

AOCs 1 and 2:

The NYSUDC has developed the Former Ammonium Picrate Area (AOC 1) into the residential/light industrial community now known as the Radisson Community (RC). The majority of the Ammonium Picrate Area structures have been demolished, but some structures remain including the ramps, Ammonium Picrate "bunkers", and assorted foundations. These remaining structures are deteriorating and do not appear to be maintained.

The USACE INPR (USACE, 1991) explicitly states that most of the DoD structures have been demolished, but "some of them were probably deemed too expensive and complex to remove." These structures include the water tower, six bunkers, acid plant, five acid tanks, pump house, and assorted foundations. In a letter dated 19 January 1990 to the USACE, M&E concluded that five tanks, three at the Former Ammonium Picrate Area (AOC 1) and two at the Former Acid Area (AOC 2) posed an imminent hazard to the public. In February 1991, NYSDEC notified the USACE of the need for an emergency response action (Gerrard, 1993; NYSDEC, 1991). NYSDEC concluded that they had the ability to conduct the clean-up more quickly than the USACE; therefore, the NYSDEC undertook the emergency response action (Gerrard, 1993). The emergency response included removal and disposal of approximately 15-20 tons of debris per tank and corrosive water, i.e., pH of less than 2, in the tanks (Gerrard, 1993; NYSDEC, 1991). NYSDEC neutralized approximately 78,000-gallons of water and disposed of the water at a local publicly owned treatment works (Gerrard, 1993; NYSDEC, 1991). NYSDEC then placed 28 cubic yards of limestone at the bottom of each tank and installed drainage holes in the tank sides (Gerrard, 1993; NYSDEC, 1991).

AOC 2 was privately owned subsequent to DoD usage, namely Fosham Corporation, First National City Bank, and the 1001 East Genesee Corporation and the title transfer document does not obligate the Government to conduct site restoration. Due to these circumstances, the five acid tanks in AOC 1 and AOC 2 are not eligible for demolition and removal under DERP-FUDS. Per Section 3-1.5.3.10 Restoration Already Initiated of Regulation No. 200-3-1 Environmental Quality - FUDS Program Policy dated 10 May 2004 (FUDS ER-200-3-1), if a Component has already initiated environmental restoration activities then the "Component" (the acid tanks) are ineligible. Additionally, in accordance with FUDS ER 200-3-1 projects where a hazard is a result of neglect by an owner/grantee subsequent to DoD use (regardless of whether the deed or disposal document required the owner/grantee to maintain the property improvements in accordance with FUDS ER-200-3-1) are ineligible under FUDS. Subsequently residual impacts from the acid tanks are also not FUDS-eligible. Potential concerns at AOC 2 stemmed from the two acid tanks and the former buildings that were removed and remediated post-DoD use. The Program Policy expressly provides that properties where restoration has already been initiated are ineligible under the FUDS Program. At AOC 2 a significant amount of work has been performed after the property was transferred out of DoD ownership. This work included the remediation of the two acid tanks involving the removal of debris and corrosive water, off-site disposal of water, and the installation of limestone and drainage holes in the tanks. Subsequently the Acid Tank Area infrastructure including all of the buildings, acid and water tanks, railroad tracks, scrubbers, silo, storage areas, loading platforms, change house, coal hopper and associated foundations were demolished and disposed off-site.

Based on the information regarding the work performed at this site and several site inspections, it is apparent that restoration has been initiated at AOC 2. The CENAE Office of Counsel has been consulted

QAPP Worksheet #10: Conceptual Site Model, Continued

and they determined that AOC 2 does not meet the criteria for property eligibility under the FUDS Program.

Subsequently, residual impacts from the acid tanks located at AOC 1, the Former Ammonium Picrate Area, are also not FUDS-eligible, and this portion of AOC 1 will not be addressed.

AOC 3: The construction of a golf course and associated structures including the club house, parking lots, and storage buildings have replaced the Former Landfill Area (AOC 3). Since there is no indication that environmental restoration has already been initiated by owners subsequent to DoD and the potential hazard is not a result of post DoD use, AOC 3 is FUDS-eligible.

AOC 4: The Former Bunker Area is located within the Three Rivers WMA. Twelve of the 14 bunkers remain intact (AOC 4). Bunkers #3 and #8 were used by NYSDEC personnel for the storage of pesticides for approximately ten years, from the early 1970s to 1983 (M&E, 1990). A small partitioned room (approximately 12 ft by 22 ft) within Bunker #3 was used to receive and package pesticides from citizens and/or businesses that had pesticides that were either unwanted or had been banned. Bunker #8 was used for receiving and repackaging pesticides, but was also used for pesticide storage (NYSDEC, 1993). Bunkers #3 and #8 are no longer in use. In 1992, NYSDEC emptied and cleaned Bunkers #3 and #8 and covered with them an impermeable cap. NYSDEC currently maintains responsibility for long-term management of Bunkers #3 and #8, such as lawn mowing and cap inspection. The remaining bunkers are being used by NYSDEC game management personnel for equipment and vehicle storage. Due to the initiation of environmental restoration subsequent to DoD-ownership, Bunkers #3 and #8, are not FUDS-eligible and will not be addressed. The remaining 12 bunkers remain FUDS-eligible.

AOC 5: Recent aerial photos indicate that the former power plant and associated structures are no longer present. No documentation was encountered regarding if the power plant was removed and disposed by the military or by some other entity after the NYOW was decommissioned. Any groundwater or soil impacts from plant operations would be FUDS-eligible unless a non-military entity conducted remediation activities subsequent to DoD ownership. There is no evidence that this occurred; therefore, AOC 5, the Former Power Plant Area, is FUDS-eligible.

10.2 PHYSICAL CHARACTERISTICS

The Site is located in the southeastern portion of the Erie-Ontario Lowlands Physiographic Province where several episodes of glacial ice advance and recession have resulted in rolling topography dominated by the deposition of drumlin hills, flanked by flat-lying glacial lake plain deposits. Elevation at the Site ranges from approximately 400 ft near AOC 3 and AOC 4 to approximately 500 ft above mean sea level (msl) on the drumlin hill adjacent to AOC 1 (M&E, 1990).

10.2.1 Meteorology

Based on data for Syracuse Hancock International Airport, NY, the area receives approximately 38.5 inches of annual precipitation. The hottest month is historically July, with an average high temperature of 81.6 Degrees Fahrenheit (°F), while its coldest month is historically January with an average high temperature of 31.5 °F (National Oceanic and Atmospheric Administration [NOAA], 2017).

QAPP Worksheet #10: Conceptual Site Model, Continued

10.2.2 Surface Water Drainage

The Seneca River flows south of the Site, meandering from west to southeast of Baldwinsville, then turns north, where it joins the Oneida River to form the Oswego River northeast of the Site. From there, the Oswego River flows north-northwest and drains into Lake Ontario (**Figure 10-1**). The Site lies entirely within the Oswego River drainage basin. It is expected that approximately half of the annual precipitation becomes surface water runoff (D&M, 1981). Small swampy areas are abundant at the Site due to the relatively flat-lying topography and poor drainage resulting from the former lakebed deposits. The glacial lake plains in the area are drained primarily by small streamlets which flow in a general northeasterly direction towards the Oswego River and eventually into Lake Ontario. Surface drainage is also influenced by local topography.

Surface drainage at the Former Ammonium Picrate Area (AOC 1) consists of former wastewater ditches that ran from the northeastern side of the Ammonium Picrate Area. These ditches joined northeast of the Former Acid Area and flowed northward to the Oswego River (**Figure 10-2**). A second former wastewater ditch originated in the Administration Area and flowed to the north and discharged into a low area east of the present-day Willette Parkway (D&M, 1981).

At the Former Landfill Area (AOC 3) there are surficial drainage features just north of the parking lot, with one draining to the west, and the other to the northeast, with intervening high ground between them (**Figure 10-4**).

At the Former Bunker Area (AOC 4), inside each bunker there are two interior drains that run the length of the bunker and discharge on exterior of either side of the front of the bunker. Drainage swales run parallel along the length of West and East Igloo Roads.

At the Former Power Plant Area (AOC 5), drainage ditches to the east and southwest of the AOC were identified by AGC (2019).

10.2.3 Geology

The Site is located within the southeastern portion of the Erie-Ontario Lowland Physiographic Province, which is characterized by the gentle topography of a Pleistocene-age lakebed punctuated by northwest-southeast trending drumlins (M&E, 1990), reflecting the direction of ice movement. Relief in the region is not controlled by bedrock but by the Pleistocene-age glacial deposition.

Glacial geology of the area has been mapped by Muller and Cadwell (1986) (**Figure 10-8**) and by Pair (2014) (**Figure 10-9**). The 1986 map (**Figure 10-8**) shows interpreted geologic/glacial units based on depositional environment, using terms such as till, lacustrine silt and clay, and lacustrine beach deposits. As can be seen in **Figure 10-8**, the drumlin hills are mapped as glacial till (t), which was formed during glacial advancement, while the majority of the area is blanketed with lacustrine silt and clay (ls), deposited in lakes that formed beyond the edge of the ice sheet (proglacial lakes). Some lacustrine beach deposits (lb) are shown flanking the drumlin hills, where erosion and wave-cutting may have occurred, winnowing out the fines from the till, leaving behind more granular deposits.

The 2014 mapping (**Figure 10-9**) uses textural terms to describe the surface geologic units, and does not make genetic interpretations. For example, in the 2014 map, the till is mapped as diamicton (Pd), the

QAPP Worksheet #10: Conceptual Site Model, Continued

lacustrine deposits are mapped as silt and clay (Psc), and the flanking beach deposits as stratified silt, sand and gravel (Ps). Conceptually, the two maps agree, but the 1986 mapping did not match up as closely with the topography of the known landforms, and the 2014 mapping appeared more precise in its definition of the extent of the various surface geologic units. For this reason, the 2014 mapping by Pair is shown as an overlay on Site figures.

AOC 1: The hill where the Former Ammonium Picrate Area is located is a drumlin, composed of diamicton/till (**Figure 10-9**), consisting of unsorted clay, silt, sand, gravel, and boulders (Pair, 2014). Drumlins were shaped by glacial ice movement over the land surface, resulting in tightly compacted heterogeneous material. Thickness of the unit ranges from 1 to 150 ft in the region. Prior borings at AOC 1 (MW-7) terminated at a maximum depth of 51 ft below ground surface (bgs) (approximately 400 ft above sea level) before reaching refusal, likely on bedrock. This unit has low permeability and may cause unstable upland slopes (Muller and Cadwell, 1986). Additional areas of diamicton deposits are in the southwestern portion of the Site west of the Former Administration Area, northwest of Greene Pond straddling Smokey Hollow Road in the northwestern portion of the Site, and in the north central portion of the Site straddling Lamson Road (**Figure 10-8**). Groundwater flow through this unit is expected to be very slow due to the tightly compacted, poorly-sorted, heterogeneous nature of the material.

Along the northeastern and southwestern edges of the diamicton (**Figure 10-8**) are deposits that Muller and Cadwell (1986) mapped as lacustrine beach deposits and Pair (2014) mapped as stratified silt, sand, and gravel. The areas covered by these deposits also differ, with Pair (2014) mapping a significantly smaller area than Muller and Cadwell (1986) for this unit. Presumably the more recent mapping in 2014 is more accurate, as the mapped diamicton areas better align with the drumlin hills.

While the different maps (**Figures 10-8** and **10-9**) vary in the extent and nomenclature for the deposits flanking the drumlin ('lacustrine beach' vs. 'stratified silt, sand, and gravel'), both reflect a water-worked deposit that is well-sorted and coarser-grained, having had fines winnowed out by water/wave action. The drumlin hill is composed of glacial diamict (till) and formed during glacial advancement. The 'diamicton' refers to the consolidated equivalent of the poorly-sorted glacial diamict/till deposits, which are generally deposited directly from the ice, rather than winnowed or reworked by water. As the glacier melted and retreated to the north, this area was inundated by a lake formed by glacial meltwater. Fine sand, silt, and clay were deposited in this predominantly isolated environment. Coarser water-worked deposits would be expected along the flanks of any nearby drumlins or within the terminal moraine. Given the differences in mapping, the areal extent of the unit is uncertain. Thicknesses are reported to range from 6 to 30 ft (Muller and Cadwell, 1986). Similar areas of the lacustrine beach deposits are in the western portion of the Site flanking the other drumlins in the area (refer to **Figure 10-8**).

AOCs 3, 4, and 5: The flat-lying area surrounding the drumlin hills, where AOCs 3, 4, and 5 are located, consist of stratified (thinly layered) lacustrine silt and clay (Pair, 2014) and range from approximately 2 to 60 ft in thickness. These sediments were deposited as the glacier retreated, in a lacustrine environment during a high period of glacial Lake Iroquois, a predecessor to Lake Ontario. Permeability, and thus infiltration and migration, is expected to be relatively low in the lacustrine deposits due to the fine-grained nature of the material.

QAPP Worksheet #10: Conceptual Site Model, Continued

Bedrock was encountered in three borings at AOC 3 (D&M-11, Boring B-2, and MW-8) at depths of approximately 15 ft bgs (385 to 389 ft above sea level). About halfway between AOC 4 and AOC 3 at well D&M-6, bedrock was encountered at a depth of 9 ft bgs, which corresponds to approximately 400 ft above sea level. Depth to bedrock has not been determined at AOC 5 because boreholes have not been completed in this area. Well D&M-10 is the closest well in proximity to AOC 5, and bedrock was not encountered in that boring.

Bedrock geology (**Figure 10-10**) beneath the Pleistocene-age deposits consists of sedimentary rock units of the Mid to Upper Silurian-age Clinton, Lockport and Salina Groups. It should be noted that the stratigraphic nomenclature assigned to various units in this region of New York has been modified over the last 60 years. The most current stratigraphy mapped beneath the Site and surrounding area includes the Upper Silurian Vernon Shale overlying the Mid-Silurian Sconondoa Formation (Fm) of the Lockport Group. To the north of the Site, the Rochester Shale member of the Clinton Group is mapped, which is characterized as interbedded calcareous gray shales and thin limestone beds. The Rochester Shale is the oldest of the three units mapped in **Figure 10-10**.

The Guelph Dolostone belongs to the Lockport Group, and is characterized as an alternating light and dark gray, bituminous and irregularly bedded dolomite with stromatolite reef bioherms at the base of the member. To the northwest near Ontario, Canada, the Guelph unit ranges from 200 to 300 feet in thickness; however, the unit thins substantially to the southeast, and appears regionally as just a thin wedge of rock (Tepper et al., 1990).

The Vernon Shale belongs to the Salina Group (Schneider, 1894) and is comprised of red and gray shales with dolomitic beds (Rickard, 1969). The Vernon Shale lies conformably over the Guelph Dolostone, and the transition between the units is marked by light bluish gray wackestone to claystone, with evaporites and vertical burrows.

The Sconondoa Fm (also part of the Lockport Group) is mapped to the south of the Site, and is described as a fine-grained, medium-bedded and fractured limestone, with interbedded shale limestone and coarse-grained dolomite (Zenger, 1965). Thicknesses of the Sconondoa Fm vary but may be as great as 150 ft. Bedrock dips gently southward and no faults or folds have been detected in the overlying Vernon Shale (Chute, 1964). Based on the morphology of the Seneca River, which flows east and south of the Site, there may be northwest-southeast and northeast-southwest trending joints in the Vernon Shale. The Sconondoa has two primary sets of joints, one north-south and one east-west (Chute, 1964). Also within the Sconondoa are thrust faults that strike North 65 to 75 degrees west and dip to the south (Chute, 1964); however, there is no surface expression of these faults in the overlying Vernon Shale. The gentle southward dip of the rock units and jointing in the Vernon Shale may influence the direction of groundwater flow, and subsequently, the direction and rate of migration of potential DoD-impacts from possible source areas. The fracture trends observed in the Sconondoa Fm may be indicative of regional trends, and continue into the formations to the north.

AOCs 1 and 5 in the south appear to be underlain by the Vernon Shale, while those to the north (AOCs 3 and 4) are likely underlain by the Guelph Dolostone. It is possible that soil borings advanced in northern portion of AOC 1 may intercept this geologic contact.

QAPP Worksheet #10: Conceptual Site Model, Continued

10.2.4 Hydrogeology

Regional groundwater flow is toward surface water bodies (Pagano, et al., 1985), and the major river drainages in the area, toward the Seneca River to the south and the Oswego River to the north (**Figures 10-1 and 10-2**). Groundwater at the Site AOCs generally flows northeast toward the Seneca and Oswego Rivers. However, groundwater also tends to mimic the land surface, and thus influenced by local topography. Therefore, shallow groundwater locally may be controlled by nearby surface water features. Deep groundwater will be consistent with regional flow toward the rivers, ultimately draining northward to Lake Ontario.

Based on previous investigations by NYSDEC, the direction of groundwater flow at the Ammonium Picrate Area (AOC 1), and the Former Landfill (AOC 3) is generally northeastward toward the Oswego River (D&M, 1981, and M&E, 1990). AOC 3 is located about 0.75 miles southwest of the confluence of the Seneca and Oswego Rivers. Groundwater flow locally at the Former Bunker Area (AOC 4) may be to the west, south, or northeast, towards local surface water drainage features. Groundwater flow at the Former Acid Area was found to be to the northwest (Alion, 2008). Flow direction and velocity will be controlled by differences in head, the locations of groundwater discharge points in the surrounding area, and the hydraulic conductivities of the surficial units.

Based on historical assessments, groundwater was encountered across the Site within the lacustrine and till deposits. However, there are currently no wells screened in the beach deposits. The lacustrine silt and clay deposits are reported to range from 2 to 60 ft thick and the till from 1 to 150 ft thick (Muller and Cadwell, 1986).

With the exception of two deeper wells (MW-3 and MW-7), the existing monitoring wells at the Site are shallow, and screened at depths of less than 20 ft bgs. Previous investigations have encountered the water table at a depth of 9 ft bgs in the till at the Ammonium Picrate Area (AOC 1), and at 4 to 7 ft bgs in the lacustrine deposits at Acid Area (AOC 2). Similar shallow water levels were encountered at AOCs 3 and 4. One of the deeper wells, MW-7, at the Ammonium Picrate Area, is screened from 31 to 51 ft bgs, and the depth to water in this well is 37 ft bgs. This well is screened in either till or weathered shale bedrock (gray silty clay with angular rock fragments). The bottom of the boring was described as red shale. The deeper water level in this well indicates a downward gradient, and possibly poor hydraulic communication with the shallow groundwater. The other deeper well, MW-3, in the Acid Area (AOC 2) is screened from 38 to 48 ft bgs. The depth to water in this well is comparable to water levels in the other wells in the Acid Area, indicating that this well is hydraulically connected to the shallow unit.

Groundwater is likely to be present within the relatively thin bedding planes and fractures of the Vernon Shale. Shales generally yield only small supplies of water; however, the dissolution of dolomitic layers present in the Vernon Shale would facilitate increased groundwater flow through the aquifer. Kantrowitz (1964) found that the Vernon Shale yields as much as 230 gallons per minute where dissolution of the interbedded dolomite has occurred. However, the average yield from this formation is 15 to 20 gallons per minute. The Lockport Formation is expected to be especially susceptible to dissolution by percolating groundwater because it consists primarily of fractured limestone and dolomite. LaSala (1968) has found wells in the Lockport to yield up to 100 gallons per minute. No site monitoring wells are screened in bedrock.

QAPP Worksheet #10: Conceptual Site Model, Continued

M&E (1990) calculated hydraulic conductivities at the Former Ammonium Picrate Area, and the Former Landfill Area. The calculated hydraulic conductivities of the deposits in the Former Landfill Area were generally higher by one or two orders of magnitude than the deposits in the Former Ammonium Picrate Area. The higher hydraulic conductivity in the Former Landfill Area [0.4 to 4 feet per day (ft/day)] may be attributed to the fact that the lacustrine deposits, while fine-grained (silt and clay), were not overridden by glacial ice. The Former Ammonium Picrate Area was composed of less permeable and more poorly sorted subsurface deposits, further compacted at the base of the ice sheet, and thus has lower hydraulic conductivities (0.004 to 0.024 ft/day) (M&E, 1990). The stratified silt, sand and gravel deposits flanking the drumlin would be expected to have even higher hydraulic conductivity, but there are no monitoring wells or conductivity data available for this unit.

10.2.5 Sources of Drinking Water

Potable water is supplied to communities in Onondaga County by the Onondaga County Water Authority, which draws water from several sources, including Otisco Lake, Lake Ontario, and/or Skaneateles Lake. For the Lysander-Baldwinsville area (including the Radisson Community and Golf Course), potable water is obtained from Otisco Lake and Lake Ontario and then treated and distributed to the community (Onondaga County Water Authority, 2019). Site impacts on these surface water sources are unlikely given that regional groundwater flow is to the north and distance from the Site. Otisco Lake is located about 20 miles south of the Site, far upgradient of the Site. Lake Ontario is about 20 miles northwest of the Site, far downgradient of the Site.

A water well services the NYSDEC field office at the Three Rivers Wildlife Management Area in AOC 4. Some residences in the surrounding area may have private supply wells. In 2007, NYSDOH was contacted by USACE to identify wells and water supply systems within a 4-mile radius of the Site. NYSDOH provided general information on the surrounding wells and source water assessment areas but was unable to provide specific information due to confidentiality protocols (Alion, 2008). A potable well survey will be conducted during the first phase of this study.

10.2.6 Terrestrial Habitat

General descriptions of the terrestrial habitat of each AOC are noted below. Potential ecological receptors are discussed in **Section 10.6.4**.

<u>AOC 1 – Former Ammonium Picrate Area</u>

The majority of the Ammonium Picrate Area terrestrial habitat is forested and is very similar to that described in the following Section for the Bunker Area, the major difference being a much less dense understory layer. In general, vegetation composition and expected wildlife are similar between the two areas. The Ammonium Picrate Area has maintained trails and recreational grassy areas interspersed throughout the Site.

<u>AOC 3 – Former Landfill Area</u>

The Former Landfill Area is covered by a blacktop parking lot. The areas south and east of the landfill are dominated by residential properties and west of the landfill is additional blacktop parking and commercial properties associated with the Radisson Greens Golf Course. North of the former landfill is a mature

QAPP Worksheet #10: Conceptual Site Model, Continued

deciduous forest (roughly 200 ft wide) that continues to the northeast and borders one of the golf course fairways.

AOC 4 – Former Bunker Area

The bunkers are covered with grass and at most places are periodically managed to discourage the rooting of trees and shrubs on top of the bunkers. Across the road from the bunkers, a heavily vegetated drainage ditch runs parallel to the road in front of each bunker. Terrestrial habitat bordering Igloo Road and associated bunkers consists primarily of grassy fields and northern deciduous forest. The following provides a brief description of the primary terrestrial habitat location within the Bunker Area.

Grass Fields – Located predominantly along the northern portion of the Bunker Area although smaller grass field patches occur around each of the bunkers. The extent of grass maintenance around the bunkers is limited.

Woodlands – Eastern deciduous forest borders the Bunker area to the north and east; second growth forest of similar composition is located between the two bunker rows except at the northern end. The southeastern Site border is a mix of maintained grassy areas and deciduous forest.

AOC 5 – Former Power Plant Area

Areas within AOC 5 appear to have been filled to raise contour elevations to support infrastructure and facilitate the power plant operation. The former power plant footprint includes a grass/lawn swath which transitions into a fairly dense growth of colonizing early-successional shrub and tree species There is a flatwoods wetland area to the west of the fill footprint that appears to have had minimal direct disturbance other than past efforts to enhance drainage with shallow open ditches.

10.2.7 Wetlands and Aquatic Resources

General descriptions of the wetlands and aquatic resources associated with the AOCs are noted below. Potential ecological receptors are discussed in **Section 10.6.4**.

According to the National Wetlands Inventory Map managed by the United States Fish and Wildlife Service (USFWS), there are several freshwater forested/shrub wetland and freshwater emergent wetland areas in the western portion of the Site (primarily in the undeveloped areas of Three Rivers WMA). The freshwater forested/shrub wetland category consists of woody wetlands, forested swamp, or shrub bogs. Freshwater emergent wetlands are described as herbaceous marsh, fen, swale, and wet meadow (USFWS, 2018). There are also several state-regulated freshwater wetland areas within the site that do not necessarily directly coincide with the National Wetlands Inventory Map (the NYSDEC Environmental Resource mapper at https://www.dec.ny.gov/animals/38801.html and Wetlands Research Services, 2020). There are several small freshwater ponds located within the Radisson Community.

During the 27 June 2018 site visit, large forested-shrub wetlands were observed roughly a half-mile west and southeast of AOC 1; and, small scrub-shrub and emergent wetland patches were observed adjacent to and southwest of AOC 4.

QAPP Worksheet #10: Conceptual Site Model, Continued

According to the Habitat Management Plan for Three Rivers WMA 2018-2027, there are 64 acres of open water, 468 acres of natural wetland, 113 acres of impounded wetland, and 20 miles of rivers and streams at Three Rivers WMA (NYSDEC, 2018).

A narrow creek, bordered primarily woodland habitat, is located approximately 75 ft north of the Former Landfill Area in AOC 3.

10.2.8 Land Use and Demography

NYOW is currently occupied by the Three Rivers WMA and the Radisson Community with its associated industrial properties (**Figures 10-2 and 10-7**). Part of the first phase of this study will be determining and verifying current and potential future land use.

The Three Rivers WMA is situated on the northern portion of the Former NYOW property and was acquired from the US Government when the property was transferred in 1947. It was named for its proximity to the junction of the Seneca and Oneida Rivers, which form the Oswego River. The WMA is flat and poorly drained, with fields, woods, ponds, and marshes. It is used primarily for recreational use - hunting, fishing, hiking, and bird watching. Future use is expected to remain the same as current use.

The Radisson Community is a planned development community that provides approximately 3,500 homes, as well as a large Corporate Park and a wide range of recreational, educational, cultural, shopping and community services. The area is served by underground water, sewage, gas, and telephone utilities. The Radisson Corporate Park provides building sites for industry and offices. To date, 40 firms employing 2,500 people have located in the park. These firms produce products as diverse as beer, plastic containers, printing, and electronic components. The Seneca River borders the community on the east, Three Rivers WMA borders the community on the north and west, and New York State Route 31 borders the area on the south (Radisson, 2017). The Radisson Community actively uses much of the property and some apartment buildings and residences in the Radisson Community appear to be constructed on or around the sites of former dryer houses, change houses, and other historical site features. In addition, paved walking paths and grassy areas wind between homes and wooded areas containing the old loading ramps, storage bunkers, and loading dock structures. Old concrete structures in disrepair exist in wooded areas of varying density. Current use of potentially impacted areas is limited to recreational use. Future use is expected to remain the same as current use.

10.3 PREVIOUS SITE INVESTIGATIONS AND AVAILABLE DATASET

Previous investigations focused on four AOCs at the Site, including AOC 2:

- AOC 1 – Ammonium Picrate Area
- AOC 2 – Acid Area
- AOC 3 – Former Landfill
- AOC 4 – Bunker Area

AOCs 1, 3, and 4, except for two of the eight bunkers in AOC 4, are HTRW FUDS-eligible. As previously discussed, AOC 2 is not HTRW FUDS-eligible; therefore, previous investigations of AOC 2 are not discussed

QAPP Worksheet #10: Conceptual Site Model, Continued

in this QAPP. There were no previous investigations of AOC 5; therefore, no historical sampling data are available for AOC 5. The locations of the AOCs are shown on **Figure 10-2**.

The historical data were compared to the following criteria to provide context for historical sampling results:

Standards/Guidance Values	Media Evaluated
EPA Regional Screening Levels (RSLs) – Residential Soil, Industrial Soil, and Tapwater, dated November 2020	Groundwater Soil
EPA Ecological Soil Screening Levels (Eco-SSLs), Interim Final, dated February 2005 through April 2008	Soil
Region III Biological Technical Assistance Group (BTAG) Freshwater Screening Values, 2006	Tank Water

These studies provide a basis for understanding the historical background of the Site and identify potential sources of impacts to groundwater. present the comparisons of historical data to the criteria above. The discussion of previous investigations is presented in the following subsections by area, and chronologically by investigation.

10.3.1 AOC 1 – Ammonium Picrate Area

Previous investigations conducted in this area were the D&M (1981) Site Investigation and M&E (1990) Preliminary Impact Evaluation.

D&M (1981)

In 1981, NYSUDC and the NYSDOH contracted D&M to investigate four AOCs at the former NYOW to evaluate the potential hazard of constituents to the Radisson Community. D&M conducted a literature search and site investigation. The site investigation included collecting soil and groundwater samples from 20 soil borings (with 2-inch slotted polyvinyl chloride [PVC] installed in 18 of the boring locations, for use as piezometers (but referred to as wells by [D&M) throughout the Radisson Community portion of the Site and three soil grab samples along former wastewater ditches.

Ten of the soil borings were conducted in the Former Ammonium Picrate Area (AOC 1), with eight locations converted to piezometers (i.e., all but locations D&M-8 [no explanation] and D&M-9 [dry]). The D&M sampling locations at AOC 1 are depicted on **Figure 10-3**. Groundwater and soil samples were analyzed for total acid; DNCB; 2,4- and 2,6-dinitrophenol (DNP); and, 2,4,6-trinitrophenol (also referred to as picric acid). Standard EPA methods were not used during the D&M investigation. Appendix 2 of the D&M report discusses the analytical methods used by Syracuse Research Corporation (SRC) for the analysis of picric acid in water [high performance liquid chromatograph (HPLC)], 2,4-DNP and DNCB in soil (HPLC), and picric acid in soil (extraction followed by HPLC, and mass spectrometry for confirmation) (D&M, 1981). SRC concluded that the matrix spike could neither confirm, nor deny, the presence of picric acid without gas chromatograph separation (D&M, 1981). In 1981, there was not a standard method for picric acid analysis and a specialty lab was employed. Using various extraction methods and HPLC, the best recovery of picric acid spiked soil was 41%. GC-MS was investigated as a possibility for confirming results; however, GC condition for picric acid was unavailable at that time and development would have

QAPP Worksheet #10: Conceptual Site Model, Continued

been fairly involved. The decision was made to analyze the soil extract directly by MS. Interferences from other components in the mixture prevented identification of the picric acid by MS (Appendix A of D&M, 1981). There is no evidence in the D&M (1981) report as to whether the picric acid results were rejected or even validated.

One grab soil sample from ditches near the Former Ammonium Picrate Area was also collected. DNCB concentrations in groundwater ranged from 0.0061 milligrams per liter (mg/L) to 0.0098 mg/L, and picric acid concentrations ranged from 0.0076 mg/L to 0.197 mg/L (**Table 10-1**). The maximum concentrations of picric acid were collected from locations D&M-10 (0.197 mg/L), located near the discharge for the Former Administration Area sewer system, and AMS-1 (0.0567 mg/L), located at the base of an industrial sewer that collected wastes from the Former Ammonium Picrate area. Each of the D&M piezometers were resampled and analyzed using the same methods; however, picric acid was not detected in subsequent sampling efforts. D&M did not compare their groundwater results to any reference criteria. The current (November 2020) Tapwater RSL for picric acid is 0.040 mg/L. One of the three picric acid detections at AOC 1 exceeded this criterion.

No DNP or DNCB were detected in soil samples; however, picric acid was detected in most of the soil samples (**Table 10-2**) in concentrations ranging from 0.0024 milligram per kilogram (mg/kg) to 0.0622 mg/kg. The current (November 2020) residential RSL for picric acid is 130 mg/kg. All of the D&M soil sampling results were well below this criterion.

The 1981 D&M investigation concluded that the Former Ammonium Picrate Area had been decontaminated by removal of equipment and supplies, that some impacts of subsurface soils and groundwater had occurred, and that the picric acid released to the environment would eventually degrade to nitrate. D&M also concluded that picric acid was not present in groundwater in concentrations indicative of a constituent plume and that the source of the impacts in the Former Administration Area was thought to be from runoff of decontamination water that resulted from pressure washing of production hardware that was done in this area during decommissioning of the plant after it was closed down.

<u>M&E (1990)</u>

A preliminary impacts evaluation was conducted by M&E for USACE Kansas City District, at the former NYOW Site as part of the DoD DERP between 1986 and 1989, with the findings published in January 1990 (M&E, 1990). The purpose of this investigation was to determine the presence or absence of chemical impacts from DoD activities at the Former Ammonium Picrate Area and the Former Acid Area, and to determine the potential for impacts to local soil, groundwater, and surface water.

During the M&E investigation, the soil sampling results were compared to New Jersey reference background values and cleanup levels, because New York standards did not exist for soils at the time of this study. The following standards, criteria, and guidance were used by M&E to screen the results (M&E, 1990):

QAPP Worksheet #10: Conceptual Site Model, Continued

Standards/Guidance Values	Media Evaluated
NYSDEC New York State Ambient Water Quality Standards and Guidance Values, dated April 1987	Groundwater Tank water
NYSDEC standards in April 1987	Groundwater Tank water
New Jersey Department of Environmental Protection (NJDEP), Summary of Approaches to Soil Cleanup Levels, dated January 1987 [New York State guidance was not available at the time]	Surface Soil
EPA Ambient Water Quality Criteria for Human Consumption, Office of Water Regulations and Standards, September 1986	Surface Water
U.S. Background, provided in Table 1 of the NJDEP Summary of Approaches to Soil Cleanup Levels, dated January 1987	Surface Soil

At the time of the M&E evaluation, the concrete foundations of the six production areas still existed (**Figure 10-3**). Houses and apartments had been built on top and at the base of the drumlin, a local topographic high, where the Former Ammonium Picrate Area was located. Six concrete storage bunkers built into the drumlin remained intact. The bunkers were open and accessible to the public. Holes existed in the walls of the three brick storage tanks, which were uncovered and contained liquid. The area was partially overgrown by trees and vegetation. Public access was unrestricted to this area. Water table elevations were the highest at the Site in the Ammonium Picrate Area. Groundwater flow in the Ammonium Picrate Area was determined by D&M (1981) to be northeasterly toward the Former Landfill area and the Oswego River. The inferred groundwater flow direction was based on six shallow piezometers (depths ranging from 8 to 14 ft bgs installed in and around the Ammonium Picrate Area.

Three groundwater monitoring wells (MW-5 through MW-7) were installed in and around the Ammonium Picrate Area (**Figure 10-3**):

- Well MW-5 – Considered to be upgradient of the production plant, was installed to a depth of 14.5 ft
- Well MW-6 – Downgradient shallow well installed to a depth of 15 ft
- Well MW-7 – Deeper well (51 ft bgs) installed adjacent to well MW-6

Groundwater samples were collected and analyzed for volatile organic compounds (VOCs) (Method 8240), semi-volatile organic compounds (SVOCs) (Method 8270), DNCB (Method 8270), picric acid [United States Geologic Survey (USGS) Method], nitrite/nitrate (Method 353.1), and total metals (arsenic [Method 3020/7060]; barium, cadmium, chromium, lead [Method 3005/6010]; mercury [Method 7470]; selenium [Method 3020/7740]; and, silver [Method 3005/6010]).

M&E compared groundwater concentrations to New York State (NYS) Groundwater Standards, published in April 1987. VOCs and SVOCs were below the laboratory detection limit (DL) in the samples collected from monitoring wells MW-5 through MW-7. Unfiltered (total) metals concentrations were below the 1987 NYS Groundwater Standards for the monitoring wells in this area, with the exception of lead in MW-5 (0.037 mg/L). Nitrates were detected in the sample from monitoring well MW-7 (0.560 mg/L), which was located adjacent to a shallower well (MW-6) where nitrates were not detected. MW-7 was screened

QAPP Worksheet #10: Conceptual Site Model, Continued

in till/weathered shale, at a greater depth than MW-6, potentially indicating that picric acid, which is very water soluble, may be degrading to nitrate and migrating to a deeper aquifer (M&E, 1990). However, all the nitrate concentrations were well below the NYS Groundwater Standard.

Table 10-3 compares the M&E groundwater results to current (November 2020) RSLs to determine if impacts at the Ammonium Picrate Area exceed present risk-based screening levels. No nitrite/nitrate concentrations exceeded the RSL. Total arsenic exceeded the current (November 2020) RSL in well MW-6. Total lead exceeded the current (November 2020) RSL, and total chromium exceeded the current (November 2020) hexavalent chromium RSL in well MW-5. Note that the comparison to hexavalent chromium RSL is conservative given most chromium present in the environment is in the trivalent form. Based on current (November 2020) criteria, metals are the primary DoD-related chemicals of potential concern in groundwater for this area; however, it should be noted that M&E indicated that the NYOW groundwater samples contained an appreciable amount of suspended sediments (M&E, 1990).

Five soil samples were collected in the Ammonium Picrate Area (**Figure 10-3**), at a depth of approximately 6 inches bgs:

- Soil Sample #7 – Collected adjacent to MW-5, intended as a background sample for the Ammonium Picrate Area
- Soil Sample #8 – Collected approximately 25 ft west of MW-6 and M-7, near the bunker opening at the base of production line #5 (marshy soil)
- Soil Sample #9 – Collected downgradient of and on the east side of Tank #3 (marshy soil)
- Soil Sample #10 – Collected downgradient of and approximately 5-10 ft east of Tank #2 (mostly rocky soil)
- Soil Sample #11 – Collected downgradient along the east side of Tank #1

Soil samples were analyzed for VOCs (Method 8240), SVOCs (Method 8270), DNCB (Method 8270), picric acid (USGS Method), nitrite/nitrate (Method 353.1), and total metals [arsenic (Method 3050); barium, cadmium, chromium, lead (Method 3050/6010); mercury (Method 7471); selenium (Method 3050/7740); and, silver (Method 3050/6010)]. The soil sampling results were compared to 1987 NJDEP standards (for US Background Levels and NJDEP Action or Cleanup Levels) for comparative purposes, as NYS standards were not identified at the time for soils. VOCs and DNCB were below method DL. SVOCs, including several polycyclic aromatic hydrocarbons (PAHs), exceeded the NJDEP Action Level for total SVOCs of 10.0 milligrams per kilogram (mg/kg) in Soil Sample #7. Total metals concentrations in soil were within expected US Background Levels and below NJDEP Cleanup Levels. Nitrate concentrations were elevated in Soil Sample #7 (1.90 mg/kg), which was located along the road between the Ammonium Picrate Area and Acid Area. However, there were no NJDEP comparison criteria available for nitrates in soil. Spillage of chemicals may have occurred along the road during operation of NYOW. A comparison of the M&E soil data to current Residential Soil RSLs (EPA, 2020) and Eco-SSLs is provided in **Table 10-4**. PAHs exceeded residential RSLs and Eco-SSLs in Soil Sample #7. Benzo(a)pyrene also exceeded the residential RSL in Soil Sample #11. Arsenic, chromium, and lead exceeded reference criteria in most soil samples.

A surface water sample was collected from one location (SW-3) near the Ammonium Picrate Area (**Figure 10-3**). The surface water sample was collected with a stainless-steel dipper from a culvert that passes underneath North Entry Road. Water was observed to be flowing west to east. The surface water sample

QAPP Worksheet #10: Conceptual Site Model, Continued

was analyzed for VOCs (Method 8240), SVOCs (Method 8270), DNCB (Method 8270), picric acid (USGS Method), nitrite/nitrate (Method 353.1), and total metals [arsenic (Method 3020/7060); barium, cadmium, chromium, lead (Method 3005/6010); mercury (Method 7470); selenium (Method 3020/7740); and, silver (Method 3005/6010)]. The surface water concentrations of all the analyzed parameters were below the laboratory DL.

Three water samples were collected from the tanks in the Ammonium Picrate Area (**Figure 10-3**), using stainless-steel dippers:

- Tank Water Sample #1 – Collected from the top rim of Tank #1, along the southeast side of production line #1 and adjacent to a public walkway
- Tank Water Sample #2 – Collected from the top rim of Tank #2, along the southeast side of production line #4
- Tank Water Sample #3 – Collected from the top rim of Tank #3, along the southeast side of production line #6

The tank water samples were analyzed for VOCs (Method 8240), SVOCs (Method 8270), DNCB (Method 8270), picric acid (USGS Method), nitrite/nitrate (Method 353.1), and total metals [arsenic (Method 3020/7060); barium, cadmium, chromium, lead (Method 3005/6010); mercury (Method 7470); selenium (Method 3020/7740); and, silver (Method 3005/6010)]. VOCs were below the laboratory DL in the tank water samples. One SVOC (phenol) was detected in Tank Water Samples #1 and #3; however, comparison criteria did not exist at the time. Total metals concentrations and nitrates were below the NYS Groundwater Standards, with the exception of arsenic, cadmium, chromium, and lead in Tank Water Sample #2. The tank water was found to be acidic (pH ranging from 2.34 to 2.50), indicating that acid residues from NYOW operations remained in the tanks. A comparison of the M&E tank water data to current (November 2020) tapwater RSLs and BTAG freshwater screening benchmarks (EPA, 2006b) is provided in **Table 10-5**. Phenol exceeded BTAG criteria in Tank #2, and nitrite/nitrate BTAG criteria were exceeded in Tanks #1 and #3. Tank #2 also contained concentrations of arsenic, barium, cadmium, chromium, lead, and mercury in concentrations exceeding criteria. Lead was detected in concentrations exceeding reference criteria in water samples from all three tanks.

Wipe samples were collected within the bunkers in the Ammonium Picrate Area, located at the base of the six ammonium picrate production lines:

- Wipe Samples #1, #7, and #9 – Collected from the floor of Bunker #1 (closest to Willett Parkway)
- Wipe Sample #2 – Collected from one of the walls of Bunker #2, near the floor (since the concrete floor was covered with dirt)
- Wipe Sample #3 – Collected from the floor of Bunker #3
- Wipe Sample #4 – Collected from the floor of Bunker #4
- Wipe Sample #5 – Collected from one of the walls in Bunker #5, near the floor (since there were 2 inches of water on the floor)
- Wipe Sample #6 – Collected from the floor of Bunker #6

The wipe samples were analyzed for picric acid (USGS Method), DNCB (Method 8270), and pesticides/polychlorinated biphenyls (PCBs) (Method 8080). Picric acid was below method DL in all except

QAPP Worksheet #10: Conceptual Site Model, Continued

two samples, Wipe Sample #1 with 14.1 microgram (µg)/wipe area (9-inch square) and Wipe Sample #6 with 34.4 µg/wipe area of picric acid were found. The concentrations of pesticides were below DL on the wipe samples from the bunkers within the Ammonium Picrate Area.

The M&E investigation concluded:

- Groundwater: Nitrates in well MW-7 [560 microgram per liter (µg/L)] greatly exceeded concentrations in adjacent shallow well MW-6 (<50 µg/L). This may be a result of degradation of water-soluble picric acid with subsequent transport of nitrate to deeper in the aquifer. Lead detected in groundwater may be a result of the use of lead paint on structures in the area.
- Tank water: The presence of metals in Tank #2 in concentrations above Maximum Contaminant Levels (MCLs) and BTAG Fresh Water criteria may be due to less dilution than Tanks #1 and #3. Tank #2 contained less water and more debris than the other tanks. The M&E field team also noticed an odor of decay at Tank #2 that was not present in the other two tanks. Tank water was acidic relative to the acidity of groundwater, with pH ranging from 2.34 to 2.5. There were likely acid residues in the tanks.
- Soil: PAHs in Soil Sample #7 exceeded the NJ action level of 10,000 microgram per kilogram for total PAHs. No metals exceeded reference criteria, and all were within the US Soil Background ranges. M&E concluded that the high nitrite/nitrate concentrations in Soil Sample #7 (1.9 mg/kg) may be due to its location along the road between the Acid Area and the Ammonium Picrate Area adjacent to well MW-5 where spillage may have occurred. Elevated nitrate/nitrite was also observed in Soil Sample #11 collected near Tank #1.
- Wipes: Picric acid was detected in the wipe samples likely due to the cool and damp conditions of the bunker walls which did not encourage volatilization to occur.

The following recommendations were made for the Ammonium Picrate Area based on this investigation:

- Resample groundwater monitoring well MW-5 and collect filtered samples to verify metals concentrations.
- Pump out and remove the contents of the tanks.
- Dismantle the tanks, since acid may continue to leach from the inner walls.
- Decontaminate and secure the bunkers in this area.

Since the time of the M&E investigation, it appears that the acid tanks and bunkers have been removed at AOC 1. It is not known if any follow-up sampling was completed in this area.

Air photo analysis (AGC, 2019) notes an open storage area to the northeast of the Ammonium Picrate area, but no prior investigations have been conducted in this area (**Figure 10-3**).

10.3.2 AOC 3 – Former Landfill

Previous investigations conducted in this area were the D&M (1981) Site Investigation, M&E (1990) Preliminary Impact Evaluation, and Alion (2008) Final Site Inspection.

QAPP Worksheet #10: Conceptual Site Model, Continued

D&M (1981)

Two borings and one piezometer (D&M-11) were installed near the Former Landfill Area. No DNP or DNCB were detected in soil samples; however, picric acid was detected in one of the soil samples from the borings. DNCB was detected in the groundwater sample at a concentration of 0.0029 mg/L. No DNP was detected in the groundwater sample. The D&M data are summarized in **Tables 10-1** (groundwater) and **10-2** (soil). No current (November 2020) comparison criteria other than picric acid are available for the analytes detected in groundwater or soil. Note that picric acid was not detected in groundwater and did not exceed the soil RSL.

The 1981 D&M investigation concluded that some impacts of subsurface soils and groundwater occurred, and that the picric acid and any 2,4-DNP released to the environment would eventually degrade to nitrate.

M&E (1990)

One groundwater monitoring well (MW-8) was installed in the northwest area of the Radisson Community, north of the golf course parking lot and Potter Road (**Figure 10-4**). The well was installed to monitor shallow groundwater in the vicinity of the Former Landfill Area, and is set at a depth of 15 ft bgs.

The groundwater sample was analyzed for VOCs (Method 8240), SVOCs (Method 8270), DNCB (Method 8270), picric acid (USGS Method), nitrite/nitrate (Method 353.1), and total metals [arsenic (Method 3020/7060); barium, cadmium, chromium, lead (Method 3005/6010); mercury (Method 7470); selenium (Method 3020/7740); and, silver (Method 3005/6010)]. Results of analyses indicated that VOCs and SVOCs were below the 1987 NYS Groundwater Standards in the groundwater sample. Total metal concentrations were found to be below NYS Groundwater Standards for groundwater samples with the exception of lead. The maximum concentration of lead in groundwater at all AOCs during this investigation was found in the sample from monitoring well MW-8 at the Former Landfill, with a concentration of 0.196 mg/L. The concentration of total lead in the groundwater sample collected from the Former Landfill was thought to be a result of disposal of cans of lead-based paint in the landfill. Nitrates were detected in this groundwater sample, but were below the NYS Guidance Criteria. Concentrations of nitrates at this location may have been influenced by the fertilizer applied to the golf course (M&E, 1990). A comparison of the M&E groundwater data to current (November 2020) tapwater RSLs is provided in **Table 10-3**. Chromium and lead exceeded the current (November 2020) tapwater RSLs.

One soil sample (number 12) was collected at a depth of approximately six inches bgs, approximately 15 ft west of monitoring well MW-8, near the Former Landfill (**Figure 10-4**). The soil sample was analyzed for VOCs (Method 8240), SVOCs (Method 8270), DNCB (Method 8270), picric acid (USGS Method), nitrite/nitrate (Method 353.1), and total metals [arsenic (Method 3050); barium, cadmium, chromium, lead (Method 3050/6010); mercury (Method 7471); selenium (Method 3050/7740); and, silver (Method 3050/6010)]. VOCs and total metal concentrations in soil were within background ranges and below the NJDEP Cleanup levels. Nitrates were found in Soil Sample #12 at 1.3 mg/kg. Concentrations of nitrates at this location may have been influenced by the fertilizer applied to the golf course. A comparison of the M&E soil data to current (November 2020) residential Soil RSLs and Eco-SSLs is provided in **Table 10-4**. Benzo(a)pyrene in the sample collected at the Former Landfill exceeded the residential RSL. Arsenic in the

QAPP Worksheet #10: Conceptual Site Model, Continued

sample collected at the Former Landfill exceeded the current (November 2020) residential soil RSL; chromium exceeded the residential RSL; and lead exceeded Eco-SSLs.

Based on the results of this investigation, M&E recommended resampling groundwater monitoring well MW-8 and collection of filtered samples to verify metals concentrations.

Alion (2008)

Alion conducted a Site Inspection (SI) at the Former Landfill Area for the Military Munitions Response Program (MMRP) from 2007-2008, under a contract with USACE. The objective of the SI was to determine if the FUDS MMRP project (C02NY029003) warranted further response under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The scope of the SI was limited to an evaluation of munitions and explosives of concern (MEC) and munitions constituents (MC) associated with the historical use of the FUDS prior to property transfer (Alion, 2008).

Alion investigated two Munitions Response sites (MRS) at NYOW: the Former Landfill Area (MRS 1) and the Former Target Range (MRS 2, located adjacent to the Former Ammonium Picrate Area to the south and extending to Route 31) using observation and historical data. The Former Target Range was used during DoD occupation of the Site; this area is currently a residential area and part of the Radisson Community.

During the D&M (1981) and M&E (1990) investigations, a total of five groundwater samples and three soil borings were collected at MRS 1; no samples were collected at MRS 2. Picric acid, a breakdown product of ammonium picrate, was detected in low concentrations in two of the three subsurface soil borings. No picric acid was detected in groundwater. No additional field sampling was conducted during the SI (Alion, 2008).

Alion concluded that both MRSs were a low risk for MEC. MRS 1, which had been sampled under previous investigations, did not contain MC (e.g., picric acid) in concentrations above screening values in subsurface soil or groundwater. MRS 2, which had not been sampled during previous investigations, was redeveloped into residential and light industrial use over 40 years prior to the Alion SI. Based on site history and the SI findings, Alion concluded that the risk of encountering MEC was low. Both MRSs were rated No DoD Action Indicated (Alion, 2008).

10.3.3 AOC 4 – Bunker Area

Previous investigations conducted in this area were the M&E (1990) Preliminary Impact Evaluation and NYSDEC (1988-1989) Site Characterization, NYSDEC (1993) Operation and Maintenance Plan for the Three Rivers Pesticide Storage Site, NYSDEC (1994) Bunker Reclassification, and NYSDEC (1998) Long Term Monitoring Sampling.

M&E (1990)

At the time of the M&E investigation, the 14 bunkers (**Figure 10-5**) remained intact. According to M&E, Bunkers #3 and #8 were used by NYSDEC personnel for the storage of pesticides for approximately ten years, from the early 1970s to 1983. A small partitioned room (approximately 12 ft by 22 ft) within Bunker #3 was used to receive and package pesticides from citizens and/or businesses that had pesticides that

QAPP Worksheet #10: Conceptual Site Model, Continued

were either unwanted or had been banned. Bunker #8 was used for receiving and repackaging pesticides, but was also used for pesticide storage (NYSDEC, 1993). Bunkers #3 and #8 are no longer in use. The remaining bunkers were being used at the time by game management personnel for equipment and vehicle storage.

Two wipe samples were collected from Bunker #8 to determine if residual contamination from NYOW operations and/or pesticide storage existed. Concentrations of several pesticides were detected in Bunker #8.

M&E recommended that Bunker #8 be secured and decontaminated. The decontamination of Bunkers #3 and #8 was later completed by NYSDEC (described below). Bunkers #3 and #8 are not eligible for further investigation under FUDS because NYSDEC stored pesticides in the bunkers after DoD transferred the property, and remediation was initiated by NYSDEC.

NYSDEC Studies (1988-1998)

Potential pesticide impacts to environmental media in the vicinity of Bunkers #3 and #8 represent a background condition due to NYSDEC use and storage of pesticides, but do not represent a DoD responsibility.

NYSDEC conducted studies at AOC 4 as follow up to their storage of pesticides in Bunkers #3 and #8. A Site Characterization Study was performed at the Three Rivers Facility by Blasland & Bouck Engineers for NYSDEC, from 1988-1989. The field activities included soil sampling and porous interior building material sampling at Bunkers #3 and #8, along with groundwater sampling. The report concluded that groundwater remediation was not required because the “analytical results indicate that groundwater has not been impacted by past operation of the pesticide storage facility” (NYSDEC, 1990).

In 1993, NYSDEC published an Operation and Maintenance Plan for the Three Rivers Pesticide Storage Site (NYSDEC, 1993). In this document, they discussed the remediation activities conducted by OBG Technical Services (OBG) in 1992, to address Bunkers #3 and #8. OBG plugged the interior drainage holes and trenches with grout and pressure washed the interior walls. They collected wipe samples and analyzed the samples for pesticides. No pesticides were detected on the wipe samples collected from Bunker #3. The samples from Bunker #8 contained butyl benzyl phthalate (93.6 parts per billion, ppb), 4,4’-dichlorodiphenyl dichloroethylene (16.6 ppb), 4,4’-dichlorodiphenyldichloroethane ([323 ppb),] and 4,4’-dichlorodiphenyltrichloroethane (DDT) (461 ppb). OBG sealed all concrete surfaces with polyurethane and removed and disposed of 2.5 ft of soil in the “full width and length of the gravel driveways [aprons] in front of each bunker” (NYSDEC, 1993).

Analysis of confirmation samples collected from the excavation at Bunker #3 indicated the presence of 2,4-dichlorophenoxyacetic acid and 2,4,5-trichlorophenoxyacetic acid (2,4,5-T) at concentrations of 0.028 mg/kg and 0.186 mg/kg, respectively (NYSDEC, 1993). Concentrations of 2,4,5-T were 0.010 mg/kg in the confirmation soil sample from Bunker #8. In addition to the pesticides, PAHs were detected in the confirmatory samples. These concentrations were attributed to the asphalt in the access ramps that were removed.

QAPP Worksheet #10: Conceptual Site Model, Continued

The excavations and bunkers were backfilled with clean fill and covered by a 12-inch low permeability layer, geocomposite liner, and geogrid. Two additional feet of clean fill and one foot of topsoil were then placed over the geogrid. The areas were then seeded and mulched.

In 1994, NYSDEC reclassified these bunkers from Class 3 (contamination does not constitute a threat to public health or the environment) to Class 4 (site has been properly closed but requires continued management). This change occurred because the bunkers had been emptied, cleaned, and covered with an impermeable cap. NYSDEC currently maintains responsibility for long-term management of Bunkers #3 and #8, such as lawn mowing and cap inspection.

Only one round of long-term monitoring data (31 March 1998) was available in the records obtained from a Freedom of Information Law (FOIL) request submitted to NYSDEC. Three groundwater monitoring wells were sampled (from NYSDEC wells MW-1B through MW-3) and analyzed for VOCs, pesticides, and SVOCs (Columbia Analytical Services, 1998). One VOC (trichloroethene) was detected at 0.0059 mg/L in the sample from MW-3, above the MCL of 0.005 mg/L (it should be noted that DoD activities would not have involved trichloroethene). Di-n-butylphthalate and bis(2-ethylhexyl) phthalate were detected at estimated (J-qualified, just above the quantitation limit) concentrations in the groundwater samples from wells RH797 MW-1 and RH797 MW-3. Pesticides were not detected in these three groundwater samples.

AOC 5 – Power Plant Area

As discussed earlier, there were no previous investigations at AOC 5. However, AOC 5 will be assessed as part of this study.

10.3.4 Recent Site Visits

The site visit team (consisting of representatives from Bluestone and USACE, New England District, referred hereafter as the team) met with Mr. Joshua Cook (NYSDEC Division of Environmental Remediation) at Three Rivers WMA on the morning of 27 June 2018. A second reconnaissance was made on 14 August 2019, following the Technical Project Planning session held 13 August 2019 for new team members to view the Site. The following notes are from the June 2018 visit.

The team observed the 12 existing and two former storage bunkers located along West and East Igloo Roads. Originally five bunkers were located on West Igloo Road and seven were located on East Igloo Road. According to Mr. Cook, Bunkers #3 and #8 were used by NYSDEC for pesticide storage. These two bunkers were decontaminated and capped by NYSDEC. They are now covered with vegetation but are mowed periodically to discourage the rooting of trees and shrubs.

Based on visual observation, half of the twelve remaining bunkers (including Bunkers 1, 2, 5, 7, 11, 12) are still in active use by NYSDEC/Three Rivers WMA for equipment and vehicle storage. The team visited each of the bunkers. All the bunkers have similar construction/ configurations, with two French drains inside the bunkers that drain toward the front of the bunker (with drainpipes exiting out the front façade). Each bunker also has an apron (some consisting of gravel, but some with only soil) and soil/vegetation ramping from the ground surface to the roof on the three remaining sides. The condition of the garage doors and siding on the front façade varied from bunker to bunker.

QAPP Worksheet #10: Conceptual Site Model, Continued

Bunker #9 had an adjacent recreational structure (cabin and covered picnic area, between Bunkers #8 and #9), which according to NYSDEC site personnel, was utilized by local law enforcement during their brief use of the area as a small arms/pistol shooting range (post DoD ownership). This area is no longer used as a shooting range, and the recreational structure has been abandoned.

Bunker #12 had an exterior tank vent. According to maintenance personnel, there is a fuel tank inside the bunker that is used for a tractor.

There were drainage swales that ran in front of most of the bunkers, along West and East Igloo Roads, which were heavily vegetated. One monitoring well was located approximately 3 ft off East Igloo Road across from Bunker #8. Another monitoring well is suspected between Bunker #7 and #8 but was not found during the site visit.

The group traveled to the Former Ammonium Picrate Area. This portion of the Site has been developed for residential use. Most of the historical structures in this area no longer exist, presumably removed/demolished during redevelopment of this area by the Radisson Community. The team did observe one of the Former Ammonium Picrate ramps in a wooded area adjacent to a walking trail in the Radisson Community. A large concrete structure was also observed at the top of the hill, near the walking trail. These items are the few remaining historical structures in this AOC.

The team and Mr. Cook then traveled to the Radisson Community Golf Course. The Former Landfill Area has been covered by an asphalt parking lot for the golf course. Monitoring well #8 was suspected in this area but was not located during the site visit. The tour concluded at the Former Landfill Area. The tour did not include a walk-through at the Former Power Plant Area as it had not yet been designated as an AOC at the time of the site visit.

Additional observations made during the 14 August 2019 visit include:

- AOC 1: The team walked into the wooded area along the ridge and observed some of the remnant structures remaining (concrete foundations, supports for tanks, derelict structures) and some evidence of trespasser activity based on items left in the woods (pizza box, milk jug, bolt cutters).
- AOC 2: The team walked AOC, including the area of the former water tank at the southeast corner of the property.
- AOC 3: No new observations at the Former Landfill.
- AOC 4: The team met with Mr. Cook and Ms. Bonnie Parton (NYSDEC Wildlife Technician), who expressed concern about minimizing disturbance of sensitive ecological habitats by site investigations.

AOC 5: This AOC had not been identified at the time of the 14 August 2019 visit; so was not visited.

10.4 OPERATIONAL HISTORY AND ENVIRONMENTAL AREAS OF CONCERN

The Site was acquired to establish a facility for the manufacture of ammonium picrate during World War II. Ammonium picrate is a stable, high explosive compound. The facility was designed to produce 60,000 pounds of ammonium picrate per 24-hour day (Reconstruction Finance Corporation , undated). The facility consisted of an Administration Area, Power Plant Area, Ammonium Picrate Area, Acid Area, Bunker

QAPP Worksheet #10: Conceptual Site Model, Continued

Area, and Landfill Area over approximately 2,100 acres of the 6,800-acre property; the remainder was farmland.

10.4.1 Process-Related Chemicals

Chemicals used or produced on-site during operations included DNCB, DNP, picric acid (2,4,6-trinitrophenol), sulfuric acid, nitric acid, sodium hydroxide, aqueous ammonia, and ammonium picrate (M&E, 1990). DNP was formed in the first step of the ammonium picrate process, where DNCB (the raw material) was converted to DNP via hydrolysis (D&M, 1981). The second step was nitration of DNP to create picric acid. The third step involved the addition of ammonia to form ammonium picrate. Low levels of DNCB were detected in historical (1981) groundwater samples. DNP was analyzed during the D&M study, listed as combined 2,4- and 2,6-DNP, but quantified as 2,4-DNP. No DNP was reported in the historical groundwater samples. Neither DNCB nor DNP were detected in samples collected from the historical soil borings.

The 2,4-isomer of DNP is included in the standard SVOC list (EPA Method 8270D); however, the 2,6-isomer is not routinely analyzed by laboratories. According to the D&M Report, 2,6-DNP is "virtually inseparable from its 2,4-isomer" (D&M, 1981). Therefore, DNP will be assessed as 2,4-DNP as a historical site-specific chemical.

Picric acid was detected at low concentrations, below current (November 2020) EPA Residential Soil RSLs, in subsurface soil. Picric acid was detected above the current (November 2020) EPA Tapwater RSL in a few historical groundwater samples collected from monitoring wells in the Former Ammonium Picrate Area and Former Administration Area. DNCB and 2,4-DNP (as SVOCs) and picric acid (on the explosives list) will be assessed as DoD-related chemicals.

The other process-related chemicals such as sulfuric acid, nitric acid, and sodium hydroxide are readily dissolved in water; thus, will not be assessed as DoD-related chemicals. Nitrate/nitrite will be assessed as potential breakdown products, but are commonly found in fertilizers applied at golf courses. Therefore, the presence of nitrate/nitrite at AOC 3 (Former Landfill), adjacent to the Radisson Golf Course, may not be directly attributed to historical DoD use. Therefore, the background levels of nitrate/nitrite associated with the Golf Course will be assessed.

10.4.2 Metals

Historical concentrations of DoD-related metals (specifically arsenic, chromium, and lead) exceed current (November 2020) Residential and Industrial Soil RSLs and Eco-SSLs, but fall within the range of background concentrations observed during the New York State Rural Surface Soil Survey (NYSDEC, 2005). Arsenic, chromium, and lead were also observed in groundwater at concentrations exceeding current (November 2020) Federal MCLs and/or Tapwater RSLs in samples from monitoring wells in the Ammonium Picrate Area (MW-5), Acid Area and Former Landfill Area (MW-8).

The chromium in soil and groundwater may have originated from the stainless-steel equipment used during NYOW operations (M&E, 1990). Arsenic may be present due to use in metallic alloys. The lead is likely present due to historical use in paints (M&E, 1990). The metals detected in groundwater and soil were also found in tank water, most likely leached from the process equipment or tank walls by the acidic

QAPP Worksheet #10: Conceptual Site Model, Continued

water. Therefore, select target analyte list (TAL) metals (EPA Method 6010/6020) including arsenic, chromium, and lead, will be assessed as DoD-related chemicals.

10.4.3 Polycyclic Aromatic Hydrocarbons

PAHs detected in soil samples collected at the Ammonium Picrate Area (Soil Sample #7) exceed current (November 2020) EPA Residential Soil RSLs and Eco-SSLs. However, most of the detected values fall within the range of concentrations observed during the New York State Rural Surface Soil Survey (NYSDEC, 2005). Fluoranthene was detected just above the maximum background value in one sample (Soil Sample #7). PAHs are ubiquitous in the environment and were not identified in any of the historical documents as chemicals used by the DoD at the former NYOW. Therefore, PAHs are not likely related to DoD activities outside of areas where aboveground storage tanks or underground storage tanks were/are located. They may, however, be present in the Former Power Plant Area due to coal storage and various processes conducted at that AOC.

10.4.4 Summary of Concerns by AOC

- AOC 1 – Former Ammonium Picrate Area: As noted above, the chemicals used or produced on-site during operations included DNCB, DNP, picric acid, sulfuric acid, nitric acid, sodium hydroxide, aqueous ammonia, and ammonium picrate (M&E, 1990). Potential releases may have resulted due to spills, leaks, or possible mishandling and improper disposal of process chemicals associated with historical operations at former magazines, change and dryer houses, laboratory and ammonium picrate buildings. Possible ongoing impacts could result from residual impacts leaching from the subsurface soils to groundwater. Much of the Former Ammonium Picrate Area has been redeveloped, and reworking of soils has occurred during the construction activities. Thus, soil concentrations may not be representative of former conditions.

- AOC 3 – Former Landfill Area: Based on site history, the waste may contain household and office trash, paint cans, stainless-steel "spiders" (e.g., equipment used to mix the ammonium picrate), and other potential manufacturing items from the operations at NYOW (D&M, 1981). There is no record indicating that the landfill was lined prior to use. The parking lot was not designed as a landfill cap; therefore, although the former landfill is currently covered by an asphalt parking lot, it is not clear if the parking lot covers the entire landfill or if there are cracks in the surface that would allow precipitation to leach to the landfill. Potential releases may have resulted from the disposal of waste materials from the NYOW operations. Air photo analysis (AGC, 2019) suggest that disposal may have extended north of the parking lot, almost to the edge of the surface drainage feature. Depth to water measured by D&M (1981) was 9.7 ft bgs, and by M&E (1990) was 9.75 ft bgs. Possible ongoing impacts could result from the buried waste materials leaching into subsurface soil and groundwater, transport of impacted groundwater to nearby surface water, vapor intrusion into the nearby golf clubhouse, or direct contact between waste materials and groundwater.

  Based on aerial photographs (AGC, 2019), a former open storage area is located approximately 500 feet west of the former landfill. This area was likely used by DoD to stage equipment associated with the former landfill. No further records concerning the use of the open storage

QAPP Worksheet #10: Conceptual Site Model, Continued

area were identified. However, possible impacts may have occurred associated with possible vehicle use and temporary waste storage. No environmental assessment has occurred at the former open storage area to date.

- AOC 4 – Bunker Area: DoD previously used the former bunkers for the storage of finished product (ammonium picrate powder). Potential releases may have resulted due to discharge from the interior French drains toward the drainage swales at each of the bunkers. Spills of chemicals may also have occurred in the aprons, resulting in runoff into the drainage swales. Residual impacts in surface or subsurface soil may be leaching to groundwater.

  Due to the similarity of the plant operations at the Former Ammonium Picrate Area (AOC 1), and Former Bunker Area (AOC 4), many of the same DoD-related chemicals are suspected within both of these areas.

- AOC 5 – Power Plant Area: This area contained a boiler house, electric substation, a coal pile, three concrete coal aggregate bins (bins A, B, and C) along the rail spur, and a possible dumping area. The residuals from the coal pile may have resulted in constituents such as metals, VOCs, SVOCs, and PAHs in soil and groundwater. The electric substation could have released the same chemicals as the coal pile plus PCBs, which were invented in 1929 and were used in transformers from some time after that until they were banned in 1979. PCBs may also have been part of the waste that was disposed of in a possible dump north of the coal pile. The following DoD-related constituents were identified based on an initial comparison of historical data with current EPA criteria (i.e., industrial soil RSLs (November 2020), tapwater RSLs (November 2020), Eco-SSLs (various dates), MCLs (November 2020), and freshwater benchmarks).

Media	Historical Site-Specific Chemical			
	D&M (1981)	**M&E (1990)**	**NYSDEC (1991)**	**OBG (1993)**
Surface Soil	Picric Acid, but below comparison criteria (AOC 1) No surface soil collected at AOC 3	Arsenic, chromium, and lead above RSLs and/or Eco-SSLs (AOCs 1 and 3) PAHs above RSLs (AOC 1)	Seven PAHs above Eco-SSLs and benz(a)anthracene and naphthalene also above RSLs (AOC 4 – confirmation sampling at Bunkers #3 and 8, after remediation by NYSDEC)	None – not sampled

QAPP Worksheet #10: Conceptual Site Model, Continued

Media	Historical Site-Specific Chemical			
	D&M (1981)	**M&E (1990)**	**NYSDEC (1991)**	**OBG (1993)**
Groundwater	DNCB, but no available comparison criteria (AOCs 1 to 3) Picric acid (above tapwater RSL at AOC 1)	Arsenic just above MCL (AOC 1) Chromium and lead above MCL/action level and/or tapwater RSLs (AOCs 1 and 3)	None – not sampled	None – not sampled
Tank Water	None – not sampled	Lead above EPA Groundwater (GW) action level and BTAG freshwater criteria (AOC 1) Arsenic, cadmium, and chromium above MCLs (AOC 1, Tank #2) Barium and phenol above freshwater criteria (AOC 1, Tank #2) Nitrate/Nitrite above freshwater criteria (AOC 1)	None – not sampled	None – not sampled
Subsurface Soil	Picric Acid, but below comparison criteria (AOCs 1 and 3)	None – not sampled	None – not sampled	None – not sampled
Surface Water	None – not sampled	None – all below laboratory DL	None – not sampled	None – not sampled

QAPP Worksheet #10: Conceptual Site Model, Continued

Media	Historical Site-Specific Chemical			
	D&M (1981)	**M&E (1990)**	**NYSDEC (1991)**	**OBG (1993)**
Sediment	None – not sampled	None – not sampled	None – not sampled	None – not sampled
Debris	None – not sampled	None – not sampled	None – not sampled	None – not sampled in FUDS-eligible area

10.5 CONSTITUENT FATE AND TRANSPORT

Potential release mechanisms for the DoD-related chemicals include:

- Primary:
    - Disposal and adsorption resulting from direct releases to the ground (including spills and leaks from tanks), dumping activities, and disturbance during redevelopment and construction activities, burial of landfill wastes, including direct contact between landfill waste and groundwater in AOC 3.
- Secondary:
    - Wind erosion
    - Infiltration/leaching to subsurface soils and groundwater
    - Runoff/erosion
- Tertiary:
    - Groundwater flow and discharge

The potential release mechanisms associated with each AOC are summarized in **Figures 10-11a through d and 10-12a through d**.

There are no documented spills in the historical records for the Site. However, in the absence of a documented release, it is assumed that constituents may have spilled or leaked onto the ground from process equipment during the ammonium picrate operations at NYOW. Based on the D&M investigation, the soils in the Radisson Community have relatively low permeability and groundwater velocities are very low (D&M, 1981). D&M concluded that ammonium picrate released to the environment would be completely dissolved in the groundwater and would be contained within 160 ft of where it was released (D&M, 1981), based on their analysis. Groundwater samples collected near the residential community contained no detectable concentrations of process-related chemicals. The presence of picric acid in soil was presumed to be from fugitive dust blown across the Site during manufacturing. While picric acid is highly soluble and would be mobile in groundwater if present, due to the relative absence of picric acid in groundwater, D&M did not anticipate that it was being transported via groundwater (D&M, 1981).

QAPP Worksheet #10: Conceptual Site Model, Continued

Wash water and acid reprocessing waste streams were released to wastewater ditches that ran north from AOC 1. These wastes would have been highly diluted by precipitation (D&M, 1981).

10.5.1 Constituent Persistence

When released to the environment, metals (such as arsenic, chromium, and lead) tend to sorb to soil, sediment, and other organic materials. Arsenic, chromium, and lead are all found naturally in the earth's crust. Arsenic occurs most often as a compound with sulfide and other minerals [Hazardous Substances Data Bank (HSDB), 2009]. Lead is typically transformed to organic complexes in the environment (HSDB, 2016b).

Chromium generally occurs in the Cr (III) (chromic) oxidation state as the Cr (III) cation, and in the (Cr (VI) oxidation state as CrO_4^{2-} (chromate). Soil conditions generally favor the Cr (III) form, a very immobile cation that complexes strongly with organic matter and sorbs on oxides and silicate clays, even at quite low pH (McBride, 1994). Cr (III) readily substitutes for iron in mineral structures, and precipitates as insoluble chromium hydroxide ($Cr(OH)_3$) at higher pH. The chromic form is, therefore, very immobile in most soil. D&M indicated that site soils were generally neutral pH.

At higher pH, a small fraction of the Cr (III) in soil can be oxidized to Cr (VI) (chromate), a very toxic form of chromium. This oxidation is promoted by manganese oxides. Chromate adsorbs less strongly than Cr (III), and the mobility and bioavailability of this anion is consequently higher. Generally, however, if pollutants containing chromate are applied to soil, most or all of the chromate is spontaneously reduced to Cr (III), especially under acid conditions and with organic matter present. Organic matter supplies reducing agents and complexing groups, stabilizing the chromic form. The soil therefore has the ability to detoxify chromate and immobilize the element.

Chromium is rated as an immobile element, most of which is difficult to extract from soil even by aggressive chemical agents. Toxicity of Cr to plants is occasionally seen in unusually Cr-rich soil formed from the parent rock, serpentinite, or under high pH conditions favorable to Cr (III) oxidation.

Picric acid is likely to exist in anion form in the environment; therefore, it is not expected to adsorb strongly to soils containing organic carbon and clay. Picric acid is resistant to aerobic biodegradation but can be degraded via photolysis by sunlight and hydrolysis in water (HSDB, 2011b). 2,4-DNP behaves similarly in the environment to picric acid (HSDB, 2011a). In contrast, DNCB has strong adsorption to clay, and adsorbs to suspended solids in water. DNCB is slow to biodegrade in water and soil and slow to volatilize from water, but is susceptible to direct photolysis in sunlight (HSDB, 2013).

10.5.2 Constituent Migration

The soils at AOCs 1 and 3 have been extensively re-worked during the development of the Radisson Community, potentially resulting in redistribution of soil horizons and any DoD-related constituents associated with them.

Due to the chemical properties of the DoD-related chemicals and time elapsed since potential releases of these DoD-related chemicals, dispersion, diffusion, and volatilization would not be considered significant contributors to constituent migration.

QAPP Worksheet #10: Conceptual Site Model, Continued

Most of the metals observed in the soil at the former NYOW would be bound to soil particles but could migrate via advective transport (bulk movement) or desorption (leaching) with the flow of surface water or groundwater toward adjacent water bodies. Desorption would be dependent on the solubility of the individual chemical and the properties of the soil. Arsenic is more mobile in soil at high pH (HSDB, 2009). In soil, chromium III has low solubility and low mobility (HSDB, 2016a). Lead compounds have limited mobility when released or deposited on soil (HSDB, 2016b).

Whereas there is some conflicting information about solubility of picric acid in water and mobility in soil, most references show that picric acid is highly soluble, on the order of grams per liter (PubChem, 2020; EPA, 2015). HDSB (2018) indicates that picric acid has an estimated carbon-water partition coefficient (Koc) value of 2,250 liters per kilogram, which indicates slight mobility in soil. United States Army Center for Health Promotion and Preventive Medicine (USACHPPM, 2005) provides a much lower Koc estimate of 130 liters per kilogram, indicating moderate to high mobility in soil. Based on this information, it will be assumed that picric acid is highly soluble and mobile in soil.

Koc values of 2,4-DNP have been reported as 13.5 and 16.6, which indicates high mobility (HSDB, 2011), and it is highly soluble in water. The Koc of DNCB is estimated at 575 (HSDB, 2012), indicating moderate mobility. DNCB is very poorly soluble in water.

10.6 POTENTIAL RECEPTORS AND EXPOSURE PATHWAYS

As defined in Engineer Manual 200-1-12 - Environmental Quality, Conceptual Site Models (USACE, 2012), a CSM describes sources of impacts, as well as complete, potentially complete, or incomplete human and ecological exposure pathways; current, determined, or reasonably anticipated future use of property; and, human and ecological potential receptors (USACE, 2012). A CSM is an iterative planning and communication tool that provides a structure to summarize and display information and to identify additional information needed to develop technically sound decisions. Preliminary CSMs for this Site are presented as **Figure 10-11a through d** (for human receptors) and **Figure 10-12a through d** (for ecological receptors).

Conversations with developers and the community and an ecological resource and receptor inventory to be conducted during Phase 1 may result in the identification of additional resources and receptors potentially impacted by site constituents. This information will be used to update the CSM after the Phase 1 studies are complete.

Analytical chemistry data for Human Health and Ecological Risk Assessment will be collected during Phase 2 based on the results of Phase 1.

10.6.1 Current and Future Site Use and Ecological Settings

The current and future land uses and ecological settings for the four AOCs are provided below. These assumptions are carried throughout the QAPP.

- AOC 1 – Ammonium Picrate Area: The Ammonium Picrate Area is within a primarily residential area with some undeveloped land with maintained paved walkways and recreational grassy areas. Homes are not built on areas of potential impacts; however, paved walking paths and grassy areas

QAPP Worksheet #10: Conceptual Site Model, Continued

wind between homes and wooded areas containing the old loading ramps, storage bunkers, and loading dock structures. Old concrete structures in disrepair exist in wooded areas of varying density. Terrestrial habitats, including maintained grassy areas and woodlands (eastern deciduous forest), are present. A large forested wetland is located west of AOC 1. Final receptors for this area will be refined following the initial site investigation.

- AOC 3 – Landfill Area: The landfill is currently a paved parking lot for a golf course, and the former open storage area straddles one of the golf course fairways, with wooded areas to the north and south. At the landfill, a grassy strip with light poles extends along the middle and grassy landscaped areas surround the parking lot. The grassy center strip will not be evaluated because it is likely reworked and above the landfill cap. Surface soils of potential concern are limited to the perimeter of the parking lot due to pavement. The area is paved so no surficial ecological habitat is present. If subsurface suitable soil habitat is found within the 1-5 ft interval, select terrestrial ecological receptors will be evaluated. The portion of the former open storage area that is located on what is the existing fairway will not be evaluated; however, the portions of the former open storage area that are covered by woody vegetation will be evaluated for terrestrial ecological receptors. Lastly, there is a small stream north of the landfill that may be receiving runoff or groundwater recharge that will be evaluated for impacts to aquatic organisms. Final receptors for this area will be refined following initial site investigation.

- AOC 4 Bunker Area: Currently located within Three Rivers WMA. NYSDEC is using several of the remaining bunkers for equipment and vehicle storage. The terrestrial habitat consists primarily of grassy fields and northern deciduous forest. Several small forested and emergent wetlands are located along the southeastern border of the Bunker Area. Future use of this area is likely to remain wildlife management. Final receptors for this area will be refined following initial site investigation.

- AOC 5 – Power Plant Area: Currently a mostly wooded area west of a residential area. Terrestrial habitats, primarily woodlands (eastern deciduous forest), are present. Future use at this AOC is unknown at this time because it was not identified as an AOC until recently; therefore, has not yet been investigated. Review of the Radisson Community General Project Plan (1971) and recent amendments through November 2012, indicates potential future use of AOC 5 as commercial/ retail. Final receptors for this area will be refined following the initial site investigation.

Aquatic and benthic receptors may be evaluated for any of the AOCs if the phased groundwater assessment indicates potential discharge of site-related groundwater into nearby wetlands or surface water bodies.

Based on conversations with Mr. Joshua Cook (NYSDEC Division of Environmental Remediation) and Ms. Bonnie Parton (NYSDEC Wildlife Technician) during the August 2019 site visit, land use is expected to remain unchanged in the future at AOC 4. AOCs 1 and 5 could be developed for residential purposes in the future and AOCs 2 and 3 could possibly be reused for residential purposes in the future unless an enforceable institutional control were placed on the areas. During an inspection of AOC 5 on 02 October 2020, NYSDEC noted stakes, flagging, and survey pins which appeared to have been placed recently. The

QAPP Worksheet #10: Conceptual Site Model, Continued

Department inquired about the survey with Empire State Development (ESD) and ESD informed the Department that they are considering marketing and selling the area along Willett Parkway.

Current residential properties are located within AOC 1 and some apartment buildings and residences in the Radisson Community appear to be constructed on the sites of former dryer houses, change houses, and other historical site features. Future residential development at AOC 1 is not currently expected in potentially impacted areas. However, as part of the first phase of the study, Bluestone is reaching out to the developers and community to determine foreseeable future development plans and aid in refining the human health receptors for Phase 2. The resource and receptor inventory along with the data regarding groundwater discharge will aid in refining the ecological receptors for Phase 2.

10.6.2 Potential Human and Ecological Exposure Pathways

The following preliminary identification of human and ecological exposure pathways is based on historical data. A re-assessment of potential human and ecological exposure pathways will be needed after completion of the data collection and analysis. **Figures 10-11a through d and 10-12a through d** summarize the potential human health and ecological receptors and exposure routes.

10.6.3 Potential Human Health Receptors and Exposure Routes

Below is a brief summary of the currently identified potential receptors and exposure routes. All HHRA will be performed during Phase 2, if needed based on Phase 1 exceedances of residential risk-based screening levels and background levels. These receptors will be re-visited and further refined in Phase 2. Based on Office of Solid Waste and Emergency Response (OSWER) guidance (EPA, OSWER Directive 9355.7, 2010) and DERP-FUDS guidance (DERP management guidance, 4715.20 4.b.(5)(a)3.b.), receptors evaluated will be limited to current receptors and those associated with reasonably foreseeable future site uses. As noted above, future residential development at AOC 1 is not currently expected in potentially impacted areas. However, as part of Phase 1, Bluestone is reaching out to the developers and community to determine foreseeable future development plans and aid in refining the receptors for Phase 2. If future development is indicated, future residents, future commercial/industrial workers, future construction workers, and/or future utility workers may be considered at AOC 1.

<u>Nearby Resident Using Groundwater as Drinking Water (AOCs 1, 3, 4, and 5)</u>

A nearby resident using a domestic well for potable water could be exposed to groundwater from the Site. Exposure pathways would include ingestion of groundwater and dermal contact with groundwater (while showering or bathing), inhalation while showering, and vapor intrusion.

<u>On-site Recreational Visitor (AOC 1 and AOC 5)</u>

An on-site recreational visitor could be exposed to surface soil while visiting the Ammonium Picrate Area (AOC 1) and Power Plant Area (AOC 5). It is assumed that exposure is occurring now at AOC 1 and will continue to occur in the future at AOC 1 (no changes to site use). Soil exposure pathways would include incidental ingestion, dermal contact, and inhalation of dust emissions released from soil.

QAPP Worksheet #10: Conceptual Site Model, Continued

Utility Worker (AOC 3)

Underground utilities are common in residential and commercial areas, and often are located around roads and the areas between the roads and buildings. A utility worker could be exposed on a short-term basis to surface and subsurface soil at areas surrounding the former landfill at AOC 3. Exposure routes could include incidental soil ingestion, dermal contact with soil, and inhalation of dust emissions released from soil.

On-site Adolescent Trespasser (AOC 1)

Adolescent Trespassers in the wooded areas of the Ammonium Picrate Area (AOC 1) could encounter site surface soil as part of their typical outdoor activities. Soil exposure pathways could include incidental ingestion, dermal contact, and inhalation of dust emissions released from soil.

Outdoor Maintenance Worker (AOCs 1, 3, and 4)

An outdoor maintenance worker, representing WMA workers, could encounter site surface soils as part of their typical outdoor maintenance or landscaping activities. WMA employees are assumed to spend the majority of their 8-hour workday outdoors with exposure to DoD-related chemicals through incidental ingestion of surface soils, dermal contact with soils, and inhalation of airborne DoD-related chemicals because of wind erosion. Maintenance workers at AOC 4 would be visiting grassy areas between bunkers as well as the apron areas, thus their area of activity is greater than the expected impacted area. Those grassy areas are not likely to have any constituents of concern, so no sampling is planned there.

10.6.4 Potential Ecological Receptors and Exposure Routes

Direct exposure to surface soil in AOC 1, AOC 4, and AOC 5 will be evaluated for the vegetative community and soil infaunal invertebrate community using soil screening values. Indirect exposure to bioaccumulative constituents in surface soil will also be evaluated using soil screening values based on food chain modeling for representative avian and/or mammalian receptors.

Subsurface soils (1 to 5 ft bgs interval) will be compared to appropriate ecological benchmarks as part of the site characterization process, provided suitable soil habitat conditions are present. The results of this evaluation will be used to determine if additional sampling and subsequent ecological risk assessments are warranted.

As part of a phased groundwater assessment, surface water, sediment and pore water samples may be collected in wetland areas near AOCs 1 and 4. In addition, if DoD-related chemicals are migrating from groundwater into the unnamed stream north of the landfill in AOC 3, pore water, surface water, and sediment may be collected. If collected, surface water and pore water samples will be compared to appropriate aquatic life benchmarks and sediment samples will be compared to appropriate benthic life benchmarks.

Ecological risk assessment will be performed during Phase 2, If needed based on Phase 1 exceedances of ecologically-based screening levels and background levels.

QAPP Worksheet #10: Conceptual Site Model, Continued

10.7 DATA GAP ANALYSIS

Data gaps exist in the information needed to adequately characterize if DoD-related constituents are present in environmental media; therefore, the first (Phase 1) collection of environmental media will be discrete samples for the purpose of completing the initial site investigation phase to assess if there are any CERCLA constituents present and potentially due to historic DoD activities, as well as, present above risk-based screening levels and background concentrations. If impacts from DoD-related constituents are encountered, then additional fieldwork will be completed as a second, separate field event to be addressed in an addendum (Phase 2) to collect data for an RI and to conduct human health and ecological risk assessments. Depending upon the AOC and potential exposure pathways, the analytical chemistry data gaps generally fall into the following categories (not all inclusive):

- Data non-existent;
- Data too old to represent current Site conditions;
- Site-specific constituents not accounted for in analyses;
- Data insufficient in quantity and/or spatial coverage to support decision-making; and
- Data from biased sampling approaches only; and therefore, not ideal for the determination of risk.

Groundwater and soil data as well as a resource and receptor inventory are the primary focus for the Phase 1 proposed field activities at the former NYOW. Residual soil impacts within the four AOCs (Ammonium Picrate Area, Former Landfill, Bunker Area, and Power Plant Area) may be serving as a continuing source to groundwater and could pose risks to human health and ecological receptors.

10.7.1 Summary of Previous Investigations

The most comprehensive environmental investigations conducted at the former NYOW were completed by D&M in 1981 and M&E in 1990. D&M collected 20 subsurface soil samples, 18 groundwater samples, and three surface water samples from wastewater ditches at the Site and analyzed the samples for 2,4,6-trinitrophenol (picric acid), 2,4-DNP, 2,6-DNP, and 2,4-DNCB. Based on a comparison to current (November 2020, THQ=1.0) criteria, the concentrations of picric acid in soil are below current RSLs. Groundwater samples AMS-1 (Administrative Area) and D&M-10 (AOC 1), both collected on 26 March 1981, had concentrations above the picric acid tapwater RSL.

M&E collected eight groundwater samples, 12 surface soil samples, six surface water samples from wastewater ditches, seven tank liquid samples, and 10 wipe samples and analyzed for VOCs, SVOCs, total metals, nitrate, picric acid, DNCB, and pesticides/PCBs (wipes only). In groundwater, chromium and lead exceeded NYS Groundwater Standards (based on 1987 criteria); whereas chromium, lead, and arsenic exceed current (November 2020) tapwater RSLs. In soil, nitrates were detected below 1987 and current (November 2020) criteria at all locations; the action level for total SVOCs was exceeded in one sample collected from the Ammonium Picrate Area. Arsenic, chromium, and lead concentrations exceed at least one of the current comparison criteria (Residential RSLs and Industrial RSLs from November 2020; or Eco-SSLs, current as of April 2020) in most of the soil samples. Due to the significant amount of redevelopment and earth moving within the Radisson Community, surface soil may not be representative of historical site conditions.

QAPP Worksheet #10: Conceptual Site Model, Continued

No compounds were detected in the M&E surface water samples collected from the wastewater ditches. Phenol was detected below reference criteria and arsenic, cadmium, chromium, and lead were detected above comparison criteria in the water sample from Tank #2.

AOC 5, the Former Power Plant Area, has never been investigated; therefore, it requires data to determine whether a release occurred.

10.7.2 Data Gaps

Limited sampling was conducted by D&M and M&E around the perimeter of the Former Landfill, because the area is covered by an asphalt parking lot. The disposal of trash and garbage, paint cans (including lead paints), office waste, and fly ash from the coal-burning boiler house potentially occurred at this landfill. At a minimum, additional groundwater sampling is needed to confirm whether the buried wastes are impacting groundwater quality in the vicinity of the Former Landfill. Per OSWER guidance (USEPA, 2010), there is no foreseeable change in land use of the Former Landfill; therefore, no future resident, future commercial/industrial worker, or future construction worker soil exposure is anticipated. However, an evaluation of current zoning and potential future land uses for the landfill area will be completed during the Phase 1 investigation and exposure scenarios based on reasonably anticipated future use will be evaluated during Phase 2. There is a potential utility worker exposure to subsurface soils around the perimeter of the Former Landfill, therefore additional subsurface soil sampling is required in that area.

As mentioned earlier, no sampling has been done at AOC 5, which has resulted in data gaps for surface soil, subsurface soil, and groundwater.

Supplemental sampling was conducted in support of the cleanup of Bunkers #3 and #8 at Three Rivers WMA and the acid tanks in the Radisson Community. The most recent groundwater sampling data available for the Former NYOW Site is very limited and over 20 years old. Groundwater sampling results for potential private drinking water wells are also unavailable. The lack of recent groundwater data is a data gap.

Subsurface soil data for the four AOCs are also limited. The D&M soil samples were collected from soil borings ranging from 2.5 ft to 25.5 ft bgs and grab samples were collected at the ground surface within the drainage ditches. However, D&M did not analyze the subsurface soil samples for metals or SVOCs. The M&E soil samples were analyzed for the site constituents of concern but were collected only six inches below the ground surface. No soil samples were collected at AOC 5. The lack of usable subsurface soil data is another data gap.

Overall, the following site-wide data gaps have been identified:

- Historical information indicates that there may be private wells in the area. A potable well survey will be conducted prior to the field work.
- Past historical information has not carried over to all of the SIs. Sampling and analyses for site-specific DoD-related chemicals is proposed.
- Background chemistry is not sufficiently characterized to support comparisons with site levels. New background sampling and analysis is proposed.
- The extent of the various surface geologic units is not precisely known at the AOCs.

QAPP Worksheet #10: Conceptual Site Model, Continued

- There are no borings/wells in the beach deposit mapped as flanking the drumlin at AOC 1.
- There is some uncertainty regarding groundwater flow direction, gradient, and velocity, and where/whether groundwater discharges to surface water bodies.
- There is almost no data for deep groundwater.
- Groundwater chemistry is not sufficiently characterized to support natural biodegradation mechanisms needed to assess persistence in the environment.
- There are no sampling data and limited historical information for AOC 5.
- There are no current field data to confirm the presence/absence or to confirm the ecological resources and receptors potentially under the influence of the Site.

If the Phase 1 screening results indicate further evaluation is needed, then the sampling design for Phase 2 will include data collection to meet risk assessment-related Daily Quality Objectives (DQOs).

10.8 REFERENCES

AGC, 2019. NYOW – Baldwinsville, New York, Historical Photo Analysis. Draft. Prepared for USACE New England District.

Alion, 2008. Final Site Inspection Report for NYOW, DERP FUDS Project Number C02NY029003. Prepared for the US Army Engineering and Support Center in Huntsville and USACE Baltimore District. Contract Number W912DY-04-D-0017, Task Order #00170001.

Chute, N.E., 1964. Structural Features in the Syracuse Area. New York State Geological Association 36th Annual Meeting, Syracuse University, Syracuse, New York. Pp. 74-79.

D&M, 1981. Evaluation of Possible Hazards of Former NYOW, Radisson New Community, Town of Lysander, New York. Prepared for the Urban Development Corporation and the New York State Department of Health.

EPA, 2006. Region III BTAG Freshwater Screening Values.

EPA, 2010. Considering Reasonably Anticipated Future Land Use and Reducing Barriers to Reuse at EPA-lead Superfund Remedial Sites. (OSWER Directive 9355.7-19). U.S. Environmental Protection Agency, Office of Emergency and Remedial Response.

EPA, 2015. Provisional Peer-Reviewed Toxicity Values for Picric Acid (2,4,6-Trinitrophenol) (CASRN 88-89-1).

Gerrard, M.B., 1993. New York State Urban Development Corporation vs. United States of America. U.S. District Court, Northern District of New York, Case Number 93-CV-546.

HSDB, 2009. Arsenic Compounds. No CASRN. National Library of Medicine. Last Revision Date: 17 February 2009. https://toxnet.nlm.nih.gov/cgi-bin/sis/search2/f?./temp/~EVuiAP:2

HSDB, 2011a. 2,4-Dinitrophenol, CASRN: 51-28-5. National Library of Medicine. Last Revision Date: 16 June 2011. https://toxnet.nlm.nih.gov/cgi-bin/sis/search2/f?./temp/~0w0ZXX:1

QAPP Worksheet #10: Conceptual Site Model, Continued

HSDB, 2011b. Picric Acid, CASRN: 88-89-1. National Library of Medicine. Last Revision Date: 04 January 2011. http://toxnet.nlm.nih.gov/cgi-bin/sis/htmlgen?HSDB

HSDB, 2012. 1-Chloro-2,4-dinitrobenzene, CASRN: 97-00-7. National Library of Medicine. Reviewed by SRP on: 17 May 2012. https://toxnet.nlm.nih.gov/cgi-bin/sis/search2/f?./temp/~QDM52I:3

HSDB, 2013. 1-Chloro-2,4-dinitrobenzene (SYN: dinitrochlorobenzene), CASRN: 97-00-7. National Library of Medicine. Last Revision Date: 08 February 2013. https://toxnet.nlm.nih.gov/cgi-bin/sis/search2/f?./temp/~P3qCqT:1

HSDB, 2016a. Chromium Compounds, No CASRN. National Library of Medicine. Last Revision Date: 25 October 2016. https://toxnet.nlm.nih.gov/cgi-bin/sis/search2/f?./temp/~Cn7yPt:2

HSDB, 2016b. Lead Compounds, No CASRN. National Library of Medicine. Last Revision Date: 25 October 2016. https://toxnet.nlm.nih.gov/cgi-bin/sis/search2/f?./temp/~SBwHYj:1

HSDB, 2018. Picric acid (SYN: 2,4,6-Trinitrophenol), CASRN: 88-89-1. National Library of Medicine. Last Revision Date: 28 November 2018. https://pubchem.ncbi.nlm.nih.gov/source/hsdb/2040

Kantrowitz, Irwin H., 1964. Groundwater Resources of the Syracuse Area. New York State Geological Association 36th Annual Meeting, Syracuse University, Syracuse, New York. pp. 39-41.

LaSala, A.M. Jr., 1968. Groundwater Resources of the Erie-Niagara Basin, New York: Basic Planning Report ENB-3, State of New York Conservation Department with Resources Commission.

Luther, D.D., 1879. Geological Map of Onondaga County, New York.

M&E, 1990. Defense Environmental Restoration Program, Contamination Evaluation at the Former NYOW, Lysander, New York. Volumes I and Volume II. Prepared for the Department of the Army, Kansas City District, Corps of Engineers. Final Report. [FUDS Document No. C02NY029001_01.09_0002_a]

Muller, E.H. and Cadwell, D.H., 1986. Surficial Geologic Map of New York, Finger Lakes Sheet, New York Geologic Survey, Education Department, Albany NY.

NOAA, 2017. Data Tools: 1981-2010 Normals. https://www.ncdc.noaa.gov/cdo-web/datatools/normals.

NYSDEC, 1990. Site Characterization Study, Three Rivers Facility, New York State Pesticide Storage Sites Project, Contract No. D001889. Prepared by Blasland & Bouck Engineers, P.C.

NYSDEC, 1991. Memorandum dated 14 May 1991 to New York State Urban Development Corporation regarding status of NYSDEC work at NYOW. [FUDS Document No. C02NY029003_01.01_0005_a]

NYSDEC, 1993. Operation and Maintenance Plan for the Three Rivers Pesticide Storage Site, Lysander, Onondaga County, Site No. 7-34-018.

NYSDEC, 2005. Concentrations of Selected Analytes in Rural New York State Surface Soils: A Summary Report on the Statewide Rural Surface Soil Survey. Provided in Appendix D to the New York State

QAPP Worksheet #10: Conceptual Site Model, Continued

Brownfield Cleanup Program, Development of Soil Cleanup Objectives, Technical Support Document. Prepared by NYSDEC and New York State Department of Health in September 2006.

NYSDEC, 2018. Habitat Management Plan for Three Rivers Wildlife Management Area 2018-2027. Prepared by the Division of Fish and Wildlife, Bureau of Wildlife, March 2018. https://www.dec.ny.gov/docs/wildlife_pdf/yfithreeriversshmp.pdf

Olsen, Fred and Goldstein, J.C., 1924. The Preparation of Picric Acid from Phenol. Industrial and Engineering Chemistry, Volume 16, Number 1, page 66.

Onondaga County Water Authority, 2019. Central New York's Water Authority, Sources of Water. https://www.ocwa.org/about/sources-of-water/

Pagano, Timothy S., Terry, David B., and Ingram, Arlynn W., 1985. Geohydrology of the Glacial-Outwash Aquifer in the Baldwinsville Area, Seneca River, Onondaga County, New York. USGS Water Resources Investigations Report 85-4094. Plates 1 to 6.

Pair, Donald L., 2014. Surficial Geology of the Baldwinsville Quadrangle, Onondaga County, New York. New York State Museum, New York State Geological Survey.

PubChem, 2020. Compound Summary, Picric Acid. https://pubchem.ncbi.nlm.nih.gov/compound/6954. Accessed 4 January 2020.

Radisson, 2017. Factsheet: Empire State Development: Radisson Community Project. https://esd.ny.gov/radisson-community-project.

Reconstruction Finance Corporation, undated. Chemical Manufacturing Plant (Ammonium Picrate), Baldwinsville, New York, Known as NYOW.

Rickard, L.V., 1969. Stratigraphy of the Upper Silurian Salina Group, University of the State of New York, State Education Department, Albany, NY.

Schneider, Philip F., 1894. Notes on the Geology of Onondaga County, New York.

USACHPPM, 2005. Wildlife Toxicity Assessment for Picric Acid (2,4,6-Trinitrophenol).

US Army Corps of Engineers (USACE), 1981. Memorandum for Commander, Kansas City District, Subject: Report on Records Search of NYOW. 13 March 1981. [FUDS Document No. C02NY029001_01.06_0006_p]

USACE, 1985. Determination and Findings of Fact, NYOW, Lysander, New York, Project No. C02NY002900.

USACE, 1991. Memorandum for Commander, North Atlantic Division, Subject: DERP-FUDS Inventory Project Report for Site No. C02NY029000, NYOW, Lysander, NY. Prepared by the Department of the Army, New York District, Corps of Engineers, New York, NY. 18 October 1991. [Enclosure to FUDS Document No. C02NY029002_01.08_0009_a]

QAPP Worksheet #10: Conceptual Site Model, Continued

USACE, 1999a. Archives Search Report, Findings for the former NYOW, Lysander Township, New York, Project Number C02NY029003. Prepared by USACE Rock Island District, September 1999. [FUDS Document No. C02NY029001_01.02_0001_a]

USACE, 2006. Fact Sheet on NYOW, Lysander, NY. Prepared by USACE New York District. [FUDS HTRW Document No. C02NY029001_08.11_0500_p]

USACE, 2012. Environmental Quality, Conceptual Site Models. Washington, DC, Engineer Manual 200-1-12.

US Fish and Wildlife Service (USFWS), 2018. National Wetlands Inventory, Wetlands Mapper. www.fws.gov/wetlands/data/Mapper.html.

Wetlands Research Services. 2020. Wetland Delineation and Vernal Pool Survey, NYOW. July 2020.

Zenger, D.H., 1965. Stratigraphy of the Lockport Formation in New York State. University of the State of New York, state Education Department, Albany NY.

QAPP Worksheet #11: Project/Data Quality Objectives
(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

In accordance with the 2012 Optimized Uniform Federal Policy (UFP)-QAPP Worksheets, **Worksheet #11** documents "the environmental decisions that need to be made and the level of data quality needed to ensure that those decisions are based on sound scientific data." In addition, the seven sections presented below are those recommended in the 2012 Optimized UFP-QAPP Worksheets and are based on EPA's 7-step Data Quality Objective (DQO) process.

11.1 PROBLEM STATEMENT

CERCLA constituents related to DoD operations at the Site may have been released to the environment. It is not known if these constituents are present or absent, and if present, whether levels represent acceptable or actionable risk. Preliminary DoD-related chemicals identified during previous sampling (1981, 1990, 1991, and 1993) are summarized below (see **Worksheet #10** for more details).

- Phenol
- PAHs
- Picric acid
- DNCB
- Arsenic
- Barium
- Cadmium
- Chromium
- Lead
- VOCs. Note, VOCs have not been encountered at the Site historically, but may be associated with the landfill at AOC 3 and the recently identified Power Plant (AOC 5).
- 2,4-DNP. Note that 2,4-DNP was not detected in previous studies but is an intermediate in the production of picric acid and therefore has been included as an analyte of interest.
- PCBs. Note, PCBs have not been encountered at the Site historically, but may be associated with the landfill at AOC 3 and former transformers associated with the recently identified Power Plant (AOC 5).

11.2 STUDY GOALS

The purpose of the study is to perform an RI to identify where and in what media DoD-related chemicals are above target risk-based action levels, delineate nature and extent of DoD-related chemicals above target risk-based action levels, evaluate risk to human and ecological receptors, determine the need for remedial action evaluations, and support the development and evaluation of effective response actions and abatement measures required per CERCLA guidelines such that site closure may be attained. The RI is proceeding with a phased approach:

- Phase 1 includes sampling and analyses at AOCs 1, 3, 4, and 5 that are designed specifically to gather additional information with respect to groundwater and surface/subsurface media quality where data gaps were identified following previous SI sampling efforts. If a release or releases of DoD-specific compounds are documented based on the Phase 1 data, then the

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

discrete sampling data will be used to provide insight in the development of viable Decision Units (DUs) for additional ISM sampling to assess risk, as needed. These components will be evaluated concurrently to further refine the comprehensive RI sampling approach.

- To achieve these objectives, sampling locations were selected to determine if any DoD-related CERCLA constituents are present above applicable risk-based screening levels and background levels. Background soil and groundwater samples will be submitted for metals and PAHs analyses only because these compounds commonly occur in soils anthropogenically and anthropologically outside the possible impacts of DoD-specific operations. If DoD-related analyte concentrations in soil or groundwater exceed human health or ecological risk-based screening levels and background concentrations, the investigation will advance to Phase 2. If executed, average concentrations generated by additional ISM samples will be used during Phase 2 for the purpose of determining risk.
- Phase 2 activities are designed to characterize and quantify information gathered during Phase 1 sampling activities, in order to refine the CSM. In turn, the improved CSM will be used to develop and maximize the RI sampling approach in order to assess human health and ecological risk at each AOC where DoD-related CERCLA chemicals are detected above both risk-based screening levels and background concentrations.

ISM controls variability associated with the heterogeneous distribution of contaminants in soil. The objective of ISM is to determine a representative estimate of the mean concentration of a contaminant within a pre-determined area (referred to as a DU). Therefore, ISM sampling of surface soils will be completed around each of the FUDS-eligible bunkers at AOC 4, and biased discrete sample locations have been placed in AOCs 1, 3, 4, and 5 where contamination is expected based upon site usage, fate and transport, and site characteristics. These discrete sample results will be used in the systematic planning and design of the Phase 2 DUs, if Phase 2 is needed. Additional ISM sampling at the site is intended for subsequent investigation to determine the average concentration over the AOC, only if contamination is identified in Phase 1.

As with Phase 1 on-site discrete sampling, corresponding discrete background sampling will be conducted for applicable AOCs. Specifically, background soil and groundwater will not be collected associated with AOC 4, the former Bunker Area, because the only DoD-specific compounds or concern at this AOC are explosives and explosives do not represent a background condition. If the investigation proceeds to Phase 2, subsequent background ISM sampling will be performed for applicable AOCs, as needed. On-site Phase 1 and Phase 2 analytical data will be compared individually with their respective background sample sets.

Phase 1 discrete sample data will not be combined with either Phase 1 or Phase 2 ISM data. The Phase 1 data will be used to determine the need for Phase 2 through comparison to RSLs and discrete background sample data. If exceedances are found, these exceedances will be used to guide the Phase 2 ISM sampling and the location for ISM sampling, if needed. If Phase 2 is needed, new site and background sample locations will be determined and collected using an ISM approach. If Phase 2 is needed, a Phase 2 addendum to the QAPP will be prepared. Currently expected Phase 2 goals and activities are presented below; however, only Phase 1 activities are detailed in this QAPP. The scope of Phase 2 investigation activities and risk evaluations performed will be determined based on the

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

results of the lines of evidence evaluated during Phase 1. Supporting goals are defined in the following sections.

11.2.1 All AOCs

Phase 1

- Determine if potential DoD-related chemical concentrations in surface and subsurface soil related to historical DoD operations are present above human health and ecological risk-based screening levels and background concentrations (Phase 1).
- Collect and develop a background data set for anthropogenic and anthropologic metals and PAHs for surface soil and subsurface soil at locations not impacted by DoD operations (Phase 1).
- Calculate background threshold values (BTVs), to which site concentration data collected using discrete sampling methods will be compared.
- Compare groundwater DoD-related chemical levels to upgradient locations and human health risk-based screening levels (Phase 1).
- Ground truth mapped geology, by logging and interpreting soil samples, to assess the influence of geologic features (such as the coarser beach sand deposits at AOC 1) on constituent migration.
- Characterize soil and bedrock properties relevant to fate and transport (Phase 1).
- Determine groundwater elevation and gradient (Phase 1).
- Determine where groundwater may discharge to surface water (Phase 1).
- Perform a potable well survey to assess current or future plans for drinking water wells in the vicinity of AOCs (Phase 1).
- Determine potential reasonably foreseeable future land uses at each of the AOCs for purposes of determining appropriate future receptors to be evaluated during Phase 2.
- Perform a resource and receptor inventory, wetlands delineation, and vernal pool survey to identify potential ecological receptors.

Phase 2

- If drinking water wells are identified in the vicinity of AOCs, assess the potential for migration of residual concentrations of DoD-related chemicals in groundwater to these wells for the purpose of evaluating risk to human health (Phase 2).
- Determine the need to sample residential wells (Phase 2).
- If residential wells should be sampled, develop sampling and analysis plan as part of a QAPP addendum, including collection of relevant upgradient groundwater.
- Determine the need to collect additional data to characterize potential risks to human health and ecological receptors (Phase 2).
- Determine the need to collect additional site and background data to characterize nature and extent of DoD-related chemicals above background and target risk levels in the environment (Phase 2).
- Determine the need to collect sufficient data to complete a field survey (Phase 2), if necessary.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

11.2.2 AOC 1 – FORMER AMMONIUM PICRATE AREA

Historical site features and proposed site investigation locations for AOC 1 (the Former Ammonium Picrate Area) are presented on **Figure 11-1**.

Phase 1

- Collect discrete soil samples to assess whether DoD-related chemicals associated with the production area historical activities representing potential source areas including the former Ammonium Picrate buildings, magazines, change houses, dryer houses, and the laboratory building are present above risk-based human health and ecological screening and background levels in the surface soil (0 to 1 ft bgs) and subsurface soil (1 to 5 ft bgs and 5 to 10 ft bgs).
- If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. After the Phase 1 sampling results/analyses are evaluated and additional delineation is deemed necessary, then additional sampling will be conducted during Phase 2.
- Soil and groundwater samples will not be collected in areas associated with the three former Acid Tanks, as the tanks are not FUDS-eligible as discussed in **Worksheet #10**.
- Advance Direct Push Technology (DPT) soil borings to collect soil data.
- Convert DPT boreholes to temporary groundwater monitoring wells to collect groundwater data.
- Collect depth-to-water measurements in temporary monitoring wells to determine the groundwater elevation and gradient.
- Collect grab groundwater samples from temporary monitoring wells to assess whether DoD-related chemicals associated with the production area historical activities representing potential source areas including the former Ammonium Picrate buildings, magazines, change houses, dryer houses, and the laboratory building are present above risk-based human health screening and background levels in groundwater.
- Record water quality parameters (i.e., temperature, pH, specific conductance, DO, Oxidation Reduction Potential (ORP), and turbidity) at each groundwater sample collection point using a multi-parameter meter.
- Assess if groundwater that flows from the Site discharges to the nearby wetland.
- Perform a resource and receptor inventory, wetlands delineation, and vernal pool survey to identify potential ecological receptors.

Phase 2

- If discrete samples indicate that DoD-related chemicals associated with the production area are present above risk-based human health and ecological screening and background levels in surface soil, perform additional site and background soil sampling via ISM as part of the RI and to characterize human health and ecological risk.
- If DoD-related chemicals are present above risk-based human health screening and background levels in groundwater, then install permanent groundwater monitoring well(s) at the suspected source areas, as well as upgradient and downgradient of the source area.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

- If DoD-related chemicals are potentially migrating from groundwater into the nearby wetland, collect pore water, surface water, and sediment samples.
- If DoD-related chemicals associated with the production area are identified in subsurface soils, assess if soil habitat suitable for supporting ecological receptors is found within subsurface soil (specifically 1 to 5 ft bgs interval).
- If DoD-related chemicals associated with the production area are detected in subsurface soils above ecological screening levels, if habitat is available at the surface soil horizon such that terrestrial plants, soil invertebrates, and burrowing mammals can access subsurface soils, and if suitable soil habitat for supporting ecological receptors is found with 1 to 5 ft bgs interval, compare concentrations with ecological benchmarks as part of the site characterization process to determine the need for additional sampling and subsequent risk characterization.
- Although the present understanding is that there are no future residential development plans for the areas of potential contamination and residential exposures are not currently anticipated, if DoD-related chemicals associated with the production area are detected in subsurface soils above human health risk-based screening levels, it will be determined whether future residential use, future industrial/commercial use, future construction, and/or future installation of utility lines are reasonably foreseeable in the impacted area(s).
- If future residential use, future industrial/commercial use, future construction, and/or future installation of utility lines are reasonably foreseeable in the impacted area(s), determine the need for additional subsurface soil sampling and subsequent human health risk characterization.
- If DoD-related chemicals are migrating from groundwater into the nearby wetland, assess potential residual DoD-related chemicals in pore water, surface water, and sediment for the purpose of evaluating risk to human and ecological receptors.
- If there are DoD-related chemicals associated with the production area in subsurface soils and/or groundwater, assess whether any of these constituents are detected above human health risk screening criteria in downgradient current or potential future drinking water and collect the data for use in the HHRA.

11.2.3 AOC 3 – FORMER LANDFILL AREA

Historical site features and proposed site investigation sampling points for the Former Landfill Area and the open storage area at AOC 3, are presented on **Figure 11-2**.

Phase 1

- Review historical maps/photos and perform surficial geophysical techniques, specifically ground-penetrating radar (GPR) and an electromagnetic induction (EM) Survey to delineate the lateral and vertical extent of the former landfill and assess if there is evidence of buried containerized waste or large metallic debris below the parking lot.
- Assess if DoD-related chemicals associated with the former landfill are present in surface and subsurface soils in the vicinity of the landfill. No soil samples will be collected within the fill material in the interval directly beneath the asphalt pavement (0 to 1 ft).
- If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. After the Phase 1 sampling

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

results/analyses are evaluated and additional delineation is deemed necessary, then additional sampling will be conducted during Phase 2.

- Assess if the former landfill is impacting local groundwater quality.
- Assess if groundwater that flows from the Site is impacting nearby wetlands.
- Assess if vapor intrusion pathways exist from possible VOC impacts associated with the former landfill.
- Collect discrete soil samples to assess if DoD-related chemicals associated with the former open storage area were released to the subsurface.
- Collect a grab groundwater sample from one co-located soil sampling location to assess whether DoD-related chemicals associated with the former open storage area have impacted local groundwater.
- Record water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) during the collection of samples from a temporary monitoring well using a multi-parameter meter.
- Perform a resource and receptor inventory, wetlands delineation, and vernal pool survey to identify potential ecological receptors.

<u>Phase 2</u>

- If potable wells are present or installation of potable wells downgradient of the former landfill is not restricted, determine potential residual constituent concentrations in site groundwater for the purpose of evaluating risk to human health through ingestion, dermal contact, inhalation while showering, and vapor intrusion.
- If buried waste is mapped with geophysics, evaluate whether it poses an unacceptable risk and/or a continuing source, and if so, consider removal as part of future work.
- If DoD-related chemicals are present above human health and ecological risk-based screening and background levels in surface soil, collect surface soil samples via ISM to determine potential residual constituent concentrations in surface soil associated with the landfill area for the purpose of evaluating risk to human health.
- If there are DoD-related chemicals reported above screening levels in subsurface soils, assess if soil habitat suitable for supporting ecological receptors is found within subsurface soil (specifically 1 to 5 ft bgs cores). If DoD-related chemicals associated with the landfill in subsurface soils exceed ecological screening levels, if habitat is available at the surface soil horizon such that terrestrial plants, soil invertebrates, and burrowing mammals can access subsurface soils, and if suitable soil habitat for supporting ecological receptors is found within 1 to 5 ft bgs, compare concentrations with ecological benchmarks to assess the need for additional sampling and subsequent ecological risk evaluation.
- If there are DoD-related chemicals reported above vapor intrusion based human health screening levels in groundwater, or DoD-related VOCs detected in subsurface soils, determine the need to collect sub-slab soil gas samples, and/or indoor air at the golf course club house to assess the potential for vapor intrusion from possible VOC impacts from the landfill.
- If DoD-related chemicals are migrating from groundwater into the unnamed stream north of the landfill, assess potential residual DoD-related chemicals in pore water, surface water, and sediment for the purpose of evaluating risk to ecological receptors.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

11.2.4 AOC 4 – FORMER BUNKER AREA

Historical site features and proposed site investigation sampling points for AOC 4 the Former Bunker Area are presented on **Figure 11-3**.

Phase 1

- Collect ISM surface soil samples from the 0 to 1 ft bgs interval within a one-half acre DU around each of the 12 FUDS-eligible bunkers.
- Collect discrete samples to assess if DoD-related chemicals associated with the bunkers are present above human health and ecological risk-based screening levels in subsurface soil (in the 1 to 5 ft bgs interval) directly beneath the discharge of the drain lines (potential source area) and along the swales (Phase 1).
- If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. After the Phase 1 sampling results/analyses are evaluated and additional delineation is deemed necessary, then additional sampling will be conducted during Phase 2.
- Assess the quality of groundwater in the vicinity of the bunkers.
- Assess if groundwater that flows from the Site discharges to the adjacent surface water bodies and wetlands.
- Perform a resource and receptor inventory, wetlands delineation, and vernal pool survey to identify potential ecological receptors.

Phase 2

- Assess for potential residual DoD-related chemicals in groundwater for the purpose of evaluating risk to human health.
- If discrete samples indicate that DoD-related chemicals associated with the bunkers are present above human health and ecological risk-based screening levels in subsurface soil beneath the discharge of the drain lines and/or in swales, assess the subsurface soils within the DUs and/or length along the swales by adding ISM grids between the bunker ISM grids.
- Using samples collected with the ISM approach, assess potential residual constituent concentrations in surface soil that are associated with the bunkers for the purpose of evaluating risk to human health and ecological receptors.
- If DoD-related chemicals associated with the bunker subsurface soils are identified, determine if soil habitat suitable for supporting ecological receptors is found within subsurface soil (specifically 1 to 5 ft bgs interval).
- If there are DoD-related chemicals associated with the bunkers in subsurface soils, if habitat is available at the surface soil horizon such that terrestrial plants, soil invertebrates, and burrowing mammals can access subsurface soils, and if suitable soil habitat for supporting ecological receptors is found (1 to 5 ft bgs), compare concentrations with ecological benchmarks as part of the site characterization process to determine the need for additional sampling and subsequent risk evaluation.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

- If DoD-related chemicals may be migrating in groundwater to the nearby surface water bodies and wetlands, sample pore water, surface water, and sediment to assess potential residual constituent concentrations for the purpose of evaluating risk to ecological receptors.
- If there are DoD-related chemicals associated with the bunker area subsurface soils and/or groundwater, assess if any of these constituents are potentially present in downgradient current or potential future drinking water and use the data for the HHRA.

11.2.5 AOC 5 – FORMER POWER PLANT AREA

Historical site features and proposed site investigation sampling points for AOC 5 the Former Power Plant are presented on **Figure 11-4**.

Phase 1

- Using historical maps/photos and surficial geophysics, specifically GPR, assess if there is evidence of demolition debris or disposal of other waste within AOC 5.
- Collect discrete soil samples to assess whether DoD-related chemicals associated with the former power plant operations (i.e., the boiler house, coal storage area, substation, possible dump, and coal aggregate bins) are present above human health and ecological risk-based screening and background levels in the surface soil (0 to 1 ft bgs) and subsurface soil (1 to 5 ft bgs and 5 to 10 ft bgs).
- If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. After the Phase 1 sampling results/analyses are evaluated and additional delineation is deemed necessary, then additional sampling will be conducted during Phase 2.
- Collect surface soil samples (0-1 ft bgs) in the coal storage area and around the coal aggregate bins to determine if coal ash is present. Although coal particles are not a regulated substance under CERLCA, impacts to subsurface soil and groundwater from the coal particles are evaluated under CERCLA.
- Advance DPT soil borings to collect soil data.
- Convert select DPT boreholes into temporary groundwater monitoring wells to collect groundwater data.
- Collect depth-to-water measurements from the temporary monitoring wells to determine groundwater elevation and gradient.
- Collect grab groundwater samples from the temporary monitoring wells to assess whether DoD-related chemicals associated with the former power plant operations (i.e., the boiler house, coal storage area, substation, possible dump, and coal aggregate bins) are present above human health risk-based screening levels in groundwater.
- Record water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) at each water sample point using a multi-parameter meter.
- Assess if groundwater that flows from the Site discharges to the nearby wetland.
- Perform a resource and receptor inventory, wetlands delineation, and vernal pool survey to identify potential ecological receptors.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

Phase 2

- If discrete sampling indicates that DoD-related chemicals associated with the former power plant are present above human health and ecological risk-based screening and background levels in surface soil, perform additional site and background soil sampling via ISM for an RI and to characterize human health and ecological risk. If these chemicals include PCBs in surface soils at the substation, additional samples will be collected and analyzed for PCBs from subsurface soil and groundwater.
- If DoD-related chemicals are present above human health risk-based screening levels in groundwater, then install permanent groundwater monitoring wells at the suspected source area, as well as upgradient and downgradient of the source area.
- If DoD-related chemicals are potentially migrating from groundwater into the nearby wetland, collect pore water, surface water, and sediment samples.
- If a DoD-related release occurred that could convey constituents to drainage and wastewater ditches via migration pathways such as runoff and/or leaching, collect pore water, surface water, and sediment samples.
- If there are DoD-related chemicals associated with the former power plant subsurface soils, assess if soil habitat suitable for supporting ecological receptors is found within subsurface soil (specifically 1 to 5 ft bgs interval).
- If DoD-related chemicals associated with the former power plant are detected in subsurface soils above screening levels, if habitat is available at the surface soil horizon such that terrestrial plants, soil invertebrates, and burrowing mammals can access subsurface soils, and if suitable soil habitat for supporting ecological receptors is found within 1 to 5 ft bgs, compare concentrations with ecological benchmarks as part of the site characterization process to determine the need for additional sampling and subsequent risk characterization.
- If DoD-related chemicals associated with the former power plant are detected in subsurface soils above human health risk-based screening levels determine whether future residential use, future industrial/commercial use, future construction, and/or future installation of utility lines are reasonably foreseeable in the impacted area(s).
- If future residential use, future industrial/commercial use, future construction, and/or future installation of utility lines are reasonably foreseeable in the impacted area(s), determine the need for additional subsurface soil sampling and subsequent human health risk characterization.
- If DoD-related chemicals are migrating from groundwater into the nearby wetland, assess potential residual DoD-related chemicals in pore water, surface water, and sediment for the purpose of evaluating risk to human and ecological receptors.
- If there are DoD-related chemicals associated with the former power plant in subsurface soils and/or groundwater, assess whether any of these constituents are detected above risk screening criteria in downgradient current or potential future drinking water and collect the data for use in the HHRA.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

11.3 INFORMATION INPUTS

The principal inputs to meet the study goals above are chemistry data obtained from soil and groundwater; as well as groundwater characteristics and geophysical data. Information required consists of the following:

- Discussion with the town and current owners (Radisson Community, the State of New York, and private owners), review of current zoning, review of the Radisson Community General Project Plan (1971) and recent amendments, and review of town master plans (Phase 1). This information will be used to determine reasonably foreseeable future land use at each of the AOCs and future receptors to be evaluated during Phase 2 HHRAs.
- Potable well survey to determine if any private wells are used for drinking water within a 2-mile radius of the Site. If there is an indication that off-site residential wells are being impacted by groundwater at the Site, and if groundwater is determined to be hazardous to human health based on the findings of the HHRA, additional groundwater sampling may be required.
- Groundwater elevation measurement survey data to contour groundwater table and determine groundwater flow direction. Existing wells might be used for groundwater elevations if they can be located and are not damaged (e.g., filled with silt or are poorly sealed at the surface).
- Drilling logs (including organic vapor screening levels, staining, odors) and well completion diagrams to document geologic formations and depths of groundwater samples.
- Electromagnetic induction and GPR geophysical surveys at AOC 3 to determine if there are large objects within the buried material beneath the Radisson Greens Golf Course parking lot. Large objects may be related to historical processing and could be excavated in future studies or remedial actions.
- Electromagnetic induction and GPR geophysical surveys at AOC 5 to determine if there are large objects beneath the ground surface, in particular in the possible dumping area. Large objects related to historical site activities could be excavated in future studies or remedial actions.
- Resource Receptor Inventory for aquatic and terrestrial habitats to ensure appropriate exposure populations are evaluated.
- Wetlands Delineations and vernal pool surveys at AOC 1, AOC 3, AOC 4, and AOC 5 to identify habitats and receptors of ecological concern that may be impacted by site-related constituents.
- Prior to collecting grab samples for chemical analysis, water quality parameters will be recorded in each temporary monitoring well, including the following: temperature, pH, specific conductance, dissolved oxygen (DO), oxidation-reduction potential (ORP), and turbidity.
- Groundwater analytical chemistry data (AOCs 1, 3, 4, and 5) to clarify the CSM and, if the potable well survey shows current or future plans for drinking water wells, determine risks to human health from DoD-related chemicals.
- Surface (0 to 1 ft bgs) soil analytical chemistry data from ISM sampling to identify potential impacts in AOC 4.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

- Surface (0 to 1 ft bgs) soil analytical chemistry data from discrete sampling to identify potentially impacted areas (AOCs 1, 3, 4, and 5) and inform potential Phase 2 placement and design of ISM sampling grids (AOCs 1, 3, 4, and 5).
- Potential Phase 2 ISM surface soil samples and analytical chemistry data collection (AOCs 1, 3, 4 and 5) to determine risks to human and ecological receptors from DoD-related chemicals.
- Subsurface soil analytical chemistry data (from discrete soil borings) to determine potential impacts to subsurface soils and evaluate potential groundwater impacts from DoD-related chemicals (AOCs 1, 3, 4, and 5) and risks to ecological receptors (AOCs 1, 3, 4, and/or 5). Chemical concentrations in discrete subsurface soil samples will be compared to human health RSLs and appropriate ecological benchmarks as part of the site characterization process and will be used to determine the need for additional sampling and subsequent risk evaluation.
- Potential Phase 2 subsurface soil sampling design may be developed to address subsurface human health and/or ecological exposure scenarios. Potential for future construction and residential exposure scenarios will be determined before the Phase 2 sampling effort.
- DPT groundwater grab sampling to determine lateral extent of potential plumes of DoD-related chemicals radiating from potential source areas.
- If DPT sampling results indicate a potential pathway exists, Phase 2 may include pore water, surface water, and sediment analytical chemistry data (AOCs 1, 3, 4 and/or 5) from discrete sampling to determine risks to ecological receptors from DoD-related chemicals.
- GPS coordinates.
- Survey data.
- Physico-chemical properties to help determine appropriate background locations and/or bioavailability:

    - Soil: Total organic carbon (TOC), grain size distribution (including hydrometer analysis),, and cation exchange capacity (CEC).
    - Groundwater: Water quality field parameters, including pH, temperature, specific conductance, DO, ORP, and turbidity.
    - Phase 2 if needed – Pore water and surface water: field measured water quality parameters including, hardness, pH, temperature, specific conductance, DO, ORP, and turbidity.
    - Phase 2 if needed – Soil: TOC, grain size distribution (logged in field in accordance with ASTM D 2487 and analyzed at the laboratory using EPA method 9060A), pH (field measurement), CEC, ORP (field measurement).
    - Phase 2 if needed – Sediment: TOC, grain size distribution (including hydrometer analysis), pH (field measurement), CEC, ORP (field measurement), Acid Volatile Sulfide (AVS)/ Simultaneously Extracted Metals (SEM).

- Background sampling will be conducted to evaluate surface and subsurface conditions outside the area of influence (i.e., topographically upgradient/upslope) of the historic DoD operations and any associated environmental impacts, in order to evaluate the degree of impacts to the Site and downgradient areas caused by historical site use. To identify soil types that are similar in chemistry, a comparison of soils will be performed for each AOC and respective background

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

sample zone(s) (refer to **Figure 11-5**). Background samples for this investigation will be collected from select areas, outside the zone of influence of historical releases from these areas, and with soil types similar to the individual AOCs. It should be noted that no background samples (soil or groundwater) are proposed for AOC 4, based on the nature of the chemicals of concern at this location, i.e., explosives (DNCB, 2,4-DNP, and picric acid).

- To confirm the representativeness of the background sample locations, CEC, TOC, and grain size distribution (including hydrometer analysis) will be analyzed from subsets of soil samples collected at each AOC location (refer to **Section 11.5.5**) and from the selected background locations for comparison. Based on regionally mapped surficial geology (Pair, 2014), a total of 15 co-located discrete surface and subsurface soil samples will be collected for each major soil type identified within the AOCs. Background locations will be collected within the Radisson Community Association property. AOC 1 has three distinct geologic units (silt and clay; stratified silt, sand, and gravel; and diamicton), AOC 3 is mapped entirely in silt and clay, and AOC 5 is mapped in silt and clay, and diamicton (refer to **Figure 11-5**).
- At each AOC, DPT soil borings will be advanced in potentially impacted areas and/or source areas, and at locations in the respective upgradient/upslope and downgradient directions. Depth-to-water measurements will be used to calculate groundwater elevation and gradient, and to confirm which DPT sampling points are representative of upgradient, source area, and downgradient conditions. Source area and downgradient DoD-related chemical concentrations in soils and groundwater will be compared to upgradient (background) concentrations.
- If DoD-related chemicals are identified in site groundwater, the permanent monitoring well installations reserved for Phase 2 activities will be completed. At least one permanent monitoring well will be installed upgradient of the source area at each AOC, for both HHRA and site characterization purposes. The subsequent groundwater monitoring program should include packer testing in order to evaluate multiple zones of groundwater quality.

Analytical chemistry data from background locations will be collected for the following media:

- Surface soil – discrete for Phase 1 and, if needed, ISM for Phase 2. Note results from ISM sampling will not be compared with discrete sampling background results.
- Subsurface soil – discrete for Phase 1 and, if needed, ISM for Phase 2.
- Groundwater – collected as grab samples from DPT soil borings/temporary monitoring wells upgradient of AOCs 1, 3, and 5 for Phase 1 and, if needed, from permanent monitoring wells during Phase 2 activities.
- Pore water – Phase 2, if needed.
- Surface water – Phase 2, if needed.
- Sediment – Phase 2, if needed.

11.4 BOUNDARIES OF THE STUDY

The boundaries of the study area presented below are based on current knowledge. It is expected that the temporal and spatial boundaries of the study area could change based on the results of Phase 1 investigations, including the resource and receptor inventory, wetland delineation, and vernal pool study.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

The potable well survey for any private wells used for drinking water will be conducted for properties within a 2-mile radius of the Site for potential use in an off-site residential exposure scenario.

All field sampling will occur in the AOCs 1, 3, 4, and 5, as well as background areas, as applicable. As stated above, a subset of samples from applicable AOCs and from background locations will be submitted for TOC and grain size distribution (including hydrometer analysis),and CEC to assess comparability/representativeness of the soils. Background sampling and associated subsets are discussed further in **Section 11.5.5**.

The DoD is not responsible for the investigation of mobilization of metals owing to the acid tanks that were remediated by NYSDEC. Therefore, no soil or groundwater sampling is planned associated with the three former acid tanks at AOC 1. However, DoD is responsible for the metals used during the picric acid manufacturing process, so the rest of AOC 1 will be assessed for metals outside the footprint of the former acid tanks and immediate groundwater.

Soil data collected during Phase 1 will determine the need for further sampling through the comparison of site detections with risk-based screening levels and background levels. Phase 1 data collected via ISM and Phase 2 soil data will be used in human and ecological risk assessments, if necessary.

Sediment, pore water, and surface water in the unnamed stream north of the landfill in AOC 3 and in the wetlands located downgradient of AOCs 1, 4, and 5 will be included in Phase 2 if the phased groundwater assessment approach indicates the potential for groundwater discharge to surface water, with sampling for the same set of physico-chemical properties. A human health exposure pathway may be identified as part of Phase 2 activities. The temporal boundaries will be dependent on ground temperature (i.e., freezing conditions and/or snow cover). Sampling has been tentatively scheduled to begin in the early fall of 2020, with completion by late fall/early winter 2020, weather permitting.

11.5 ANALYTICAL APPROACH

Below is a summary of the analytical approach. Decision points are expressed in "if—then" statements. Specific methods used and constituents analyzed in each method are presented in **Worksheet #15** and details of the sampling plan are in **Worksheet #17**. The preliminary soil and groundwater investigation at all AOCs will be completed using Geoprobe® Direct-Push Technology (DPT) drilling methods.

Bluestone evaluated the need to include degradation produced of the organic contaminants. Nitrates, which are degradation products of picric acid and DNP, may not be associated with historical DoD use. If DoD-related contamination is found during the Phase 1 investigation, the need for evaluating degradation products (e.g., nitrates) will be assessed.

Picric acid is resistant to aerobic biodegradation (*HSDB. Picric Acid, CASRN: 88-89-1. National Library of Medicine. Last Revision Date: 04 January 2011*). Degradation of picric acid under anaerobic conditions is a potentially minor fate process (*U.S. Army Public Health Center. 2019. Wildlife Toxicity Assessment for Picric Acid [2,4,6-Trinitrophenol]*). With the exception of wetland areas, on-site soils

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

are not expected to support picric acid degradation. If wetland areas will be investigated as part of Phase 2 and analysis of picramic acid, the degradation product of picric acid, will be evaluated. Based on biodegradability measurements, biodegradation is not an important environmental fate process of DNCB and DNP (*HSDB, 2,4-Dinitrophenol, CASRN: 51-28-5. National Library of Medicine. Last Revision Date: 16 June 2011; HSDB, 1-Chloro-2,4-dinitrobenzene (SYN: dinitrochlorobenzene), CASRN: 97-00-7. National Library of Medicine. Last Revision Date: 08 February 2013*).

There is no indication that DNCB, DNP or TNP biodegrade to volatile compounds. (Agency for Toxic Substances and Disease Registry (ATSDR). 2019. *Toxicological profile for Dinitrophenols (Draft for Public Comment)*. Atlanta, GA: Brannon and Pennington. 2002. *Environmental Fate and Transport Process Descriptors for Explosives. ERDC/EL TR-02-10; U.S. Department of Health and Human Services, Public Health Service; U.S. Army Public Health Center. 2019. Wildlife Toxicity Assessment for Picric Acid (2,4,6-Trinitrophenol)*.

11.5.1 AOC 1 – Former Ammonium Picrate Area

Phase 1 sampling is designed to assess the presence/absence of DoD-related constituents. In Phase 2, DUs will be designed based on the Phase 1 results to determine extent of contamination and assess risk using ISM samples. During Phase 1, a total of 24 soil borings will be advanced in the vicinity of the former Ammonium Picrate structures (see **Figure 11-1**). Grab groundwater samples will be collected from a total of 10 locations, eight of which will be co-located with soil borings, to assess groundwater flow and the potential for DoD-related chemical transport to stream systems north and east of the six production lines (see **Figure 11-1**). As stated above in **Section 11.4**, impacts to soil and groundwater associated with the acid tanks previously remediated by NYSDEC will not be evaluated as part of this project.

Soil borings will be advanced to refusal and logged continuously by an experienced glacial geologist. Details regarding logging methods are discussed in **Worksheet #17**. If shallow refusal is encountered (i.e., less than 5 ft bgs), three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped, and the drilling methodology will be reassessed during Phase 2. Co-located discrete surface soil samples (0 to 1 ft bgs) and subsurface soil samples (1 to 5 ft bgs and 5 to 10 ft bgs) will be collected from 22 of the soil borings to identify any residual constituents within the production area, and the influence of the beach sand deposits on constituent migration (see **Figure 11-1**). If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. The soil borings are placed strategically around the Former Ammonium Picrate area structures including the dryer houses, magazines, change houses, the laboratory, and the associated buildings.

At refusal, and upon completion of logging and soil sampling, each borehole will be converted into a temporary monitoring well, for the purpose of collecting depth-to-water measurements and/or to collect a discrete grab groundwater sample, as directed by the WP. Construction of the temporary monitoring wells is detailed in **Worksheet #17**. Any additional water bearing zones identified during the logging of soil cores will be sampled by advancing a separate soil boring to the target depth and constructing a temporary monitoring well. It should be noted that due to the unconsolidated nature of subsurface media and preferred drilling methods, separate soil borings/temporary monitoring

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

wells are the most reliable method for collecting discrete groundwater samples. In addition, it is possible that refusal may occur in till or saprolite (weathered shale) before competent bedrock is encountered.

Groundwater sampling locations are selected along transects roughly perpendicular to the estimated direction of groundwater flow and in the vicinity of possible source area locations. Up to 20 groundwater samples will be collected from AOC 1. The samples will be collected using a peristaltic pump, provided that hydraulic head does not exceed 30 to 35 feet. If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples. Water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) will be recorded at the time of sample collection, using a multi-parameter meter. The temporary wells will remain in place until the end of the subsurface investigation, for the purpose of collecting site-wide synoptic groundwater elevation data. At the completion of site work, the temporary monitoring wells will be properly abandoned in accordance with NYSDEC Policy CP-43: Groundwater Monitoring Well Decommissioning Policy, dated 3 November 2009 (NYSDEC, 2009).

The sampling plan was based on historical use and the results of the previous investigations. AOC 1 was used for the production of picric acid. Therefore, the investigation in this area is limited to the compounds associated with the production and degradation of picric acid (see **Section 11.5** introductory text for more information regarding degradation products). There is no evidence from either historical site usage or preliminary sampling to suggest the need for including VOC and PCB analysis at AOC 1.

Wastewater ditches were excluded from the Phase 1 sampling because previous investigations (D&M, 1981 and M&E, 1990) indicated that wastes would have been highly diluted by precipitation and previous sampling of the wastewater ditches did not detect any constituents of concern (see **Subsections 10.3.1 and 10.5**).

The following analyses will be performed per medium in AOC 1 during Phase 1:

- Surface soil: TAL metals, DNCB and 2,4-DNP, picric acid; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
- Subsurface soil: TAL metals, DNCB and 2,4-DNP, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
- Groundwater (grab samples): TAL metals (field-filtered), DNCB and 2,4-DNP, and picric acid; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Results of the Phase 1 investigation at AOC 1 will be integrated into the CSM, in order to refine the model and more precisely direct the design of the RI sampling. This includes strategic placement of the permanent monitoring wells and the development of DUs for ISM sampling to capture any potential releases associated with the former Ammonium Picrate operations. This additional information will be used to enhance the comprehensive human health and ecological risk assessments for AOC 1. If, based on the Phase 1 sampling, it appears as though a release occurred that could convey constituents to drainage and wastewater ditches via migration pathways such as runoff and/or

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

leaching, drainage ditches may potentially be sampled during Phase 2 activities. In addition, delineation sampling of observed impacts may be included in Phase 2 based on Phase 1 sampling results/analyses.

If the results of the groundwater assessment, wetlands delineation, and vernal pool study indicate the potential for discharge of DoD-related constituents into neighboring wetlands, then co-located pore water, surface water, and sediment samples will be collected to determine whether constituents have migrated to the wetlands. If needed, the pore water, surface water and sediment sampling program (0 to 6 inches bgs) at AOC 1 will be implemented based on the preliminary groundwater analytical results and groundwater flow analysis.

11.5.2 AOC 3 – Former Landfill Area

Phase 1 sampling is designed to assess the presence/absence of DoD-related constituents. In Phase 2, DUs will be designed based on the Phase 1 results to determine extent of contamination and assess risk using ISM samples.

Two areas within AOC 3 will be investigated, the Landfill Area and the open storage area. To begin the investigation, geophysical field methods (i.e., ground-penetrating radar (GPR) and electromagnetic survey (EM-31) will be performed to determine the extent of the landfill. A total of 12 DPT soil borings will be advanced in grassy areas along the perimeter of the landfill (see **Figure 11-2**). Note that samples will not be collected from beneath the asphalt. In addition, sampling will remain on the RCA Common Property and not infringe on the private properties (e.g., private residential properties on Darting Bird Lane) that the area borders. Grab groundwater samples will be collected from a subset (i.e., six, of the soil borings) to assess if the landfill is impacting local groundwater. If possible, multiple zones of groundwater will be sampled in soil borings.

In the Landfill Area, the soil borings will be advanced in 5-ft increments until refusal is encountered and logged continuously by an experienced glacial geologist as described above. If shallow refusal is encountered (i.e., less than 5 ft bgs), three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped, and the drilling methodology will be reassessed during Phase 2. Co-located discrete surface soil samples (0 to 1 ft bgs) and subsurface soil samples (1 to 5 ft bgs) will be collected from each DPT soil boring (see **Figure 11-2**). If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval.. It should be noted that refusal may occur in till or saprolite (weathered shale) before competent bedrock is encountered. If this occurs, the deeper groundwater in bedrock will be targeted during Phase 2 activities, with the installation of permanent monitoring wells.

Up to 6 groundwater samples will be collected from the AOC 3 Landfill Area. At refusal, and upon completion of logging and soil sampling, each borehole will be converted into a temporary monitoring well, as described above. The samples will be collected using a peristaltic pump, provided that hydraulic head does not exceed 30 to 35 feet. If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples. Water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) will be recorded at the time of sample collection.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

Currently there are no records pertaining to items disposed of in the landfill; however, incidental information suggests that the landfill may contain household and office trash, paint cans, and stainless-steel "spiders" (e.g., equipment used to mix the ammonium picrate). Therefore, two Phase 1 soil borings are proposed in grassy areas adjacent to the golf course club house to assess the potential for vapor intrusion from possible VOC impacts associated with the landfill (see **Figure 11-2**). If VOCs are detected in soils and/or groundwater above screening levels, sub-slab soil gas and/or indoor air will be considered during Phase 2 activities to rule out potential vapor intrusion at the golf course club house.

In the open storage area, the DPT soil borings will be advanced in 5-ft increments until refusal is encountered and logged continuously by an experienced glacial geologist as described above. If shallow refusal is encountered (i.e., less than 5 ft bgs), three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped, and the drilling methodology will be reassessed during Phase 2. Co-located discrete surface soil samples (0 to 1 ft bgs) and subsurface soil samples (1 to 5 ft bgs) soil samples will be collected from each DPT soil boring (see **Figure 11-2**). If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval.. Note that refusal may be in till or weathered shale and groundwater samples may not be attainable at depth in bedrock. If this occurs, the deeper groundwater in bedrock will be targeted during Phase 2 activities, with the installation of permanent monitoring wells.

One groundwater sample will be collected from a DPT soil boring in the Open Storage Area. At refusal, and upon completion of logging and soil sampling, the borehole will be converted into a temporary monitoring well as described above. The sample will be collected using a peristaltic pump, provided that hydraulic head does not exceed 30 to 35 feet. If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples. Water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) will be recorded at the time of sample collection.

Currently there are no records available regarding items stored in the former open storage area.

The following analyses will be performed in AOC 3 during Phase 1:

- Landfill Area
    - Surface soil – TAL metals, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, PCBs, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Subsurface soil – TAL metals, VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, PCBs, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Groundwater (grab samples): TAL metals (field-filtered), VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, and PCBs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).
    - VOCs in subsurface soil adjacent to the golf club house.

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

- Open Storage Area

    - Surface soil – TAL metals, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Subsurface soil – TAL metals, VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Groundwater (grab samples) – TAL metals (field-filtered), VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, and picric acid; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Note that PAHs will be analyzed by 8270D-SIM because project action limits cannot be met without the use of selective ion monitoring (see **Worksheet #15**).

Results of the Phase 1 investigations at AOC 3, including the wetlands delineation and vernal pool assessment, will be integrated into the CSM, in order to refine the model and more precisely direct the design of the RI sampling. This includes strategic placement of future permanent monitoring wells and the development of DUs for ISM sampling to capture any potential releases associated with the former Ammonium Picrate operations. This additional information will be used to enhance the comprehensive human health and ecological risk assessments for AOC 3.

It is possible that metal and/or PAH constituents are present at low concentrations in upgradient and background soil and/or groundwater due to anthropogenic and anthropologic conditions. Site concentrations will be compared with BTVs calculated from project-specific data to evaluate anthropogenic and local anthropologic non-DoD contamination and site contamination.

If the results of the soil and groundwater assessment indicate the potential for discharge of DoD-related constituents, then co-located pore water, surface water, and sediment samples may be collected during Phase 2 to determine whether constituents have migrated into local surface water bodies.

11.5.3 AOC 4 – Former Bunker Area

Each bunker at AOC 4 is equipped with two trench drains that extend along the interior sides and discharge to ground surface at the front of each bunker. In addition, drainage swales extend the length of West and East Igloo Roads.

For surface soil sampling, each bunker will be designated as an individual DU for ISM sampling, and labeled as shown in **Figure 11-3**. Each one-half acre DU begins at the roadway and includes the bunker, bunker aprons, and surrounding cleared area. A systematic grid system will be established within each DU, and will be sampled in triplicate, with each replicate comprised of 30 soil increments. ISM soil increments will be collected from the 0 to 1 ft bgs interval and composited as one bulk sample, for a total of 36 ISM samples (i.e., three replicates for each of the 12 DUs).

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

The remainder of Phase 1 sampling is designed to assess the presence/absence of DoD-related constituents. In Phase 2, DUs may be designed based on the Phase 1 results to determine extent of contamination and assess risk using ISM samples.

Subsurface soil (1 to 5 ft bgs) samples will be collected from each discharge point of the FUDS-eligible bunker trench drains (a total of 24). Additional soil samples (1 to 5 ft bgs) will be collected and spaced equidistant along the length of the drainage swales (45 total samples) (see **Figure 11-3**). If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. All borings along the drainage swales and at the trench rain discharges will be advanced to 5 ft bgs. If shallow refusal is encountered, three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped, and the drilling methodology will be reassessed during Phase 2.

A total of 16 DPT soil borings will be advanced along three transects: eastern, middle, and western portions of AOC 4 (see **Figure 11-3**). The three transects are located to capture potential constituent migration from the bunkers. If possible, multiple zones of groundwater will be sampled in soil borings. Surface soil (0 to 1 ft bgs) and subsurface soil (1 to 5 ft bgs and 5 ft to 10 ft bgs) samples will be collected from each DPT (total of 16 surface and 32 subsurface samples).

- The DPT soil borings will be advanced in 5-ft increments until refusal is encountered and logged continuously by an experienced glacial geologist as described above. Co-located discrete surface (0 to 1 ft bgs) and subsurface (selected from intervals of 1 to 5 ft bgs and 5 to 10 ft bgs) soil samples will be collected from each DPT boring (see **Figure 11-3**). If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval. It should be noted that refusal may occur in till or saprolite (weathered shale), before competent bedrock is encountered. If this occurs, the deeper groundwater in bedrock will be targeted during Phase 2 activities, with the installation of permanent monitoring wells.
- A middle, parallel transect includes three DPT boring locations (see **Figure 11-3**). The locations in the Middle Transect will be sited with the assistance of the local NYSDEC biologist to avoid impacting the existing habitat and limit clearing and grubbing activities.
- One transect is oriented parallel to and west of West Igloo Road on the western limits of AOC 4, along Bunkers 1 through 6, labeled Western Transect (see **Figure 11-3**). A total of six DPT boring locations are included in this transect, five locations are situated directly west of the nearest bunker. One DPT location is sited at the southern end of the Western Transect to determine if groundwater discharges to the surface water body to the southwest. The locations associated with the Western Transect are proposed along the edge of the currently cleared area behind each bunker, again to avoid impacting the existing habitat. Bunker 3 is excluded because NYSDEC beneficially re-used this bunker and performed remediation activities to address pesticide release(s); therefore, it is not FUDS-eligible (USACE, 1991).
- One transect is oriented parallel to and east of East Igloo Road along the eastern limits of AOC 4, along Bunkers 7 through 14, labeled the Eastern Transect (see **Figure 11-3**). Seven DPT boring locations are located on the Eastern Transect. The locations are proposed on the eastern side of Igloo Road in the toe of the gravel apron of the bunkers to further avoid

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

impacting the existing habitat. Bunker 8 is excluded because NYSDEC beneficially re-used this bunker and performed remediation activities to address pesticide release(s); therefore, it is not FUDS-eligible (USACE, 1991).

Up to 16 groundwater samples will be collected from the 16 DPT soil borings. At refusal, and upon completion of logging and soil sampling, each borehole will be converted into a temporary monitoring well as described above. The samples will be collected using a peristaltic pump, provided that hydraulic head does not exceed 30 to 35 feet. If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples. Water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) will be recorded at the time of sample collection.

The following analyses will be performed in AOC 4 during Phase 1:

- Surface soil: DNCB and 2,4-DNP, picric acid; (refer to **Section 11.5.5**).
- Subsurface soil: DNCB and 2,4-DNP, picric acid; (refer to **Section 11.5.5**).
- Groundwater (grab samples): DNCB and 2,4-DNP, and picric acid; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Results of the Phase 1 investigations at AOC 4, including the wetlands delineation and vernal pool assessment, will be integrated into the CSM, in order to refine the model and more precisely direct the design of the Phase 2 RI sampling. This includes strategic placement of the permanent monitoring wells and the development of DUs for additional ISM sampling (if necessary) to capture any potential releases associated with the former Ammonium Picrate operations. This additional information will be used to enhance the comprehensive human health and ecological risk assessments for AOC 4.

Additional ISM sampling will be designed (DU size, number, locations, etc.) based on the discrete soil sample results. Based on the results of the DPT investigation, locations will be selected/refined for the installation of permanent monitoring wells. If the groundwater assessment indicates the potential for discharge into neighboring wetlands, co-located pore water, surface water, and sediment samples will be collected to determine whether constituents have migrated to the wetlands.

11.5.4 AOC 5 – Former Power Plant Area

Phase 1 sampling is designed to assess the presence/absence of DoD-related constituents. In Phase 2, DUs may be designed based on the Phase 1 results to determine extent of contamination and assess risk using ISM samples.

A total of 25 DPT soil borings will be advanced in the vicinity of the former Power Plant (see **Figure 11-4**). Grab groundwater samples will be collected from 12 borings to assess groundwater flow and potential DoD-related chemical transport to wetlands west of the coal storage area (see **Figure 11-4**). If possible, groundwater will be sampled in soil borings.

The DPT soil borings will be advanced in 5-ft increments and logged continuously by an experienced glacial geologist until refusal is encountered. If shallow refusal is encountered (i.e., less than 5 ft bgs), three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped and the drilling

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

methodology will be reassessed during Phase 2. Five co-located discrete surface (0 to 1 ft bgs) and subsurface soil samples (1 to 5 ft bgs and 5 to 10 ft bgs) each will be collected from the boiler house area, substation area, coal storage area, possible dump area, and coal aggregate bins for a total of the 25 borings to determine potential residual constituents within the former power plant area (see **Figure 11-4**). If field observations indicate impacts (i.e., elevated readings on the PID, staining, and/or odors), then a sample will be collected from that impacted interval.

Up to 12 groundwater samples will be collected from the 12 DPT borings. At refusal, and upon completion of logging and soil sampling, each borehole will be converted into a temporary monitoring well as described above. The samples will be collected using a peristaltic pump, provided that hydraulic head does not exceed 30 to 35 feet. If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples. Water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) will be recorded at the time of sample collection.

The following analyses will be performed at AOC 5 during Phase 1:

- Boiler House – 5 soil and 2 groundwater locations as follows:
    - Surface soil: TAL metals, SVOCs, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Subsurface soil: TAL metals, SVOCs, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).
- Coal Storage Area – 5 soil and 3 groundwater locations as follows:
    - Surface soil: coal ash (by microscopy), TAL metals, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Subsurface soil: TAL metals, SVOCs, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).
- Sub-Station – 5 soil and 2 groundwater locations as follows:
    - Surface soil: TAL metals, SVOCs, PAHs, and PCBs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Subsurface soil: TAL metals, SVOCs, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
    - Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).
- Possible Dump – 5 soil and 3 groundwater locations as follows:

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

- Surface soil: TAL metals, SVOCs, PAHs, and PCBs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
- Subsurface soil: TAL metals, VOCs, SVOCs, PAHs, and PCBs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
- Groundwater (grab samples): TAL metals (field-filtered), VOCs, SVOCs, PAHs, and PCBs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

- Coal Aggregate Bins – 5 soil and 2 groundwater locations as follows:

  - Surface soil: coal ash (by microscopy), TAL metals, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
  - Subsurface soil: TAL metals, SVOCs, and PAHs; subset for TOC, grain size distribution (including hydrometer analysis), and CEC (refer to **Section 11.5.5**).
  - Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Note that PAHs will be analyzed by 8270D-SIM because project action limits cannot be met without the use of the selective ion monitoring (see **Worksheet #15**).

Given the nature of picric acid, it would not have been DoD's practice to dispose of it at the dump, nor is there any evidence that it was ever manufactured or stored at AOC 5; therefore, the sampling program does not include picric acid or any of its precursor's at AOC 5. Also, because there is insufficient evidence that indicates pesticides were released or not applied per their intended use, sampling for pesticides is not warranted under CERCLA in the potential dump area.

Results of the Phase 1 investigations at AOC 5, including the wetlands delineation and vernal pool assessment, will be incorporated into the CSM. The findings of the DPT investigation will be used to refine the CSM, and subsequently the design of the RI sampling approach. This includes strategic placement of the permanent monitoring wells and the development of DUs for ISM sampling to capture any potential releases associated with former Power Plant operations. This additional information will be used to improve the comprehensive human health and ecological risk assessments for AOC 5. If, based on the Phase 1 sampling, it appears as though a release occurred that could convey constituents to drainage and wastewater ditches via migration pathways such as runoff and/or leaching, drainage ditches may potentially be sampled during Phase 2 activities.

If the groundwater assessment indicates the potential for discharge of DoD-related constituents into neighboring wetlands, then co-located pore water, surface water, and sediment samples will be collected to determine whether constituents have migrated to the wetlands. If needed, the pore water, surface water and sediment sampling program at AOC 5 will be designed based on the initial groundwater sampling analytical results and groundwater flow analysis.

11.5.5 Background

As detailed in **Section 11.3**, AOC 1 has three distinct surface geologic units (silt and clay; stratified silt, sand and gravel; and diamicton), AOC 3 has the same surface geologic unit (silt and clay), and AOC 5

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

has two distinct surface geologic units (silt and clay, and diamicton). Background for AOC 1, 3, and AOC 5 will be collected from the Radisson Community Association property. In addition, surficial soil will not be collected from landscaped (i.e., re-worked) or from fill material directly under the asphalt pavement. Subsurface samples will be collected in native material only.

A total of 15 background sample locations will be selected from each of the three major soil types (refer to **Figure 11-5**) and installed to refusal. If shallow refusal is encountered (i.e., less than 5 ft bgs), three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped, and the drilling methodology will be reassessed during Phase 2.

Each background location will be sampled at three depths: the surface interval (0 to 1 ft bgs) and two subsurface intervals (1 to 5 ft and 5 to 10 ft), for a total of 135 background samples. All background samples will be analyzed for TAL metals, PAHs, TOC, grain size distribution (including hydrometer analysis), and CEC.

Based on the results of the TOC and grain size distribution (including hydrometer analysis), background samples that are identified as similar matches will be submitted for chemical analysis, provided laboratory holding times have not been exceeded. It is assumed that a maximum of 60 grain size samples can be processed simultaneously within the standard 10-day turn-around period. Preliminary grain size distribution data may be available as early as five to seven business days. As such, grain size analysis will proceed upon sample receipt by the laboratory, while soil/water volumes for chemical analysis will be preserved as necessary and held until Bluestone completes statistical analyses on preliminary grain size results. Approved samples or 'matches', will be selected for chemical analysis. In the event of a delay due to laboratory backlog, Bluestone will request that the laboratory proceed with chemical analysis within holding times, regardless of the status of corresponding grain size analysis. If the initial comparison does not yield 15 matches per AOC during the first round of sampling, a second round will be completed in an attempt to establish 15 viable samples; however, no more than two rounds of sampling will be completed. Background samples must represent regional surficial geology to the respective AOC surface and subsurface media.

Site soil and groundwater concentrations will be compared with background concentrations using summary statistics (e.g., ranges, means, medians, standard deviations), exploratory graphical displays (e.g., side-by-side box plots, histograms, and quantile-quantile plots), and if sufficient data exist, BTVs will be calculated. Summary statistics and exploratory graphical displays may be used for sites with small data sets (n<10); however, in the case of AOC 5 where the investigation is more of an initial Site Investigation (SI), any comparisons with background would be for informational purposes and not used to eliminate methods and/or analytes from further investigation. Lastly, AOC 1 results from the six production lines will be grouped for background comparisons.

The following analyses will be performed for background sampling:

- Discrete surface soil samples at each of the three selected background areas for comparison to individual AOC discrete surface soil samples - 15 sample locations (at a depth of 0 to 1 ft bgs), to be analyzed for TAL metals, PAHs, TOC, and grain size distribution (including hydrometer analysis).

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

- Discrete subsurface soil samples of each of the three selected background areas for comparison to individual AOC discrete subsurface soil samples - 15 sample locations (at depths of 1 to 5 ft bgs and 5 to 10 ft bgs), to be analyzed for TAL metals, PAHs, TOC, and grain size distribution (including hydrometer analysis).
- Groundwater samples will be collected from a total of nine (9) DPT soil borings, three (3) upgradient locations for each of the applicable AOCs, i.e., AOC 1, AOC 3, and AOC 5. Background groundwater samples will be analyzed for TAL metals (field-filtered), PAHs, and water quality field parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity).

11.6 PERFORMANCE OR ACCEPTANCE CRITERIA

Requirements for analytical sensitivity are presented in **Worksheet #15**. Requirements for precision, accuracy, completeness, and comparability are presented in **Worksheets #12, 28, 36, and 37**.

11.7 PLAN FOR OBTAINING DATA

Phase 1 work will be performed at NYOW AOCs 1, 3, 4, 5, and selected background locations (see **Figures 11-1 through 11-5**). The field work and sampling activities are tentatively scheduled for Fall 2020. Sampling design and rationale are based on data gaps identified in **Worksheet #10** and study goals listed above. Additional detail regarding sampling design is presented in **Worksheet #17**. In summary, the following methods will be used for sample/data collection:

- Soils:
    - Surface ISM triplicate sample sets will be collected in AOC 4, from 12 DUs, one for each bunker, for a total of 36 ISM samples. Surface soil increments will be collected from the 0 to 1 ft bgs interval.
    - Co-located surface soil and subsurface soil samples will be collected from soil cores extracted using DPT drilling methods. Surface soil samples include the 0 to 1 ft bgs interval; subsurface soil samples include the 1 to 5 ft bgs and 5 to 10 ft bgs intervals or where impacts (i.e., elevated readings on the PID, staining, and/or odors) are observed except at the background locations.

- Discrete grab groundwater samples will be collected from the temporary monitoring wells using a peristaltic pump, provided that hydraulic head does not exceed 30 to 35 feet. If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples.
- Water quality parameters (i.e., temperature, pH, specific conductance, DO, ORP, and turbidity) will be recorded during the collection of samples from a temporary monitoring well, using a multi-parameter meter.

Field data will be recorded using field forms, logbooks, and other hard copy deliverables. Any significant observations made by field personnel regarding findings and/or visual impacts (i.e., buried objects, elevated PID measurements, heavy staining, odors, etc.) will be conveyed to the USACE PM

QAPP Worksheet #11: Project/Data Quality Objectives, Continued

immediately. Work activities will be temporarily halted, and field crews will await direction from USACE on how to proceed.

The laboratories will provide analytical data in a Staged Electronic Data Deliverable (SEDD) version 5.2, and electronic copies of Level 4 data packages per the most recent USEPA National Functional Guidelines (NFG) and meeting NYSDEC requirements for all parameters, plus sample chromatograms for chromatographic methods and instrument calibrations (see **Worksheet #35** for details).

Worksheets #19/30, 20, and 24-28 specify analysis design requirements.

QAPP Worksheet #12: Measurement Performance Criteria – General and Contents
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Quality criteria from QSM Table Cs were presented when available, else laboratory-specific quality criteria were used to populate this Worksheet.

- Solid (Discrete) – SW8330A
- Solid (ISM) – SW8330B
- Aqueous – SW8330B
- Solid – SW9060A
- Solid – SW9081
- Solid – D2216
- Aqueous – SW7470A
- Solid – SW7471B
- Solid – SW8082A
- Aqueous – SW8082A
- Solid – SW8270D-SIM
- Aqueous – SW8270D-SIM
- Solid – SW8270D
- Aqueous – SW8270D
- Solid – SW6010C
- Aqueous – SW6010C
- Solid – SW6020A
- Aqueous – SW6020A
- Solid – SW8260C
- Aqueous – SW8260C

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid - Discrete**

Analytical Group or Method: **Explosives (SW8330A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Analytical Precision (laboratory)	Confirmation Column Difference	RPD ≤ 40%
	Lab Control Sample Duplicate	RPD ≤ 30%
	Lab Replicate	RPD ≤ 20%
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	Picric acid: 50 - 150%
Analytical Accuracy (matrix interference)	Matrix Spike	Picric acid: 50 - 150%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 30%
Analytical Accuracy	Surrogate	1,2-Dinitrobenzene: 89 - 123%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid - ISM**

Analytical Group or Method: **Explosives (SW8330B)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Analytical Precision (laboratory)	Confirmation Column Difference	RPD ≤ 40%
	Lab Control Sample Duplicate	RPD ≤ 30%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (laboratory)	Lab Control Sample	Picric acid: 38 - 154%
Analytical Accuracy (matrix interference)	Matrix Spike	Picric acid: 38 - 154%
Analytical Accuracy	Surrogate	1,2-Dinitrobenzene: 78 - 119%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Explosives (SW8330B)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Analytical Precision (laboratory)	Confirmation Column Difference	RPD ≤ 40%
	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	Picric acid: 80 - 120%
Analytical Accuracy (matrix interference)	Matrix Spike	Picric acid: 80 - 120%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%
Analytical Accuracy	Surrogate	1,2-Dinitrobenzene: 83 - 119%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **General Chemistry (SW9060A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	46 - 130%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	46 - 130%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **General Chemistry (SW9081)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Precision (laboratory)	Lab Replicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **General Chemistry Percent Solids/Moisture (D2216)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Mercury (SW7470A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	82 - 119%
	Low Level Calibration Check Verification	80 - 120%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	82 - 119%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Mercury (SW7471B)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	80 - 124%
	Low Level Calibration Check Verification	80 - 120%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	80 - 124%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Polychlorinated Biphenyls (SW8082A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Analytical Precision (laboratory)	Confirmation Column Difference	RPD ≤ 40%
	Lab Control Sample Duplicate	RPD ≤ 30%
	Lab Replicate	RPD ≤ 30%
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	PCB-1016 (Aroclor 1016): 47 - 134%
		PCB-1260 (Aroclor 1260): 53 - 140%
Analytical Accuracy (matrix interference)	Matrix Spike	PCB-1016 (Aroclor 1016): 47 - 134%
		PCB-1260 (Aroclor 1260): 53 - 140%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 30%
Analytical Accuracy	Surrogate	2,4,5,6-Tetrachloro-meta-xylene: 44 - 130%
		Decachlorobiphenyl: 59 - 130%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Polychlorinated Biphenyls (SW8082A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Analytical Precision (laboratory)	Confirmation Column Difference	RPD ≤ 40%
	Lab Control Sample Duplicate	RPD ≤ 30%
	Lab Replicate	RPD ≤ 30%
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	PCB-1016 (Aroclor 1016): 46 - 129%
		PCB-1260 (Aroclor 1260): 45 - 134%
Analytical Accuracy (matrix interference)	Matrix Spike	PCB-1016 (Aroclor 1016): 46 - 129%
		PCB-1260 (Aroclor 1260): 45 - 134%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 30%
Analytical Accuracy	Surrogate	2,4,5,6-Tetrachloro-meta-xylene: 25 - 120%
		Decachlorobiphenyl: 30 - 136%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (Method 8270D SIM)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	1-Methylnaphthalene: 43 - 111%
		2-Methylnaphthalene: 39 - 114%
		Acenaphthene: 44 - 111%
		Acenaphthylene: 39 - 116%
		Anthracene: 50 - 114%
		Benzo(a)anthracene: 54 - 122%
		Benzo(a)pyrene: 50 - 125%
		Benzo(b)fluoranthene: 53 - 128%
		Benzo(g,h,i)perylene: 49 - 127%
		Benzo(k)fluoranthene: 56 - 123%
		Chrysene: 57 - 118%
		Dibenz(a,h)anthracene: 50 - 129%
		Fluoranthene: 55 - 119%
		Fluorene: 47 - 114%
		Indeno(1,2,3-c,d)pyrene: 49 - 130%
		Naphthalene: 38 - 111%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (Method 8270D SIM)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Phenanthrene: 49 - 113%
		Pyrene: 55 - 117%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 40%
	Lab Replicate	RPD ≤ 40%
Analytical Accuracy (matrix interference)	Matrix Spike	1-Methylnaphthalene: 43 - 111%
		2-Methylnaphthalene: 39 - 114%
		Acenaphthene: 44 - 111%
		Acenaphthylene: 39 - 116%
		Anthracene: 50 - 114%
		Benzo(a)anthracene: 54 - 122%
		Benzo(a)pyrene: 50 - 125%
		Benzo(b)fluoranthene: 53 - 128%
		Benzo(g,h,i)perylene: 49 - 127%
		Benzo(k)fluoranthene: 56 - 123%
		Chrysene: 57 - 118%
		Dibenz(a,h)anthracene: 50 - 129%
		Fluoranthene: 55 - 119%
		Fluorene: 47 - 114%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (Method 8270D SIM)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Indeno(1,2,3-c,d)pyrene: 49 - 130%
		Naphthalene: 38 - 111%
		Phenanthrene: 49 - 113%
		Pyrene: 55 - 117%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 40%
Analytical Accuracy	Surrogate	2-Fluorobiphenyl: 46 - 115%
		Nitrobenzene-d5: 44 - 125%
		Terphenyl-d14: 58 - 133%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (Method 8270D SIM)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	1-Methylnaphthalene: 41 - 115%
		2-Methylnaphthalene: 39 - 114%
		Acenaphthene: 48 - 114%
		Acenaphthylene: 35 - 121%
		Anthracene: 53 - 119%
		Benzo(a)anthracene: 59 - 120%
		Benzo(a)pyrene: 53 - 120%
		Benzo(b)fluoranthene: 53 - 126%
		Benzo(g,h,i)perylene: 44 - 128%
		Benzo(k)fluoranthene: 54 - 125%
		Chrysene: 57 - 120%
		Dibenz(a,h)anthracene: 44 - 131%
		Fluoranthene: 58 - 120%
		Fluorene: 50 - 118%
		Indeno(1,2,3-c,d)pyrene: 48 - 130%
		Naphthalene: 43 - 114%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (Method 8270D SIM)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Phenanthrene: 53 - 115%
		Pyrene: 53 - 121%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 40%
	Lab Replicate	RPD ≤ 40%
Analytical Accuracy (matrix interference)	Matrix Spike	1-Methylnaphthalene: 41 - 115%
		2-Methylnaphthalene: 39 - 114%
		Acenaphthene: 48 - 114%
		Acenaphthylene: 35 - 121%
		Anthracene: 53 - 119%
		Benzo(a)anthracene: 59 - 120%
		Benzo(a)pyrene: 53 - 120%
		Benzo(b)fluoranthene: 53 - 126%
		Benzo(g,h,i)perylene: 44 - 128%
		Benzo(k)fluoranthene: 54 - 125%
		Chrysene: 57 - 120%
		Dibenz(a,h)anthracene: 44 - 131%
		Fluoranthene: 58 - 120%
		Fluorene: 50 - 118%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (Method 8270D SIM)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Indeno(1,2,3-c,d)pyrene: 48 - 130%
		Naphthalene: 43 - 114%
		Phenanthrene: 53 - 115%
		Pyrene: 53 - 121%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 40%
Analytical Accuracy	Surrogate	2-Fluorobiphenyl: 53 - 106%
		Nitrobenzene-d5: 55 - 111%
		Terphenyl-d14: 58 - 132%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	1,2,4,5-Tetrachlorobenzene: 37 - 119%
		1,4-Dioxane (p-Dioxane): 14 - 130%
		2,2'-Oxybis(1-chloropropane): 33 - 131%
		2,3,4,6-Tetrachlorophenol: 44 - 125%
		2,4,5-Trichlorophenol: 41 - 124%
		2,4,6-Trichlorophenol: 39 - 126%
		2,4-Dichlorophenol: 40 - 122%
		2,4-Dimethylphenol: 30 - 127%
		2,4-Dinitrochlorobenzene: 10 - 130%
		2,4-Dinitrophenol: 10 - 130%
		2,4-Dinitrotoluene: 48 - 126%
		2,6-Dinitrotoluene: 46 - 124%
		2-Chloronaphthalene: 41 - 114%
		2-Chlorophenol: 34 - 121%
		2-Methylphenol (o-Cresol): 32 - 122%
		2-Nitroaniline: 44 - 127%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	2-Nitrophenol: 36 - 123%
		3,3'-Dichlorobenzidine: 22 - 121%
		3-Nitroaniline: 33 - 119%
		4,6-Dinitro-2-methylphenol: 29 - 132%
		4-Bromophenyl phenyl ether: 46 - 124%
		4-Chloro-3-methylphenol: 45 - 122%
		4-Chloroaniline: 17 - 106%
		4-Chlorophenyl phenyl ether: 45 - 121%
		4-Nitroaniline: 41 - 130%
		4-Nitrophenol: 30 - 132%
		Acetophenone: 33 - 115%
		Atrazine: 47 - 127%
		Benzaldehyde: 10 - 130%
		Benzyl butyl phthalate: 48 - 132%
		Biphenyl (Diphenyl): 40 - 117%
		Bis(2-chloroethoxy) methane: 36 - 121%
		Bis(2-chloroethyl) ether (2-Chloroethyl ether): 31 - 120%
		Bis(2-ethylhexyl) phthalate: 51 - 133%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Caprolactam: 46 - 117%
		Carbazole: 50 - 123%
		Cresols, m- & p-: 34 - 119%
		Dibenzofuran: 44 - 120%
		Diethyl phthalate: 50 - 124%
		Dimethyl phthalate: 48 - 124%
		Di-n-butyl phthalate: 51 - 128%
		di-n-Octyl phthalate: 45 - 140%
		Hexachlorobenzene: 45 - 122%
		Hexachlorobutadiene: 32 - 123%
		Hexachlorocyclopentadiene: 28 - 130%
		Hexachloroethane: 28 - 117%
		Isophorone: 30 - 122%
		Nitrobenzene: 34 - 122%
		N-Nitrosodi-n-propylamine: 36 - 120%
		N-Nitrosodiphenylamine: 38 - 127%
		Pentachlorophenol: 25 - 133%
		Phenol: 34 - 121%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	1,2,4,5-Tetrachlorobenzene: 37 - 119%
		1,4-Dioxane (p-Dioxane): 14 - 130%
		2,2'-Oxybis(1-chloropropane): 33 - 131%
		2,3,4,6-Tetrachlorophenol: 44 - 125%
		2,4,5-Trichlorophenol: 41 - 124%
		2,4,6-Trichlorophenol: 39 - 126%
		2,4-Dichlorophenol: 40 - 122%
		2,4-Dimethylphenol: 30 - 127%
		2,4-Dinitrochlorobenzene: 10 - 130%
		2,4-Dinitrophenol: 10 - 130%
		2,4-Dinitrotoluene: 48 - 126%
		2,6-Dinitrotoluene: 46 - 124%
		2-Chloronaphthalene: 41 - 114%
		2-Chlorophenol: 34 - 121%
		2-Methylphenol (o-Cresol): 32 - 122%
		2-Nitroaniline: 44 - 127%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	2-Nitrophenol: 36 - 123%
		3,3'-Dichlorobenzidine: 22 - 121%
		3-Nitroaniline: 33 - 119%
		4,6-Dinitro-2-methylphenol: 29 - 132%
		4-Bromophenyl phenyl ether: 46 - 124%
		4-Chloro-3-methylphenol: 45 - 122%
		4-Chloroaniline: 17 - 106%
		4-Chlorophenyl phenyl ether: 45 - 121%
		4-Nitroaniline: 41 - 130%
		4-Nitrophenol: 30 - 132%
		Acetophenone: 33 - 115%
		Atrazine: 47 - 127%
		Benzaldehyde: 10 - 130%
		Benzyl butyl phthalate: 48 - 132%
		Biphenyl (Diphenyl): 40 - 117%
		Bis(2-chloroethoxy) methane: 36 - 121%
		Bis(2-chloroethyl) ether (2-Chloroethyl ether): 31 - 120%
		Bis(2-ethylhexyl) phthalate: 51 - 133%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Caprolactam: 46 - 117%
		Carbazole: 50 - 123%
		Cresols, m- & p-: 34 - 119%
		Dibenzofuran: 44 - 120%
		Diethyl phthalate: 50 - 124%
		Dimethyl phthalate: 48 - 124%
		Di-n-butyl phthalate: 51 - 128%
		di-n-Octyl phthalate: 45 - 140%
		Hexachlorobenzene: 45 - 122%
		Hexachlorobutadiene: 32 - 123%
		Hexachlorocyclopentadiene: 28 - 130%
		Hexachloroethane: 28 - 117%
		Isophorone: 30 - 122%
		Nitrobenzene: 34 - 122%
		N-Nitrosodi-n-propylamine: 36 - 120%
		N-Nitrosodiphenylamine: 38 - 127%
		Pentachlorophenol: 25 - 133%
		Phenol: 34 - 121%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%
Analytical Accuracy	Surrogate	2,4,6-Tribromophenol: 39 - 132%
		2-Fluorobiphenyl: 44 - 115%
		2-Fluorophenol: 35 - 115%
		Nitrobenzene-d5: 37 - 122%
		Phenol-d5: 33 - 122%
		Terphenyl-d14: 54 - 127%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	1,2,4,5-Tetrachlorobenzene: 35 - 121%
		1,4-Dioxane (p-Dioxane): 30 - 76%
		2,2'-Oxybis(1-chloropropane): 37 - 130%
		2,3,4,6-Tetrachlorophenol: 50 - 128%
		2,4,5-Trichlorophenol: 53 - 123%
		2,4,6-Trichlorophenol: 50 - 125%
		2,4-Dichlorophenol: 47 - 121%
		2,4-Dimethylphenol: 31 - 124%
		2,4-Dinitrochlorobenzene: 51 - 130%
		2,4-Dinitrophenol: 23 - 143%
		2,4-Dinitrotoluene: 57 - 128%
		2,6-Dinitrotoluene: 57 - 124%
		2-Chloronaphthalene: 40 - 116%
		2-Chlorophenol: 38 - 117%
		2-Methylphenol (o-Cresol): 30 - 117%
		2-Nitroaniline: 55 - 127%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	2-Nitrophenol: 47 - 123%
		3,3'-Dichlorobenzidine: 27 - 129%
		3-Nitroaniline: 41 - 128%
		4,6-Dinitro-2-methylphenol: 44 - 137%
		4-Bromophenyl phenyl ether: 55 - 124%
		4-Chloro-3-methylphenol: 52 - 119%
		4-Chloroaniline: 33 - 117%
		4-Chlorophenyl phenyl ether: 53 - 121%
		4-Nitroaniline: 44 - 119%
		4-Nitrophenol: 41 - 118%
		Acetophenone: 46 - 118%
		Atrazine: 44 - 142%
		Benzaldehyde: 10 - 152%
		Benzyl butyl phthalate: 53 - 134%
		Biphenyl (Diphenyl): 40 - 117%
		Bis(2-chloroethoxy) methane: 48 - 120%
		Bis(2-chloroethyl) ether (2-Chloroethyl ether): 43 - 118%
		Bis(2-ethylhexyl) phthalate: 55 - 135%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Caprolactam: 30 - 111%
		Carbazole: 60 - 122%
		Cresols, m- & p-: 29 - 110%
		Dibenzofuran: 53 - 118%
		Diethyl phthalate: 56 - 125%
		Dimethyl phthalate: 45 - 127%
		Di-n-butyl phthalate: 59 - 127%
		di-n-Octyl phthalate: 51 - 140%
		Hexachlorobenzene: 53 - 125%
		Hexachlorobutadiene: 22 - 124%
		Hexachlorocyclopentadiene: 10 - 130%
		Hexachloroethane: 21 - 115%
		Isophorone: 42 - 124%
		Nitrobenzene: 45 - 121%
		N-Nitrosodi-n-propylamine: 49 - 119%
		N-Nitrosodiphenylamine: 51 - 123%
		Pentachlorophenol: 35 - 138%
		Phenol: 35 - 101%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	1,2,4,5-Tetrachlorobenzene: 35 - 121%
		1,4-Dioxane (p-Dioxane): 30 - 76%
		2,2'-Oxybis(1-chloropropane): 37 - 130%
		2,3,4,6-Tetrachlorophenol: 50 - 128%
		2,4,5-Trichlorophenol: 53 - 123%
		2,4,6-Trichlorophenol: 50 - 125%
		2,4-Dichlorophenol: 47 - 121%
		2,4-Dimethylphenol: 31 - 124%
		2,4-Dinitrochlorobenzene: 51 - 130%
		2,4-Dinitrophenol: 23 - 143%
		2,4-Dinitrotoluene: 57 - 128%
		2,6-Dinitrotoluene: 57 - 124%
		2-Chloronaphthalene: 40 - 116%
		2-Chlorophenol: 38 - 117%
		2-Methylphenol (o-Cresol): 30 - 117%
		2-Nitroaniline: 55 - 127%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	2-Nitrophenol: 47 - 123%
		3,3'-Dichlorobenzidine: 27 - 129%
		3-Nitroaniline: 41 - 128%
		4,6-Dinitro-2-methylphenol: 44 - 137%
		4-Bromophenyl phenyl ether: 55 - 124%
		4-Chloro-3-methylphenol: 52 - 119%
		4-Chloroaniline: 33 - 117%
		4-Chlorophenyl phenyl ether: 53 - 121%
		4-Nitroaniline: 44 - 119%
		4-Nitrophenol: 41 - 118%
		Acetophenone: 46 - 118%
		Atrazine: 44 - 142%
		Benzaldehyde: 10 - 152%
		Benzyl butyl phthalate: 53 - 134%
		Biphenyl (Diphenyl): 49 - 115%
		Bis(2-chloroethoxy) methane: 48 - 120%
		Bis(2-chloroethyl) ether (2-Chloroethyl ether): 43 - 118%
		Bis(2-ethylhexyl) phthalate: 55 - 135%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Caprolactam: 30 - 111%
		Carbazole: 60 - 122%
		Cresols, m- & p-: 29 - 110%
		Dibenzofuran: 53 - 118%
		Diethyl phthalate: 56 - 125%
		Dimethyl phthalate: 45 - 127%
		Di-n-butyl phthalate: 59 - 127%
		di-n-Octyl phthalate: 51 - 140%
		Hexachlorobenzene: 53 - 125%
		Hexachlorobutadiene: 22 - 124%
		Hexachlorocyclopentadiene: 10 - 130%
		Hexachloroethane: 21 - 115%
		Isophorone: 42 - 124%
		Nitrobenzene: 45 - 121%
		N-Nitrosodi-n-propylamine: 49 - 119%
		N-Nitrosodiphenylamine: 51 - 123%
		Pentachlorophenol: 35 - 138%
		Phenol: 35 - 101%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Semivolatile Organic Compounds (SW8270D)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%
Analytical Accuracy	Surrogate	2,4,6-Tribromophenol: 43 - 140%
		2-Fluorobiphenyl: 44 - 119%
		2-Fluorophenol: 19 - 119%
		Nitrobenzene-d5: 44 - 120%
		Phenol-d5: 27 - 110%
		Terphenyl-d14: 50 - 134%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Trace Metals (SW6010C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	Aluminum: 74 - 119%
		Barium: 83 - 113%
		Beryllium: 83 - 113%
		Calcium: 81 - 116%
		Chromium: 85 - 113%
		Cobalt: 85 - 112%
		Copper: 81 - 117%
		Iron: 81 - 118%
		Lead: 81 - 112%
		Magnesium: 78 - 115%
		Manganese: 84 - 114%
		Nickel: 83 - 113%
		Potassium: 81 - 116%
		Silver: 82 - 112%
		Sodium: 83 - 118%
		Vanadium: 82 - 114%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Trace Metals (SW6010C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Zinc: 82 - 113%
	Low Level Calibration Check Verification	80 - 120%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	Aluminum: 74 - 119%
		Barium: 83 - 113%
		Beryllium: 83 - 113%
		Calcium: 81 - 116%
		Chromium: 85 - 113%
		Cobalt: 85 - 112%
		Copper: 81 - 117%
		Iron: 81 - 118%
		Lead: 81 - 112%
		Magnesium: 78 - 115%
		Manganese: 84 - 114%
		Nickel: 83 - 113%
		Potassium: 81 - 116%
		Silver: 82 - 112%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Trace Metals (SW6010C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Sodium: 83 - 118%
		Vanadium: 82 - 114%
		Zinc: 82 - 113%
	Post Spike	80 - 120%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Trace Metals (SW6010C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	Aluminum: 86 - 115%
		Barium: 88 - 113%
		Beryllium: 89 - 112%
		Cadmium: 88 - 113%
		Calcium: 87 - 113%
		Copper: 86 - 114%
		Iron: 87 - 115%
		Magnesium: 85 - 113%
		Manganese: 90 - 114%
		Nickel: 88 - 113%
		Potassium: 86 - 114%
		Selenium: 83 - 114%
		Silver: 84 - 115%
		Sodium: 87 - 115%
		Vanadium: 90 - 111%
		Zinc: 87 - 115%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Trace Metals (SW6010C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (laboratory)	Low Level Calibration Check Verification	80 - 120%
Analytical Accuracy (matrix interference)	Matrix Spike	Aluminum: 86 - 115%
		Barium: 88 - 113%
		Beryllium: 89 - 112%
		Cadmium: 88 - 113%
		Calcium: 87 - 113%
		Copper: 86 - 114%
		Iron: 87 - 115%
		Magnesium: 85 - 113%
		Manganese: 90 - 114%
		Nickel: 88 - 113%
		Potassium: 86 - 114%
		Selenium: 83 - 114%
		Silver: 84 - 115%
		Sodium: 87 - 115%
		Vanadium: 90 - 111%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Trace Metals (SW6010C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Zinc: 87 - 115%
	Post Spike	80 - 120%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Trace Metals (SW6020A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	Antimony: 72 - 124%
		Arsenic: 82 - 118%
		Cadmium: 84 - 116%
		Selenium: 80 - 119%
		Thallium: 83 - 118%
	Low Level Calibration Check Verification	80 - 120%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	Antimony: 72 - 124%
		Arsenic: 82 - 118%
		Cadmium: 84 - 116%
		Selenium: 80 - 119%
		Thallium: 83 - 118%
	Post Spike	80 - 120%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Trace Metals (SW6020A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	Antimony: 85 - 117%
		Arsenic: 84 - 116%
		Chromium: 85 - 116%
		Cobalt: 86 - 115%
		Lead: 88 - 115%
		Thallium: 82 - 116%
	Low Level Calibration Check Verification	80 - 120%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%
	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	Antimony: 85 - 117%
		Arsenic: 84 - 116%
		Chromium: 85 - 116%
		Cobalt: 86 - 115%
		Lead: 88 - 115%
		Thallium: 82 - 116%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Trace Metals (SW6020A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Trace Metals (SW6020A)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Post Spike	80 - 120%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 50%
Analytical Accuracy (laboratory)	Lab Control Sample	1,1,1-Trichloroethane: 73 - 130%
		1,1,2,2-Tetrachloroethane: 70 - 124%
		1,1,2-Trichloro-1,2,2-trifluoroethane: 66 - 136%
		1,1,2-Trichloroethane: 78 - 121%
		1,1-Dichloroethane: 76 - 125%
		1,1-Dichloroethene: 70 - 131%
		1,2,3-Trichlorobenzene: 66 - 130%
		1,2,4-Trichlorobenzene: 67 - 129%
		1,2-Dibromo-3-chloropropane: 61 - 132%
		1,2-Dibromoethane (EDB): 78 - 122%
		1,2-Dichlorobenzene: 78 - 121%
		1,2-Dichloroethane: 73 - 128%
		1,2-Dichloropropane: 76 - 123%
		1,3-Dichlorobenzene: 77 - 121%
		1,3-Dichloropropane: 77 - 121%
		1,4-Dichlorobenzene: 75 - 120%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	2-Butanone (MEK): 51 - 148%
		2-Hexanone: 53 - 145%
		4-Methyl-2-pentanone (MIBK): 65 - 135%
		Acetone: 36 - 164%
		Benzene: 77 - 121%
		Bromochloromethane: 78 - 125%
		Bromodichloromethane: 75 - 127%
		Bromoform: 67 - 132%
		Bromomethane: 53 - 143%
		Carbon disulfide: 63 - 132%
		Carbon tetrachloride: 70 - 135%
		Chlorobenzene: 79 - 120%
		Chloroethane: 59 - 139%
		Chloroform: 78 - 123%
		Chloromethane: 50 - 136%
		cis-1,2-Dichloroethene: 77 - 123%
		cis-1,3-Dichloropropene: 74 - 126%
		Cyclohexane: 67 - 131%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Dibromochloromethane: 74 - 126%
		Dichlorodifluoromethane: 29 - 149%
		Ethylbenzene: 76 - 122%
		Isopropylbenzene (Cumene): 68 - 134%
		m,p-Xylene: 77 - 124%
		Methyl acetate: 53 - 144%
		Methyl tert-butyl ether (MTBE): 73 - 125%
		Methylcyclohexane: 66 - 133%
		Methylene chloride: 70 - 128%
		o-Xylene: 77 - 123%
		Styrene: 76 - 124%
		Tetrachloroethene (PCE): 73 - 128%
		Toluene: 77 - 121%
		trans-1,2-Dichloroethene: 74 - 125%
		trans-1,3-Dichloropropene: 71 - 130%
		Trichlorofluoromethane: 62 - 140%
		Vinyl chloride: 56 - 135%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	1,1,1-Trichloroethane: 73 - 130%
		1,1,2,2-Tetrachloroethane: 70 - 124%
		1,1,2-Trichloro-1,2,2-trifluoroethane: 66 - 136%
		1,1,2-Trichloroethane: 78 - 121%
		1,1-Dichloroethane: 76 - 125%
		1,1-Dichloroethene: 70 - 131%
		1,2,3-Trichlorobenzene: 66 - 130%
		1,2,4-Trichlorobenzene: 67 - 129%
		1,2-Dibromo-3-chloropropane: 61 - 132%
		1,2-Dibromoethane (EDB): 78 - 122%
		1,2-Dichlorobenzene: 78 - 121%
		1,2-Dichloroethane: 73 - 128%
		1,2-Dichloropropane: 76 - 123%
		1,3-Dichlorobenzene: 77 - 121%
		1,3-Dichloropropane: 77 - 121%
		1,4-Dichlorobenzene: 75 - 120%
		2-Butanone (MEK): 51 - 148%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	2-Hexanone: 53 - 145%
		4-Methyl-2-pentanone (MIBK): 65 - 135%
		Acetone: 36 - 164%
		Benzene: 77 - 121%
		Bromochloromethane: 78 - 125%
		Bromodichloromethane: 75 - 127%
		Bromoform: 67 - 132%
		Bromomethane: 53 - 143%
		Carbon disulfide: 63 - 132%
		Carbon tetrachloride: 70 - 135%
		Chlorobenzene: 79 - 120%
		Chloroethane: 59 - 139%
		Chloroform: 78 - 123%
		Chloromethane: 50 - 136%
		cis-1,2-Dichloroethene: 77 - 123%
		cis-1,3-Dichloropropene: 74 - 126%
		Cyclohexane: 67 - 131%
		Dibromochloromethane: 74 - 126%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Dichlorodifluoromethane: 29 - 149%
		Ethylbenzene: 76 - 122%
		Isopropylbenzene (Cumene): 68 - 134%
		m,p-Xylene: 77 - 124%
		Methyl acetate: 53 - 144%
		Methyl tert-butyl ether (MTBE): 73 - 125%
		Methylcyclohexane: 66 - 133%
		Methylene chloride: 70 - 128%
		o-Xylene: 77 - 123%
		Styrene: 76 - 124%
		Tetrachloroethene (PCE): 73 - 128%
		Toluene: 77 - 121%
		trans-1,2-Dichloroethene: 74 - 125%
		trans-1,3-Dichloropropene: 71 - 130%
		Trichlorofluoromethane: 62 - 140%
		Vinyl chloride: 56 - 135%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%
Analytical Accuracy	Surrogate	1,2-Dichloroethane-d4: 71 - 136%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy	Surrogate	1-Bromo-4-fluorobenzene (4-Bromofluorobenzene): 79 - 119%
		Dibromofluoromethane: 78 - 119%
		Toluene-d8: 85 - 116%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Blanks	No target analyte concentrations > 1/2 LOQ
Overall Precision	Field Duplicate	RPD ≤ 30%
Analytical Accuracy (laboratory)	Lab Control Sample	1,1,1-Trichloroethane: 74 - 131%
		1,1,2,2-Tetrachloroethane: 71 - 121%
		1,1,2-Trichloro-1,2,2-trifluoroethane: 70 - 136%
		1,1,2-Trichloroethane: 80 - 119%
		1,1-Dichloroethane: 77 - 125%
		1,1-Dichloroethene: 71 - 131%
		1,2,3-Trichlorobenzene: 69 - 129%
		1,2,4-Trichlorobenzene: 69 - 130%
		1,2-Dibromo-3-chloropropane: 62 - 128%
		1,2-Dibromoethane (EDB): 77 - 121%
		1,2-Dichlorobenzene: 80 - 119%
		1,2-Dichloroethane: 73 - 128%
		1,2-Dichloropropane: 78 - 122%
		1,3-Dichlorobenzene: 80 - 119%
		1,3-Dichloropropane: 80 - 119%
		1,4-Dichlorobenzene: 79 - 118%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	2-Butanone (MEK): 56 - 143%
		2-Hexanone: 57 - 139%
		4-Methyl-2-pentanone (MIBK): 67 - 130%
		Acetone: 39 - 160%
		Benzene: 79 - 120%
		Bromochloromethane: 78 - 123%
		Bromodichloromethane: 79 - 125%
		Bromoform: 66 - 130%
		Bromomethane: 53 - 141%
		Carbon disulfide: 64 - 133%
		Carbon tetrachloride: 72 - 136%
		Chlorobenzene: 82 - 118%
		Chloroethane: 60 - 138%
		Chloroform: 79 - 124%
		Chloromethane: 50 - 139%
		cis-1,2-Dichloroethene: 78 - 123%
		cis-1,3-Dichloropropene: 75 - 124%
		Cyclohexane: 71 - 130%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample	Dibromochloromethane: 74 - 126%
		Dichlorodifluoromethane: 32 - 152%
		Ethylbenzene: 79 - 121%
		Isopropylbenzene (Cumene): 72 - 131%
		m,p-Xylene: 80 - 121%
		Methyl acetate: 56 - 136%
		Methyl tert-butyl ether (MTBE): 71 - 124%
		Methylcyclohexane: 72 - 132%
		Methylene chloride: 74 - 124%
		o-Xylene: 78 - 122%
		Styrene: 78 - 123%
		Tetrachloroethene (PCE): 74 - 129%
		Toluene: 80 - 121%
		trans-1,2-Dichloroethene: 75 - 124%
		trans-1,3-Dichloropropene: 73 - 127%
		Trichlorofluoromethane: 65 - 141%
		Vinyl chloride: 58 - 137%
Analytical Precision (laboratory)	Lab Control Sample Duplicate	RPD ≤ 20%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Lab Replicate	RPD ≤ 20%
Analytical Accuracy (matrix interference)	Matrix Spike	1,1,1-Trichloroethane: 74 - 131%
		1,1,2,2-Tetrachloroethane: 71 - 121%
		1,1,2-Trichloro-1,2,2-trifluoroethane: 70 - 136%
		1,1,2-Trichloroethane: 80 - 119%
		1,1-Dichloroethane: 77 - 125%
		1,1-Dichloroethene: 71 - 131%
		1,2,3-Trichlorobenzene: 69 - 129%
		1,2,4-Trichlorobenzene: 69 - 130%
		1,2-Dibromo-3-chloropropane: 62 - 128%
		1,2-Dibromoethane (EDB): 77 - 121%
		1,2-Dichlorobenzene: 80 - 119%
		1,2-Dichloroethane: 73 - 128%
		1,2-Dichloropropane: 78 - 122%
		1,3-Dichlorobenzene: 80 - 119%
		1,3-Dichloropropane: 80 - 119%
		1,4-Dichlorobenzene: 79 - 118%
		2-Butanone (MEK): 56 - 143%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	2-Hexanone: 57 - 139%
		4-Methyl-2-pentanone (MIBK): 67 - 130%
		Acetone: 39 - 160%
		Benzene: 79 - 120%
		Bromochloromethane: 78 - 123%
		Bromodichloromethane: 79 - 125%
		Bromoform: 66 - 130%
		Bromomethane: 53 - 141%
		Carbon disulfide: 64 - 133%
		Carbon tetrachloride: 72 - 136%
		Chlorobenzene: 82 - 118%
		Chloroethane: 60 - 138%
		Chloroform: 79 - 124%
		Chloromethane: 50 - 139%
		cis-1,2-Dichloroethene: 78 - 123%
		cis-1,3-Dichloropropene: 75 - 124%
		Cyclohexane: 71 - 130%
		Dibromochloromethane: 74 - 126%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (matrix interference)	Matrix Spike	Dichlorodifluoromethane: 32 - 152%
		Ethylbenzene: 79 - 121%
		Isopropylbenzene (Cumene): 72 - 131%
		m,p-Xylene: 80 - 121%
		Methyl acetate: 56 - 136%
		Methyl tert-butyl ether (MTBE): 71 - 124%
		Methylcyclohexane: 72 - 132%
		Methylene chloride: 74 - 124%
		o-Xylene: 78 - 122%
		Styrene: 78 - 123%
		Tetrachloroethene (PCE): 74 - 129%
		Toluene: 80 - 121%
		trans-1,2-Dichloroethene: 75 - 124%
		trans-1,3-Dichloropropene: 73 - 127%
		Trichlorofluoromethane: 65 - 141%
		Vinyl chloride: 58 - 137%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicate	RPD ≤ 20%
Analytical Accuracy	Surrogate	1,2-Dichloroethane-d4: 81 - 118%

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**

Analytical Group or Method: **Volatile Organic Compounds (SW8260C)**

Concentration Level (if applicable):

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy	Surrogate	1-Bromo-4-fluorobenzene (4-Bromofluorobenzene): 85 - 114%
		Dibromofluoromethane: 80 - 119%
		Toluene-d8: 89 - 112%

QAPP Worksheet #13: Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements for Evaluating Existing Data)

Data Type	Data Source	Data Use Relative to Current Project	Factors affecting the Reliability of Data and Limitations on Data Use
Background	Final Site Inspection Report for NYOW, DERP FUDS Project Number C02NY029003 (Alion, 2008); Evaluation of Possible Hazards of Former NYOW, Radisson New Community, Town of Lysander, New York (D&M, 1981); Defense Environmental Restoration Program, Contamination Evaluation at the Former NYOW, Lysander, New York (M&E, 1990); Site Characterization Study, Three Rivers Facility, New York State Pesticide Storage Sites Project, Contract No. D001889 (NYSDEC, 1990); Operation and Maintenance Plan for the Three Rivers Pesticide Storage Site, Lysander, Onondaga County, Site No (NYSDEC, 1993); Archives Search Report, Findings for the former NYOW, Lysander Township, New York, Project Number C02NY029003 (USACE, 1999a)	Background information	None
Chemical, hydrogeology	Alion, 2008; D&M, 1981; M&E, 1990; NYSDEC, 1990; USACE, 1999a	Boring logs and cross-sections	None
Background, Geology	Alion, 2008; D&M, 1981; M&E, 1990; NYSDEC, 1990; USACE, 1999a	Background information, geological data	None
Chemical, Surface Water Drainage	M&E, 1990; USACE, 1999a	Historical data	Age of the data
Chemical, Background	Alion, 2008; D&M, 1981; M&E, 1990; NYSDEC, 1990; USACE, 1999a	Historical data	Age of the data

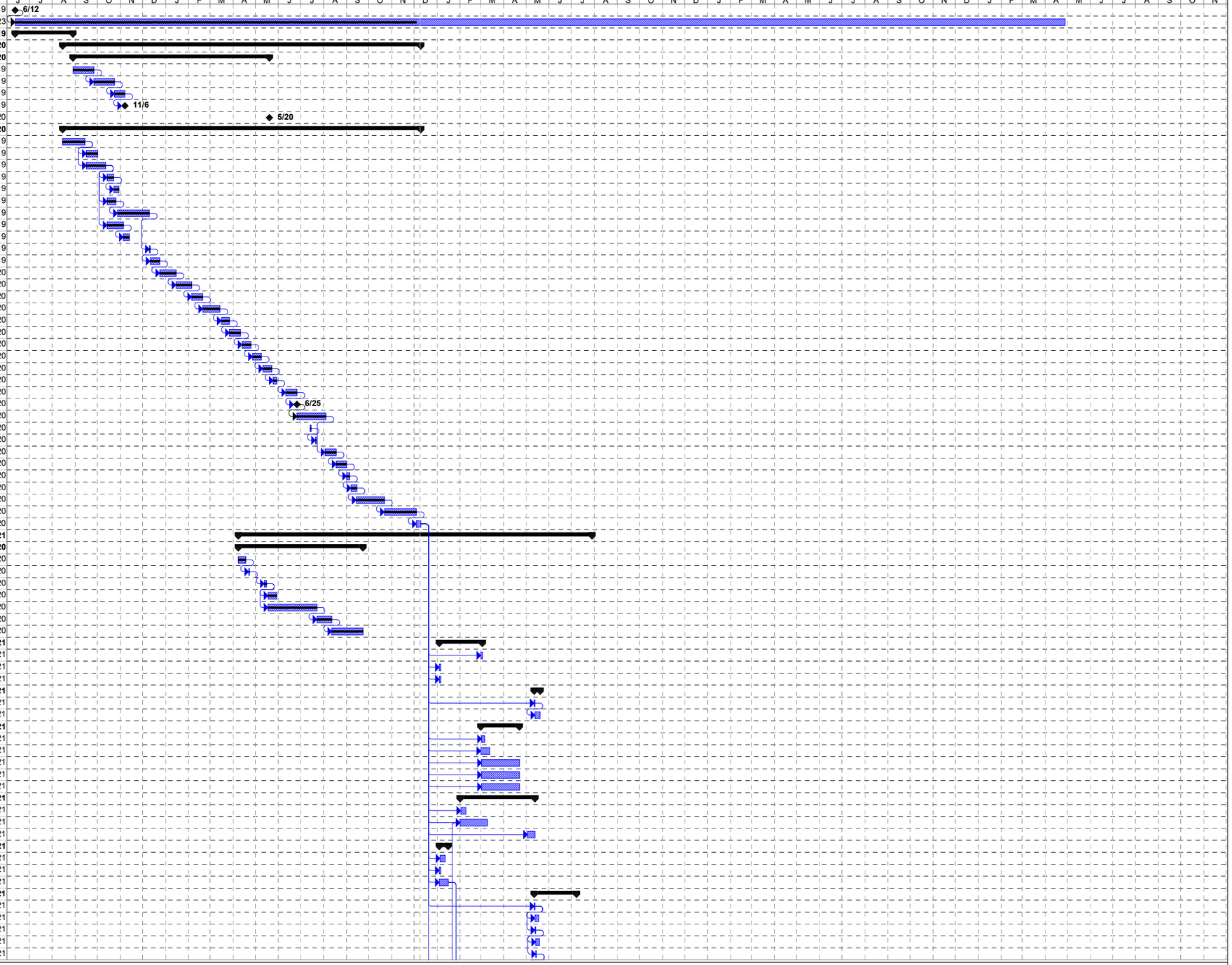
QAPP Worksheet #13: Secondary Data Criteria and Limitations Table, Continued

Data Type	Data Source	Data Use Relative to Current Project	Factors affecting the Reliability of Data and Limitations on Data Use
Photographic	Environmental Risk Information Services (2018); NYOW – Baldwinsville, New York Historical Photographic Analysis (USACE, 2019)	Aerial photographs to document land use	Photographs taken between 1938-2019.
Meteorology	Data Tools website NOAA (2017)	Climatic data	Data was collected from a website.
Land Use and Demography	Factsheet: Empire State Development: Radisson Community Project (Radisson, 2017)	Land use	Data was collected from a website.

QAPP Worksheet #14/16: Project Tasks & Schedule

New York Ordnanace Works RI/FS, PP, DD Schedule

ID	Task Name	Duration	Start	Finish
1	NOTICE TO PROCEED	0 days	Wed 6/12/19	Wed 6/12/19
2	Task 1.1 Project and Financial Management	197.8 wks	Wed 6/12/19	Thu 4/27/23
3	**Task 2. Review of Data, Data Gap Analysis, Coord Meet**	**56 days**	**Wed 6/12/19**	**Wed 8/28/19**
9	**Task 3. Project Plans**	**339 days**	**Thu 8/15/19**	**Wed 12/9/20**
10	**Task 3.1 APP/SSHP**	**185 days**	**Thu 8/29/19**	**Wed 5/20/20**
11	Prepare Client Draft APP/SSHP	20 days	Thu 8/29/19	Wed 9/25/19
12	CENAE Review of APP/SSHP	20 days	Thu 9/26/19	Wed 10/23/19
13	RTC, Prepare Final APP/SSHP	10 days	Thu 10/24/19	Wed 11/6/19
14	Final APP/SSHP Submitted	0 days	Wed 11/6/19	Wed 11/6/19
15	Submit Final APP/SSHP (Mod 2 AOC 5)	0 days	Wed 5/20/20	Wed 5/20/20
16	**Task 3.2-3.4 Work Plan and UFP-QAPP**	**339 days**	**Thu 8/15/19**	**Wed 12/9/20**
17	Prepare RTCs for CX WP Comments	22 days	Thu 8/15/19	Fri 9/13/19
18	Submit Revised WP and UFP-QAPP (Delete AOC 2)	11 days	Mon 9/16/19	Mon 9/30/19
19	CENAE Review of Revised WP	20 days	Mon 9/16/19	Fri 10/11/19
20	Prepare and Submit Revised Wrkshts 3, 5, 6, & 15	7 days	Mon 10/14/19	Tue 10/22/19
21	CENAE Review of Revised Wrkshts 3, 5, 6, & 15	5 days	Wed 10/23/19	Tue 10/29/19
22	Prepare and Submit Revised Wrkshts 10 & 11	10 days	Mon 10/14/19	Fri 10/25/19
23	CENAE Review of Revised Wrkshts 10 & 11	29 days	Mon 10/28/19	Mon 12/9/19
24	Prepare and Submit Revised Figures 4.2 through 4.6	16 days	Mon 10/14/19	Mon 11/4/19
25	CENAE Review of Revised Figures 4.2 through 4.6	5 days	Tue 11/5/19	Tue 11/12/19
26	Submit Draft 10, 11, 15 and figures for meeting	1 day	Tue 12/10/19	Tue 12/10/19
27	Prepare and Resubmit 10, 11, 15 and figures for Review	9 days	Wed 12/11/19	Mon 12/23/19
28	CENAE Review of 10, 11, 15 and figures	14 days	Tue 12/24/19	Tue 1/14/20
29	Prepare and Resubmit Worksheets 10, 11, 15 and Figures	15 days	Wed 1/15/20	Tue 2/4/20
30	CENAE Review of Revised Wrkshts 15 and Figures	11 days	Wed 2/5/20	Wed 2/19/20
31	Prepare and Resubmit Worksheets 10, 11, and 15	17 days	Thu 2/20/20	Fri 3/13/20
32	CENAE Review of Revised Worksheets 10, 11, and 15	9 days	Mon 3/16/20	Thu 3/26/20
33	Submittal of Entire QAPP	11 days	Fri 3/27/20	Fri 4/10/20
34	CENAE Review of Entire QAPP	10 days	Mon 4/13/20	Fri 4/24/20
35	Prepare and submit revised QAPP for backcheck	10 days	Mon 4/27/20	Fri 5/8/20
36	CENAE Backcheck of Revised QAPP Submittal	10 days	Mon 5/11/20	Fri 5/22/20
37	CX Submittal of Revised QAPP	5 days	Mon 5/25/20	Fri 5/29/20
38	CX Review of Revised QAPP	11 days	Thu 6/11/20	Thu 6/25/20
39	Submitted draft RTCs to CENAE	0 days	Thu 6/25/20	Thu 6/25/20
40	Prepare Revised CX QAPP	27 days	Fri 6/26/20	Mon 8/3/20
41	PDT Call to discuss CX RTCs	1 day	Tue 7/14/20	Tue 7/14/20
42	Submitted Revised CX RTCs to CENAE	1 day	Tue 7/21/20	Tue 7/21/20
43	CX Review of Revised QAPP	11 days	Mon 8/3/20	Mon 8/17/20
44	Submit Revised Worksheets 11, 17 and Figures	10 days	Tue 8/18/20	Mon 8/31/20
45	CX Review of WS 11, 17 and Figures	4 days	Tue 9/1/20	Fri 9/4/20
46	RTC, Prepare and Submit Draft Final Plans to Stakeholders	6 days	Mon 9/7/20	Mon 9/14/20
47	Stakeholder Review of Draft Plans	28 days	Mon 9/14/20	Wed 10/21/20
48	Resolve Stakeholder Comments	29 days	Thu 10/22/20	Thu 12/3/20
49	Final QAPP Submitted	4 days	Fri 12/4/20	Wed 12/9/20
50	**Task 4. Field Work/Execute the RI Work Plan**	**335 days**	**Wed 4/8/20**	**Wed 7/28/21**
51	**Wetlands Delineation and Vernal Pool Surveys**	**120 days**	**Wed 4/8/20**	**Tue 9/22/20**
52	Desktop survey	8 days	Wed 4/8/20	Fri 4/17/20
53	Site Recon/Vernal Pool Survey #1	1 day	Wed 4/22/20	Wed 4/22/20
54	Vernal Pool Survey #2	3 days	Wed 5/13/20	Fri 5/15/20
55	Wetlands Delineation	10 days	Mon 5/18/20	Fri 5/29/20
56	Prepare and Submit Draft Report	48 days	Mon 5/18/20	Wed 7/22/20
57	CENAE Review of Draft Report	14 days	Thu 7/23/20	Tue 8/11/20
58	Submit Summary Tech Memo and Final Wetlands Report	30 days	Wed 8/12/20	Tue 9/22/20
59	**Pre-Sampling Investigation**	**42 days**	**Mon 1/4/21**	**Tue 3/2/21**
60	Potable Well Survey	2 days	Mon 3/1/21	Tue 3/2/21
61	AOC MW Assessment	2 days	Mon 1/4/21	Tue 1/5/21
62	Staging Area Setup	2 days	Mon 1/4/21	Tue 1/5/21
63	**AOC 1 Field work**	**6 days**	**Wed 5/12/21**	**Wed 5/19/21**
64	Vegetation Clearance and Utility Markout	1 day	Wed 5/12/21	Wed 5/12/21
65	DPT Sampling	5 days	Thu 5/13/21	Wed 5/19/21
66	**AOC 3 Field Work**	**38 days**	**Mon 3/1/21**	**Wed 4/21/21**
67	Geophysical Survey (AOC 3 and 5)	4 days	Tue 3/2/21	Fri 3/5/21
68	Vegetation Clearance and Utility Markout	10 days	Mon 3/1/21	Fri 3/12/21
69	DPT Sampling at Landfill	37 days	Tue 3/2/21	Wed 4/21/21
70	Soil Vapor Borings and Field Gas Montioring	37 days	Tue 3/2/21	Wed 4/21/21
71	DPT Sampling at Open Storage Area	37 days	Tue 3/2/21	Wed 4/21/21
72	**AOC 4 Field Work**	**73 days**	**Mon 2/1/21**	**Wed 5/12/21**
73	Vegetation Clearance and Utility Markout	5 days	Tue 2/2/21	Mon 2/8/21
74	DPT Sampling	27 days	Mon 2/1/21	Tue 3/9/21
75	ISM Soil Sampling	8 days	Mon 5/3/21	Wed 5/12/21
76	**AOC 5 Field Work**	**10 days**	**Mon 1/4/21**	**Fri 1/15/21**
77	Vegetation Clearance and Utility Markout	5 days	Tue 1/5/21	Mon 1/11/21
78	Geophysical Survey (AOC 3 and 5)	2 days	Mon 1/4/21	Tue 1/5/21
79	DPT Sampling	10 days	Mon 1/4/21	Fri 1/15/21
80	**Background Field Work**	**39 days**	**Wed 5/12/21**	**Wed 7/7/21**
81	Background Location 1 Land Clearing and Utility Clearance	1 day	Wed 5/12/21	Wed 5/12/21
82	DPT Sampling	3 days	Thu 5/13/21	Mon 5/17/21
83	Background Location 2 Land Clearing and Utility Clearance	1 day	Thu 5/13/21	Thu 5/13/21
84	DPT Sampling	3 days	Fri 5/14/21	Tue 5/18/21
85	Background Location 3 Land Clearing and Utility Clearance	1 day	Fri 5/14/21	Fri 5/14/21

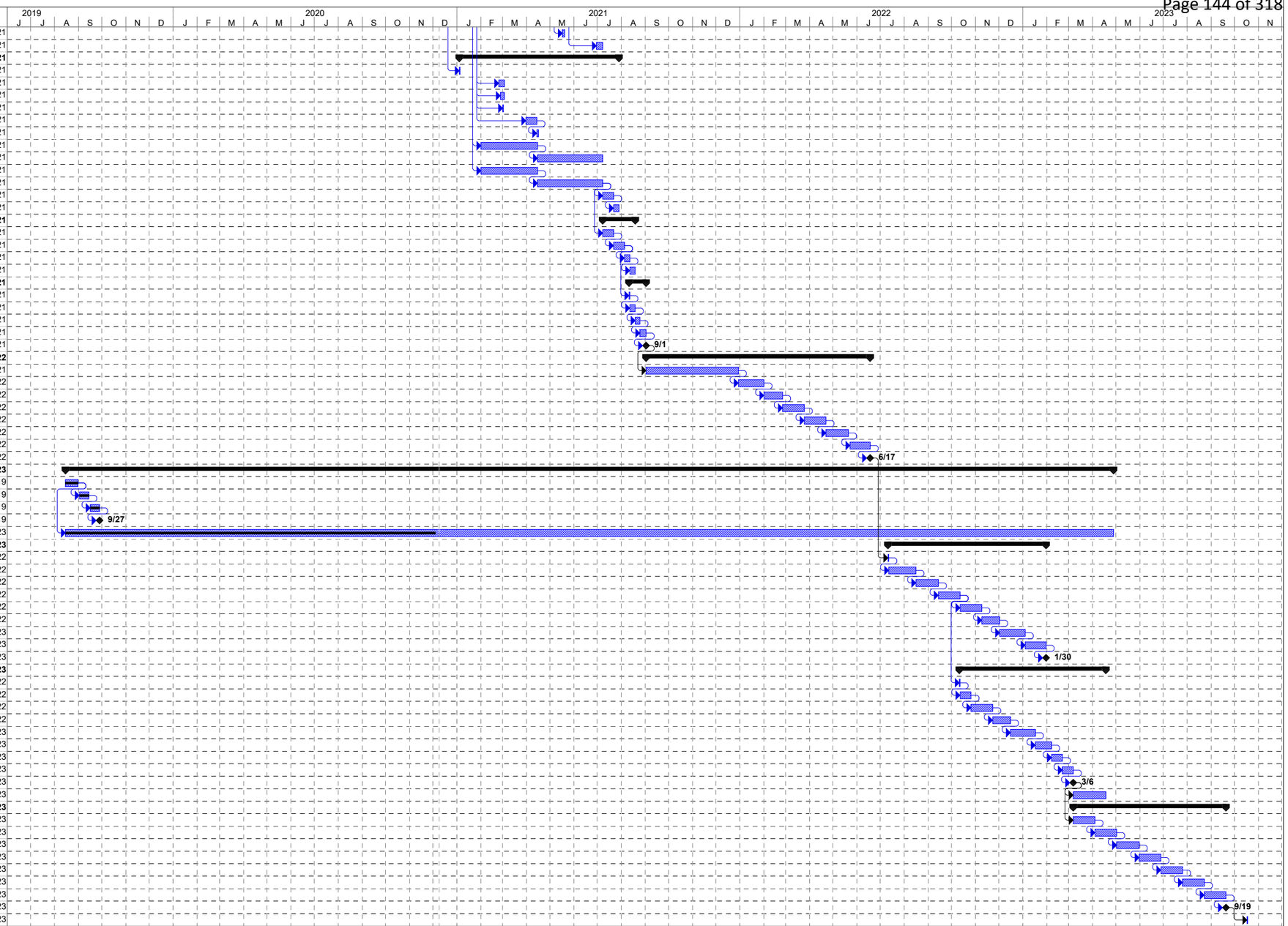


All durations are in work-days
Date: Wed 12/9/20

Task | Milestone | Summary | Project Summary | Progress

New York Ordnanace Works RI/FS, PP, DD Schedule

ID	Task Name	Duration	Start	Finish
86	DPT Sampling	3 days	Mon 5/17/21	Wed 5/19/21
87	Groundwater Survey	5 days	Wed 6/30/21	Wed 7/7/21
88	**All AOC Field Work Tasks**	**146 days**	**Mon 1/4/21**	**Wed 7/28/21**
89	Existing Well Assessment	1 day	Mon 1/4/21	Mon 1/4/21
90	Well Survey	5 days	Wed 2/24/21	Tue 3/2/21
91	GW Elevation Survey - Site	3 days	Fri 2/26/21	Tue 3/2/21
92	IDW Disposal Sampling	1 day	Mon 3/1/21	Mon 3/1/21
93	Temp MW Abandonment - Site	10 days	Wed 3/31/21	Tue 4/13/21
94	Demob Staging Area/IDW Disposal	2 days	Wed 4/14/21	Thu 4/15/21
95	Soil Sample Lab Analysis - 15 day TAT	53 days	Mon 2/1/21	Wed 4/14/21
96	Soil Sample Data Validation - 29 day TAT	58 days	Thu 4/15/21	Wed 7/7/21
97	DPT GW Sample Lab Analysis - 15 day TAT	53 days	Mon 2/1/21	Wed 4/14/21
98	DPT GW Sample Data Validation – 28 day TAT	58 days	Thu 4/15/21	Wed 7/7/21
99	DPT GW Data Summary and MW Recommendations	10 days	Thu 7/8/21	Wed 7/21/21
100	Data Summary Review, Call/Meeting	5 days	Thu 7/22/21	Wed 7/28/21
101	**Field Report**	**30 days**	**Thu 7/8/21**	**Wed 8/18/21**
102	Prepare and Submit Draft Field Report	10 days	Thu 7/8/21	Wed 7/21/21
103	CENAE Review of Draft Field Report	10 days	Thu 7/22/21	Wed 8/4/21
104	RTC	5 days	Thu 8/5/21	Wed 8/11/21
105	Prepare and Submit Final Field Report	5 days	Thu 8/12/21	Wed 8/18/21
106	**Task 5. RI Project Team Meeting**	**16 days**	**Wed 8/11/21**	**Wed 9/1/21**
107	RI Project Team Meeting	1 day	Wed 8/11/21	Wed 8/11/21
108	Draft Minutes Submitted to CENAE	5 days	Thu 8/12/21	Wed 8/18/21
109	CENAE Review of Draft Minutes	4 days	Thu 8/19/21	Tue 8/24/21
110	RTC and Prepare Final Minutes	6 days	Wed 8/25/21	Wed 9/1/21
111	Submit Final Minutes	0 days	Wed 9/1/21	Wed 9/1/21
112	**Task 6. RI Report**	**200 days**	**Thu 9/2/21**	**Fri 6/17/22**
113	Prepare Client Draft RI Report	80 days	Thu 9/2/21	Wed 12/29/21
114	CENAE Review of Client Draft RI Report	22 days	Thu 12/30/21	Mon 1/31/22
115	RTC and Prepare CX Draft RI Report	18 days	Tue 2/1/22	Thu 2/24/22
116	CX Review of Draft RI Report	20 days	Fri 2/25/22	Thu 3/24/22
117	RTC and Prepare Stakeholder Draft RI Report	20 days	Fri 3/25/22	Thu 4/21/22
118	Stakeholder Review of Draft RI Report	21 days	Fri 4/22/22	Fri 5/20/22
119	RTC and Prepare Final RI Report	19 days	Mon 5/23/22	Fri 6/17/22
120	Submit Final RI Report	0 days	Fri 6/17/22	Fri 6/17/22
121	**Task 7. PIP/Community Relations Support**	**943 days**	**Thu 8/15/19**	**Thu 4/27/23**
122	Prepare Client Draft CRP	12 days	Thu 8/15/19	Fri 8/30/19
123	CENAE Review of Client Draft CRP	10 days	Mon 9/2/19	Fri 9/13/19
124	RTC, Prepare Final CRP	10 days	Mon 9/16/19	Fri 9/27/19
125	Submit Final CRP	0 days	Fri 9/27/19	Fri 9/27/19
126	CR Support	943 days	Thu 8/15/19	Thu 4/27/23
127	**Task 8. CERCLA Feasibility Study**	**141 days**	**Mon 7/11/22**	**Mon 1/30/23**
128	FS Project Team Meeting	1 day	Mon 7/11/22	Mon 7/11/22
129	Prepare Client Draft FS Report	25 days	Tue 7/12/22	Mon 8/15/22
130	CENAE Review of Client Draft FS Report	20 days	Tue 8/16/22	Tue 9/13/22
131	RTC and Prepare CX Draft FS Report	20 days	Wed 9/14/22	Tue 10/11/22
132	CX Review of Draft FS Report	20 days	Wed 10/12/22	Tue 11/8/22
133	RTC and Prepare Stakeholder Draft FS Report	15 days	Wed 11/9/22	Thu 12/1/22
134	Stakeholder Review of Draft FS Report	21 days	Fri 12/2/22	Tue 1/3/23
135	RTC and Prepare Final FS Report	19 days	Wed 1/4/23	Mon 1/30/23
136	Submit Final FS Report	0 days	Mon 1/30/23	Mon 1/30/23
137	**Task 9. Proposed Plan**	**131 days**	**Tue 10/11/22**	**Mon 4/17/23**
138	PP Project Team Meeting	1 day	Tue 10/11/22	Tue 10/11/22
139	Prepare Client Draft Proposed Plan	10 days	Wed 10/12/22	Tue 10/25/22
140	CENAE Review of Client Draft Proposed Plan	20 days	Wed 10/26/22	Tue 11/22/22
141	RTC and Prepare CX Draft Proposed Plan	15 days	Wed 11/23/22	Thu 12/15/22
142	CX Review of Draft Proposed Plan	20 days	Fri 12/16/22	Mon 1/16/23
143	RTC and Prepare Stakeholder Draft PP	15 days	Tue 1/17/23	Mon 2/6/23
144	Stakeholder Review of Draft PP	10 days	Tue 2/7/23	Mon 2/20/23
145	RTC and Prepare Final Proposed Plan	10 days	Tue 2/21/23	Mon 3/6/23
146	Submit Final Proposed Plan	0 days	Mon 3/6/23	Mon 3/6/23
147	Public Review and Public Meeting	30 days	Tue 3/7/23	Mon 4/17/23
148	**Task 10. Decision Document and Resp Sum**	**140 days**	**Tue 3/7/23**	**Tue 9/19/23**
149	Prepare Client Draft Decision Document & RS	20 days	Tue 3/7/23	Mon 4/3/23
150	CENAE Review of Client Draft DD & RS	20 days	Tue 4/4/23	Mon 5/1/23
151	RTC and Prepare CX Draft DD & RS	20 days	Tue 5/2/23	Tue 5/30/23
152	CX Review of Draft Decision Document & RS	20 days	Wed 5/31/23	Tue 6/27/23
153	RTC and Prepare Stakeholder Draft DD & RS	20 days	Wed 6/28/23	Tue 7/25/23
154	Stakeholder Review of Draft DD & RS	20 days	Wed 7/26/23	Tue 8/22/23
155	RTC and Prepare Final DD & RS	20 days	Wed 8/23/23	Tue 9/19/23
156	Submit Final Decision Document & RS	0 days	Tue 9/19/23	Tue 9/19/23
157	**Task 11. Site Walk/Transition Meeting**	1 day	Tue 10/17/23	Tue 10/17/23



All durations are in work-days
Date: Wed 12/9/20

Task | Milestone | Summary | Project Summary | Progress

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits - Text
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

15.1 PROJECT ACTION LIMITS

Project Action Limits (PAL) on **Worksheets #15** are the lower of human health and ecological benchmarks identified based on the hierarchies presented below. Groundwater PALs are based on human health values only and soil PALs are based on the lower of human health and ecological values.

Human Health

- Groundwater (GW):
    - EPA RSL (November 2020): Tapwater Tables for Cancer Target Risk of 1E-06 and Target Hazard Quotient of 0.1 (https://semspub.epa.gov/work/HQ/200045.pdf); then
    - New York State Department of Environmental Conservation (NYSDEC; 6 NYCRR Part 703-5, 2019): Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations – Groundwater standards (https://govt.westlaw.com/nycrr/Document/I4ed90418cd1711dda432a117e6e0f345?viewType=FullText&originationContext=documenttoc&transitionType=CategoryPageItem&contextData=(sc.Default)&bhcp=1).

- Soil (SO):
    - EPA RSL (November 2020) for Residential Soil Tables for Cancer Target Risk of 1E-06 and Target Hazard Quotient of 0.1 (https://semspub.epa.gov/work/HQ/200045.pdf)

Ecological

Supporting documentation for the ecological PALs is presented in **Appendix A**. **Appendix A.1** presents the methodology for selecting surrogates for key constituents without ecological benchmarks and **Appendix A.2** presents all of the values within the QAPP ecological PAL selection hierarchy.

The purpose of identifying ecological PALs in the QAPP is to ensure the analytical methods selected are sensitive enough to detect concentrations at or below the screening benchmark or absent that, the most sensitive method available.

Soil screening benchmarks are derived for multiple receptor categories and all expected site-specific terrestrial receptor categories need to be considered when selecting the ecological soil PALs.

For this Site, the expected site-specific terrestrial receptor categories are plants, soil invertebrates, birds, and mammals. Although the ecological PALs presented in **Table A-2** are not presented for each of these expected terrestrial site-specific receptor categories, each was considered because the screening benchmark presented in **Table A-2** for each reference in the hierarchy is based on the lowest screening benchmark available from plants, soil invertebrates, birds, and mammals.

As noted previously, the analyte concentrations obtained from the Phase 1 sampling will be used to determine if the Site evaluation will proceed to Phase 2. As part of the determination, analyte concentrations will be compared with ecological PALs. During this Phase 1 screening, ecological PALs for

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, Continued

soil will be presented for each of the site-specific receptor categories identified during the site-specific resource receptor inventory (see **Worksheet 17, Section 17.2.3**). Comparing site-specific receptor category-based PALs with analytical results will provide multiple lines of evidence to determine whether or not there is an ecological basis to proceed to Phase 2 and ensure that the site-specific terrestrial ecological receptor categories are considered when determining whether or not to proceed to Phase 2. If it is determined that the investigation will proceed to Phase 2, screening on a site-specific terrestrial receptor category basis will ensure that ecological PAL DQOs for soil are tailored to the receptors to be evaluated in the Phase 2 ecological evaluation.

- Soil (SO) (if reference has more than one receptor, the lowest value was selected):
    - EPA Eco-SSLs (Eco-SSLs; various dates) (https://www.epa.gov/chemical-research/interim-ecological-soil-screening-level-documents)
    - EPA Region 4 Ecological Risk Assessment Supplemental Guidance (2018) –Soil Screening Values for Hazardous Waste Sites (https://www.epa.gov/sites/production/files/2018-03/documents/era_regional_supplemental_guidance_report-march-2018_update.pdf).
    - EPA Region 5 (2003) RCRA Ecological Screening Levels (ESLs) (https://archive.epa.gov/region5/waste/cars/web/pdf/ecological-screening-levels-200308.pdf)
    - Los Alamos National Laboratory (LANL) ECORISK Database Ecological Screening Levels (2017) (https://www.lanl.gov/environment/protection/eco-risk-assessment.php)

Note that PALs for m,p-xylene and m,p-cresol are the lower of the two isomers.

Appendix A.3 presents the selected soil human health and ecological PALs side-by-side along with the value selected as the final PAL that is shown on **Worksheet #15** tables.

15.1 LABORATORY LIMITS

Laboratory Limits (Limit of Quantitation (LOQ), Limit of Detection (LOD), and Detection Limit (DL)) are current as of when the information was requested for the UFP-QAPP and should be considered representative. The following laboratories are not DoD accredited for the noted analyses; therefore three-tiered reporting (i.e., LOQ, LOD, and Detection Limit) is not available:

- TA Corpus Christi – Methods 9081/6010B for CEC.

TA Savannah has recently been certified for DNCB. Three-tiered reporting limits have not yet been provided.

15.2 PROJECT QUANTITATION LIMIT GOALS

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, (factors as per the Intergovernmental Data Quality Task Force (IDQTF)) UFP-QAPP Manual Section 2.6.2.3; EPA, DoD and Department of Energy (DoE), 2005) unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG.

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, Continued

For human health-based PALs, setting the PQLG to ½ the PAL introduces a safety factor, which equates the PQLG to a cancer target risk of 5E-07 or a target hazard quotient of 0.05.

15.3 ANALYTE LISTS

Routine analytes for which there is no PAL and which are unlikely to be site-related were considered for elimination from the analysis unless there was a PAL for that analyte in another matrix. Based on these criteria, no analytes were eliminated. Note that VOCs without PALs were retained at AOC 3 and AOC 5 because they are frequently associated with landfills and therefore will allow for a more comprehensive assessment of potential risk. The retained analytes without PALs had the PQLG set to five times the LOQ.

15.4 ANALYTES WITH LOQS GREATER THAN PALS

Some analytes have limits (LOQs and/or LODs) greater than PALs. This indicates that achievement of the PAL is technically infeasible by the laboratory with the selected method. Prior to the finalization of **Worksheet #15**, methods were changed for known constituents of potential concern when possible to meet PALs; however, many analytes do not have alternatives to achieve lower limits.

Non-detects will be reported at the LOD; therefore, if the LOD meets the PAL, there is no effect on the risk assessment process. If the LOD does not meet the appropriate PAL for the specific human health or ecological risk assessment, how non-detects are handled will depend on the characteristics of the data set. When the PAL is less than the LOQ, detections less than the LOQ (J qualified results) cannot be used to determine whether contamination is above or below the PAL, unless these results are part of a data set that contains at least one result greater or equal to the LOQ.

- If detected concentrations of an analyte exceed the PAL, the analyte will be carried through the Phase 1 risk screening using the LOD as the concentration for non-detects when calculating summary statistics and as the input in ProUCL (if applicable).
- In the cases when no detects are greater or equal to the LOQ in a data set (i.e., all detects are J-qualified), for the purposes of the Phase 1 risk screening, the concentration of the analyte will be set equal to the LOQ because the LOQ is a more defensible concentration than the J-qualified result and this will be discussed in the Uncertainty Analysis.
- If there are no detected concentrations, the Uncertainty Analysis will include a discussion of potential risks. The Uncertainty Analysis will first evaluate the likelihood of non-detected analytes being DoD-related chemicals based on site history and operation, and eliminate analytes that are unlikely to be DoD-related from the risk assessment. The Uncertainty Analysis will then assume the maximum LOD for a DoD-related chemical as the exposure point concentration for the exposure area and the results will be qualitatively discussed in the Uncertainty Analysis.

The results of the Phase 1 risk screening, among other evaluations (e.g., frequency of detection, comparisons with background), will be used to determine the need for and scope of a Phase 2 sampling program.

For analytes with LOQs and/or LODs greater than PALs, the impacts on data usability vary as noted below. Note that the majority of these analytes are VOCs/SVOCs that are being analyzed for in AOC 3 (Landfill Area) and AOC 5 (Former Power Plant) and are not directly related to the manufacture of ammonium picrate.

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, Continued

This evaluation does not account for sample-specific limits that may be elevated due to matrix effects, QC issues, or other analytical issues; which, if present, would need to be considered during the risk assessment process.

15.4.1 Groundwater

Considers human health screening levels only. See method- and medium-specific #15 tables below the text.

Residential Tapwater PAL is less than the LOQ and/or LOD, MCL equal to or greater than the LOQ – Although not meeting one type of risk-based screening level, these analytes would be adequately regulated based on meeting the promulgated Federal standard. See text above regarding how non-detects will be treated in the risk assessments. Analytes to which this applies are:

- Antimony
- Arsenic
- Cadmium
- Mercury
- Selenium
- Thallium
- 1,2,4-Trichlorobenzene
- 1,1,2-Trichloroethane
- 1,2-Dichloroethane
- 1,2-Dichloropropane
- 1,4-Dichlorobenzene
- Benzene
- Bromodichloromethane, chloroform, dibromochloromethane (individual LOQs of 1 µg/L versus MCL for total trihalomethanes of 80 µg/L)
- Carbon tetrachloride
- Vinyl chloride
- Benzo(a)pyrene
- Hexachlorocyclopentadiene

Residential Tapwater PAL is less than the LOD; cancer-based RSL is within a factor of 10 of the LOQ and/or LOD – If concentrations in the dataset are all non-detected and/or comprised of J values, decisions whether the analyte is detected still can be made within the 1E-05 excess lifetime cancer risk (ELCR) range, which is within EPA's acceptable risk range. As per CERCLA guidelines, these data would be deemed usable in the risk assessments; however, quantitative risk estimates would not be reliable.

- Benzo(a)anthracene
- Dibenz(a,h)anthracene
- 2,4,6-Trichlorophenol
- 4-Nitroaniline
- Atrazine
- Hexachloroethane

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, Continued

- Pentachlorophenol

Residential Tapwater PAL is less than the LOD; cancer-based RSL is within a factor of 100 of the LOQ and/or LOD – If concentrations in the dataset are all non-detected and/or comprised of J values, decisions whether the analyte is detected still can be made within the 1E-04 excess lifetime cancer risk (ELCR) range, which is within EPA's acceptable risk range. As per CERCLA guidelines, these data would be deemed usable in the risk assessments; however, quantitative risk estimates would not be reliable.

- 1,1,2,2-Tetrachloroethane
- 1,2-Dibromoethane
- 1,4-Dioxane
- 2,4-Dinitrotoluene
- 2,6-Dinitrotoluene
- 4-Chloroaniline
- Nitrobenzene

Residential Tapwater PAL falls between the LOQ and the LOD – These chemicals may be positively identified as detected, but the quantification would be estimated. As per CERCLA guidelines, these data would be deemed usable in the risk assessments. Chemicals to which this applies are:

- Nickel
- 2-Chlorophenol
- 2-Nitroaniline
- 2,4-Dichlorophenol
- Bis(2-chloroethoxy)methane
- Bis(2-ethylhexyl) phthalate

Residential Tapwater PAL is less than or equal to the LOD – Reliable risk-based decisions cannot be made unless the concentrations are greater than the LOQ. See text above regarding how non-detects will be treated in the risk assessments.

- Cobalt
- Silver
- Vanadium
- 1,2-Dibromo-3-chloropropane
- 1,2,4,5-Tetrachlorobenzene
- 2,4-DNP (constituent of potential concern)
- 3,3'-Dichlorobenzidine
- 4,6-Dinitro-2-methylphenol
- Biphenyl
- Bis(2-chloroethyl) ether
- Dibenzofuran
- Hexachlorobenzene
- Hexachlorobutadiene

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, Continued

- N-Nitrosodi-n-propylamine

3-Nitroaniline NYSDEC Groundwater Standards PAL is equal to the DL. Reliable risk-based decisions cannot be made unless the concentrations are greater than the LOQ. See text above regarding how non-detects will be treated in the risk assessments.

15.4.2 Soils

Considers the lower of human health and ecological screening levels. See method- and medium-specific #15 tables below the text.

Soil PAL (i.e., the lower of the human health and ecological screening value) falls between the LOQ and the LOD – These chemicals may be positively identified as detected, but the quantification would be estimated. As per CERCLA guidelines, these data would be deemed usable in the risk assessments. Chemicals to which this applies are:

- Thallium
- cis-1,3-Dichloropropene
- trans-1,3-Dichloropropene
- 1,2,4,5-Tetrachlorobenzene
- 2-Methylphenol
- Bis(2-chloroethoxy)methane
- Bis(2-chloroethyl) ether
- Carbazole
- Cresols, m- & p-
- Dibenzofuran
- Diethyl phthalate
- Hexachlorobenzene
- N-Nitrosodi-n-propylamine
- Pentachlorophenol

Human health-based PAL selected and the residential soil RSL is less than the LOD; cancer-based RSL is within a factor of 10 of the LOQ and the LOD – If concentrations in the dataset are all non-detected and/or comprised of J values, decisions whether the analyte is detected still can be made within the 1E-05 excess lifetime cancer risk (ELCR) range, which is within EPA's acceptable risk range. As per CERCLA guidelines, these data would be deemed usable in the risk assessments; however, quantitative risk estimates would not be reliable.

- 1,2-Dibromo-3-chloropropane
- 3,3'-Dichlorobenzidine

Soil PAL (i.e., the lower of the human health and ecological screening value) is less than the LOD – Reliable risk-based decisions cannot be made unless the concentrations are greater than the LOQ. See text above regarding how non-detects will be treated in the risk assessments.

- Mercury

QAPP Worksheet #15a: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, Continued

- Bromomethane
- 2,3,4,6-Tetrachlorophenol
- 2-Chlorophenol
- 2,4-Dichlorophenol
- 2,4-Dimethylphenol
- 2,4-DNP (constituent of potential concern)
- 2-Chloronaphthalene
- 2-Nitroaniline
- 4,6-Dinitro-2-methylphenol
- Atrazine
- Biphenyl
- Bis(2-ethylhexyl)phthalate
- Di-n-butyl phthalate
- Hexachlorobutadiene
- Hexachlorocyclopentadiene
- Hexachloroethane
- Picric acid (constituent of potential concern)

QAPP Worksheet #15b: PALs and Laboratory-Specific DL/QL – Tables

Project Quantitation Limit Goals, LOQs, LODs, and DLs are highlighted on the **Worksheet #15** tables using the following rules.

- PQLG: Highlighted yellow if zero or greater than 1/3 of associated PAL or Screening Level. Note that all PQLGs are highlighted yellow because they were set to ½ of the associated PAL.
- LOQ: Highlighted yellow if zero or greater than ½ of associated Project Quantitation Limit Goal.
- LOD: Highlighted yellow if zero or greater than ½ of associated LOQ.
- DL: Highlighted yellow if zero or greater than associated LOD.

Contents List

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **GC/MS-SIM Analysis by SW8270D (Method 8270D SIM PAHs GW and SO)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1-Methylnaphthalene	1.1	2020 Nov EPA Tapwater (THQ 0.1)	0.55	0.1	0.04	0.0183
2-Methylnaphthalene	3.6	2020 Nov EPA Tapwater (THQ 0.1)	1.8	0.1	0.05	0.0214
Acenaphthene	53	2020 Nov EPA Tapwater (THQ 0.1)	26.5	0.1	0.04	0.0042
Acenaphthylene			0.5	0.1	0.04	0.0051
Anthracene	180	2020 Nov EPA Tapwater (THQ 0.1)	90	0.1	0.1	0.0307
Benzo(a)anthracene	0.03	2020 Nov EPA Tapwater (THQ 0.1)	0.1	0.1	0.1	0.0283
Benzo(a)pyrene	0.025	2020 Nov EPA Tapwater (THQ 0.1)	0.1	0.1	0.05	0.0248
Benzo(b)fluoranthene	0.25	2020 Nov EPA Tapwater (THQ 0.1)	0.125	0.1	0.1	0.0396
Benzo(g,h,i)perylene			0.5	0.1	0.1	0.0372
Benzo(k)fluoranthene	2.5	2020 Nov EPA Tapwater (THQ 0.1)	1.25	0.1	0.05	0.0229
Chrysene	25	2020 Nov EPA Tapwater (THQ 0.1)	12.5	0.1	0.1	0.0331
Dibenz(a,h)anthracene	0.025	2020 Nov EPA Tapwater (THQ 0.1)	0.1	0.1	0.1	0.0277
Fluoranthene	80	2020 Nov EPA Tapwater (THQ 0.1)	40	0.1	0.1	0.0486
Fluorene	29	2020 Nov EPA Tapwater (THQ 0.1)	14.5	0.1	0.04	0.0192
Indeno(1,2,3-c,d)pyrene	0.25	2020 Nov EPA Tapwater (THQ 0.1)	0.125	0.1	0.1	0.0392
Naphthalene	0.12	2020 Nov EPA Tapwater (THQ 0.1)	0.1	0.1	0.05	0.023
Phenanthrene			0.5	0.1	0.1	0.0494
Pyrene	12	2020 Nov EPA Tapwater (THQ 0.1)	6	0.1	0.1	0.0451

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C ICP Metals GW and SO)**

Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	2000	2020 Nov EPA Tapwater (THQ 0.1)	1000	300	70	18
Barium	380	2020 Nov EPA Tapwater (THQ 0.1)	190	10	2	0.82
Beryllium	2.5	2020 Nov EPA Tapwater (THQ 0.1)	1.5	1.5	0.5	0.165
Cadmium	0.92	2020 Nov EPA Tapwater (THQ 0.1)	5	5	1.8	0.452
Calcium			5000	1000	160	77.8
Copper	80	2020 Nov EPA Tapwater (THQ 0.1)	40	15	10	4.2
Iron	1400	2020 Nov EPA Tapwater (THQ 0.1)	700	100	85	22
Magnesium			2500	500	60	26.4
Manganese	43	2020 Nov EPA Tapwater (THQ 0.1)	21.5	10	4	1.88
Nickel	39	2020 Nov EPA Tapwater (THQ 0.1)	40	40	8	2.56
Potassium			15000	3000	940	237
Selenium	10	2020 Nov EPA Tapwater (THQ 0.1)	22	22	19	6.3
Silver	9.4	2020 Nov EPA Tapwater (THQ 0.1)	15	15	5	1.96
Sodium	20000	New York State Groundwater 703.5	10000	5000	1000	373
Vanadium	8.6	New York State Groundwater 703.5	15	15	4	1.11
Zinc	600	2020 Nov EPA Tapwater (THQ 0.1)	300	150	15	4.53

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A ICPMS Metals GW and SO)**

Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	0.78	2020 Nov EPA Tapwater (THQ 0.1)	6	6	1	0.4
Arsenic	0.052	2020 Nov EPA Tapwater (THQ 0.1)	5	5	1	0.33
Chromium	2200	2020 Nov EPA Tapwater (THQ 0.1) - Chromium	1100	10	1.8	0.5
Cobalt	0.6	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.35	0.0923
Lead	15	2020 Nov EPA Tapwater (THQ 0.1)	7.5	3	0.7	0.18
Thallium	0.02	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.2	0.089

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Mercury in Water (Manual Cold-Vapor Technique) (SW7470A Mercury GW)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.063	2020 Nov EPA Tapwater (THQ 0.1)	0.2	0.2	0.08	0.027

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Polychlorinated Biphenyls (PCB) (SW8082A PCBs SO and GW)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
PCB-1016 (Aroclor 1016)	0.14	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.124
PCB-1221 (Aroclor 1221)	0.0047	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.25	0.214
PCB-1232 (Aroclor 1232)	0.0047	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.6	0.166
PCB-1242 (Aroclor 1242)	0.0078	2020 Nov EPA Tapwater (THQ 0.1)	1	1	1	0.419
PCB-1248 (Aroclor 1248)	0.0078	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.3	0.0915
PCB-1254 (Aroclor 1254)	0.0078	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.25	0.114
PCB-1260 (Aroclor 1260)	0.0078	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.16
PCB-1262 (Aroclor 1262)			5	1	0.5	0.222
PCB-1268 (Aroclor 1268)			5	1	1	0.363

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C VOCs GW and SO)**
Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	800	2020 Nov EPA Tapwater (THQ 0.1)	400	1	0.4	0.16
1,1,2,2-Tetrachloroethane	0.076	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.8	0.21
1,1,2-Trichloro-1,2,2-trifluoroethane	1000	2020 Nov EPA Tapwater (THQ 0.1)	500	3	0.4	0.181
1,1,2-Trichloroethane	0.041	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.8	0.27
1,1-Dichloroethane	2.8	2020 Nov EPA Tapwater (THQ 0.1)	1.4	1	0.8	0.22
1,1-Dichloroethene	28	2020 Nov EPA Tapwater (THQ 0.1)	14	1	0.8	0.23
1,2,3-Trichlorobenzene	0.7	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.8	0.21
1,2,4-Trichlorobenzene	0.4	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.8	0.21
1,2-Dibromo-3-chloropropane	0.00033	2020 Nov EPA Tapwater (THQ 0.1)	5	5	1.6	0.47
1,2-Dibromoethane (EDB)	0.0075	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.18
1,2-Dichlorobenzene	30	2020 Nov EPA Tapwater (THQ 0.1)	15	1	0.4	0.15
1,2-Dichloroethane	0.17	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.13
1,2-Dichloropropane	0.82	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.18
1,3-Dichlorobenzene	3	New York State Groundwater 703.5	1.5	1	0.4	0.13
1,3-Dichloropropane	37	New York State Groundwater 703.5	18.5	1	0.2	0.0903
1,4-Dichlorobenzene	0.48	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.16
2-Butanone (MEK)	560	2020 Nov EPA Tapwater (THQ 0.1)	280	6	4	2
2-Hexanone	3.8	2020 Nov EPA Tapwater (THQ 0.1)	5	5	4	1.7
4-Methyl-2-pentanone (MIBK)	630	2020 Nov EPA Tapwater (THQ 0.1)	315	5	3.2	0.98
Acetone	1400	2020 Nov EPA Tapwater (THQ 0.1)	700	10	6.4	1.9
Benzene	0.46	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.16
Bromochloromethane	8.3	2020 Nov EPA Tapwater (THQ 0.1)	4.15	1	0.2	0.1
Bromodichloromethane	0.13	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.17
Bromoform	3.3	2020 Nov EPA Tapwater (THQ 0.1)	1.65	1	1	0.458
Bromomethane	0.75	2020 Nov EPA Tapwater (THQ 0.1)	2	2	0.8	0.21
Carbon disulfide	81	2020 Nov EPA Tapwater (THQ 0.1)	40.5	2	0.8	0.167

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C VOCs GW and SO)**
Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Carbon tetrachloride	0.46	2020 Nov EPA Tapwater (THQ 0.1)	2	2	0.4	0.19
Chlorobenzene	7.8	2020 Nov EPA Tapwater (THQ 0.1)	3.9	1	0.4	0.17
Chloroethane	2100	2020 Nov EPA Tapwater (THQ 0.1)	1050	2	1.6	0.41
Chloroform	0.22	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.16
Chloromethane	19	2020 Nov EPA Tapwater (THQ 0.1)	9.5	2	0.8	0.3
cis-1,2-Dichloroethene	3.6	2020 Nov EPA Tapwater (THQ 0.1)	1.8	1	0.4	0.15
cis-1,3-Dichloropropene			5	1	0.4	0.16
Cyclohexane	1300	2020 Nov EPA Tapwater (THQ 0.1)	650	2	0.8	0.28
Dibromochloromethane	0.87	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.17
Dichlorodifluoromethane	20	2020 Nov EPA Tapwater (THQ 0.1)	10	2	0.8	0.31
Ethylbenzene	1.5	2020 Nov EPA Tapwater (THQ 0.1)	1	1	0.4	0.16
Isopropylbenzene (Cumene)	45	2020 Nov EPA Tapwater (THQ 0.1)	22.5	1	0.4	0.19
m,p-Xylene	19	2020 Nov m&p Cresol and m&p Xylene	9.5	2	0.8	0.153
Methyl acetate	2000	2020 Nov EPA Tapwater (THQ 0.1)	1000	5	4	1.64
Methyl tert-butyl ether (MTBE)	14	2020 Nov EPA Tapwater (THQ 0.1)	7	5	0.8	0.25
Methylcyclohexane			10	2	0.4	0.102
Methylene chloride	11	2020 Nov EPA Tapwater (THQ 0.1)	5.5	5	2	0.938
o-Xylene	19	2020 Nov EPA Tapwater (THQ 0.1)	9.5	1	0.4	0.19
Styrene	120	2020 Nov EPA Tapwater (THQ 0.1)	60	1	0.8	0.356
Tetrachloroethene (PCE)	4.1	2020 Nov EPA Tapwater (THQ 0.1)	2.05	1	0.4	0.2
Toluene	110	2020 Nov EPA Tapwater (THQ 0.1)	55	1	0.4	0.17
trans-1,2-Dichloroethene	6.8	2020 Nov EPA Tapwater (THQ 0.1)	3.4	1	0.4	0.15
trans-1,3-Dichloropropene			5	1	0.4	0.19
Trichlorofluoromethane	520	2020 Nov EPA Tapwater (THQ 0.1)	260	2	0.8	0.29
Vinyl chloride	0.019	2020 Nov EPA Tapwater (THQ 0.1)	1.5	1.5	0.2	0.1

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C VOCs GW and SO)**
Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (SW8270D SVOCs TCL DNCB and DNP GW and SO)**

Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,2,4,5-Tetrachlorobenzene	0.17	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.76
1,4-Dioxane (p-Dioxane)	0.46	2020 Nov EPA Tapwater (THQ 0.1)	10	10	10	3.4
2,2'-Oxybis(1-chloropropane)	71	2020 Nov EPA Tapwater (THQ 0.1)	35.5	10	2	0.78
2,3,4,6-Tetrachlorophenol	24	2020 Nov EPA Tapwater (THQ 0.1)	12	10	2	0.72
2,4,5-Trichlorophenol	120	2020 Nov EPA Tapwater (THQ 0.1)	60	10	2	1.2
2,4,6-Trichlorophenol	1.2	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.85
2,4-Dichlorophenol	4.6	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.1
2,4-Dimethylphenol	36	2020 Nov EPA Tapwater (THQ 0.1)	18	10	10	4
2,4-Dinitrochlorobenzene			100	20	10	4.5
2,4-Dinitrophenol	3.9	2020 Nov EPA Tapwater (THQ 0.1)	50	50	20	10
2,4-Dinitrotoluene	0.24	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.2
2,6-Dinitrotoluene	0.049	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.1
2-Chloronaphthalene	75	2020 Nov EPA Tapwater (THQ 0.1)	37.5	10	2	0.8
2-Chlorophenol	9.1	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.87
2-Methylphenol (o-Cresol)	93	2020 Nov EPA Tapwater (THQ 0.1)	46.5	10	2	0.89
2-Nitroaniline	19	2020 Nov EPA Tapwater (THQ 0.1)	50	50	2	1.3
2-Nitrophenol			50	10	2	0.76
3,3'-Dichlorobenzidine	0.13	2020 Nov EPA Tapwater (THQ 0.1)	60	60	60	30
3-Nitroaniline	5	New York State Groundwater 703.5	50	50	10	5
4,6-Dinitro-2-methylphenol	0.15	2020 Nov EPA Tapwater (THQ 0.1)	50	50	20	10
4-Bromophenyl phenyl ether			50	10	2	0.77
4-Chloro-3-methylphenol	140	2020 Nov EPA Tapwater (THQ 0.1)	70	10	2	1
4-Chloroaniline	0.37	2020 Nov EPA Tapwater (THQ 0.1)	20	20	5	2.2
4-Chlorophenyl phenyl ether			50	10	2	0.84

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (SW8270D SVOCs TCL DNCB and DNP GW and SO)**

Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
4-Nitroaniline	3.8	2020 Nov EPA Tapwater (THQ 0.1)	50	50	10	5
4-Nitrophenol			250	50	4	1.9
Acetophenone	190	2020 Nov EPA Tapwater (THQ 0.1)	95	10	2	0.57
Atrazine	0.3	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.2
Benzaldehyde	19	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.1
Benzyl butyl phthalate	16	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.2
Biphenyl (Diphenyl)	0.083	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.58
Bis(2-chloroethoxy) methane	5.9	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.94
Bis(2-chloroethyl) ether (2-Chloroethyl ether)	0.014	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	1.1
Bis(2-ethylhexyl) phthalate	5.6	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2.5	1.6
Caprolactam	990	2020 Nov EPA Tapwater (THQ 0.1)	495	10	2	0.79
Carbazole			50	10	2	0.71
Cresols, m- & p-	93	2020 Nov m&p Cresol and m&p Xylene	46.5	10	2	1.3
Dibenzofuran	0.79	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.79
Diethyl phthalate	1500	2020 Nov EPA Tapwater (THQ 0.1)	750	10	2	0.88
Dimethyl phthalate			50	10	2	0.99
Di-n-butyl phthalate	90	2020 Nov EPA Tapwater (THQ 0.1)	45	10	2	0.83
di-n-Octyl phthalate	20	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2.5	1.4
Hexachlorobenzene	0.0098	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.79
Hexachlorobutadiene	0.14	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.62
Hexachlorocyclopentadiene	0.041	2020 Nov EPA Tapwater (THQ 0.1)	10	10	5	2.5
Hexachloroethane	0.33	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.76
Isophorone	78	2020 Nov EPA Tapwater (THQ 0.1)	39	10	2	0.9

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (SW8270D SVOCs TCL DNCB and DNP GW and SO)**

Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Nitrobenzene	0.14	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.73
N-Nitrosodi-n-propylamine	0.011	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.72
N-Nitrosodiphenylamine	12	2020 Nov EPA Tapwater (THQ 0.1)	10	10	2	0.92
Pentachlorophenol	0.041	2020 Nov EPA Tapwater (THQ 0.1)	50	50	4	2
Phenol	580	2020 Nov EPA Tapwater (THQ 0.1)	290	10	2	0.83

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Aqueous**
Analytical Group: **Nitroaromatics and Nitramines by HPLC (SW8330B Explosives GW)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Picric acid	4	2020 Nov EPA Tapwater (THQ 0.1)	2	0.4	0.12	0.0436

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **GC/MS-SIM Analysis by SW8270D (Method 8270D SIM PAHs GW and SO)**
Concentration Level (if applicable): Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1-Methylnaphthalene	18000	2020 Nov EPA Resident Soil (THQ 0.1)	9000	10	2	0.52
2-Methylnaphthalene	24000	2020 Nov EPA Resident Soil (THQ 0.1)	12000	10	2	0.618
Acenaphthene	360000	2020 Nov EPA Resident Soil (THQ 0.1)	180000	10	1.07	0.32
Acenaphthylene			50	10	1.07	0.34
Anthracene	1800000	2020 Nov EPA Resident Soil (THQ 0.1)	900000	10	4.33	1.44
Benzo(a)anthracene	1100	2020 Nov EPA Resident Soil (THQ 0.1)	550	10	4.33	1.8
Benzo(a)pyrene	110	2020 Nov EPA Resident Soil (THQ 0.1)	55	10	4.33	1.48
Benzo(b)fluoranthene	1100	2020 Nov EPA Resident Soil (THQ 0.1)	550	10	6.67	2.4
Benzo(g,h,i)perylene			50	10	6.67	2.2
Benzo(k)fluoranthene	11000	2020 Nov EPA Resident Soil (THQ 0.1)	5500	10	4.33	2
Chrysene	110000	2020 Nov EPA Resident Soil (THQ 0.1)	55000	10	4.33	2
Dibenz(a,h)anthracene	110	2020 Nov EPA Resident Soil (THQ 0.1)	55	10	6.67	2.6
Fluoranthene	240000	2020 Nov EPA Resident Soil (THQ 0.1)	120000	10	4.33	2
Fluorene	240000	2020 Nov EPA Resident Soil (THQ 0.1)	120000	10	2.67	0.94
Indeno(1,2,3-c,d)pyrene	1100	2020 Nov EPA Resident Soil (THQ 0.1)	550	10	6.67	2.2
Naphthalene	2000	2020 Nov EPA Resident Soil (THQ 0.1)	1000	10	2	0.652
Phenanthrene			50	10	6.67	2.2
Pyrene	180000	2020 Nov EPA Resident Soil (THQ 0.1)	90000	10	6.67	2.2

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

Note: For ecological assessment, PAHs will be totaled, which yields a PAL of 29,000 µg/kg for low molecular weight PAHs and 1,100 µg/kg for high molecular weight PAHs. See benchmarks in Appendix A.2 and A.3.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Percent Solids (D2216 Percent Moisture SO and SD)**
Concentration Level (if applicable):
Units: **Percent**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Solids			0.5	0.1	0.05	0

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C ICP Metals GW and SO)**

Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	7700	2020 Nov EPA Resident Soil (THQ 0.1)	3850	50	6	1.55
Barium	330	EPA Ecological Soil Screening Levels	165	2	0.9	0.296
Beryllium	16	2020 Nov EPA Resident Soil (THQ 0.1)	8	0.5	0.12	0.033
Calcium			500	100	50	14.1
Chromium	26	EPA Ecological Soil Screening Levels	13	3.5	0.4	0.123
Cobalt	2.3	2020 Nov EPA Resident Soil (THQ 0.1)	1.15	1	0.2	0.0679
Copper	28	EPA Ecological Soil Screening Levels	14	5	0.8	0.217
Iron	5500	2020 Nov EPA Resident Soil (THQ 0.1)	2750	80	20	8.27
Lead	11	EPA Ecological Soil Screening Levels	5.5	0.9	0.8	0.31
Magnesium			150	30	20	7.92
Manganese	180	2020 Nov EPA Resident Soil (THQ 0.1)	90	4.5	0.4	0.1
Nickel	38	EPA Ecological Soil Screening Levels	19	4	0.45	0.132
Potassium			1500	300	160	41
Silver	4.2	EPA Ecological Soil Screening Levels	2.1	1.5	0.6	0.16
Sodium		EPA Ecological Soil Screening Levels	2500	500	100	28.8
Vanadium	7.8	EPA Ecological Soil Screening Levels	3.9	2	0.35	0.094
Zinc	46	EPA Ecological Soil Screening Levels	23	8	1.5	0.398

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A ICPMS Metals GW and SO)**

Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	0.27	EPA Ecological Soil Screening Levels	0.2	0.2	0.05	0.014
Arsenic	0.68	2020 Nov EPA Resident Soil (THQ 0.1)	0.6	0.6	0.2	0.0506
Cadmium	0.36	EPA Ecological Soil Screening Levels	0.18	0.1	0.035	0.00938
Selenium	0.52	EPA Ecological Soil Screening Levels	0.5	0.5	0.4	0.133
Thallium	0.05	EPA Region 4 Soil	0.1	0.1	0.01	0.00351

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Mercury in Soil (Manual Cold-Vapor Technique) (SW7471B Mercury SO)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.013	EPA Region 4 Soil	0.017	0.017	0.0133	0.00553

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Polychlorinated Biphenyls (PCB) (SW8082A PCBs SO and GW)**
Concentration Level (if applicable):

Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
PCB-1016 (Aroclor 1016)	410	2020 Nov EPA Resident Soil (THQ 0.1)	205	66	30.7	10.2
PCB-1221 (Aroclor 1221)	200	2020 Nov EPA Resident Soil (THQ 0.1)	100	94	80	31.2
PCB-1232 (Aroclor 1232)	170	2020 Nov EPA Resident Soil (THQ 0.1)	85	66	30.7	10.2
PCB-1242 (Aroclor 1242)	230	2020 Nov EPA Resident Soil (THQ 0.1)	115	66	48	18.2
PCB-1248 (Aroclor 1248)	230	2020 Nov EPA Resident Soil (THQ 0.1)	115	66	15	4.78
PCB-1254 (Aroclor 1254)	120	2020 Nov EPA Resident Soil (THQ 0.1)	66	66	33.3	11
PCB-1260 (Aroclor 1260)	240	2020 Nov EPA Resident Soil (THQ 0.1)	120	66	6.67	2.33
PCB-1262 (Aroclor 1262)			330	66	16	5.47
PCB-1268 (Aroclor 1268)			330	66	6.67	2.72

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

Note: For ecological assessment, PCBs will be totaled, which yields a PAL of 41 µg/kg. See benchmarks in Appendix A.2 and A.3.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C VOCs GW and SO)**
Concentration Level (if applicable):
Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	40	EPA Region 4 Soil	20	5	5	1.98
1,1,2,2-Tetrachloroethane	127	EPA Region 4 Soil	63.5	5	0.8	0.285
1,1,2-Trichloro-1,2,2-trifluoroethane	670000	2020 Nov EPA Resident Soil (THQ 0.1)	335000	20	6.4	1.66
1,1,2-Trichloroethane	150	2020 Nov EPA Resident Soil (THQ 0.1)	75	5	3.2	0.88
1,1-Dichloroethane	140	EPA Region 4 Soil	70	5	0.8	0.21
1,1-Dichloroethene	40	EPA Region 4 Soil	20	5	1.6	0.59
1,2,3-Trichlorobenzene	6300	2020 Nov EPA Resident Soil (THQ 0.1)	3150	5	3.2	0.81
1,2,4-Trichlorobenzene	270	EPA Region 4 Soil	135	5	1.6	0.73
1,2-Dibromo-3-chloropropane	5.3	2020 Nov EPA Resident Soil (THQ 0.1)	10	10	10	3.66
1,2-Dibromoethane (EDB)	36	2020 Nov EPA Resident Soil (THQ 0.1)	18	5	1.6	0.52
1,2-Dichlorobenzene	90	EPA Region 4 Soil	45	5	5	1.87
1,2-Dichloroethane	400	EPA Region 4 Soil	200	5	1.6	0.7
1,2-Dichloropropane	280	EPA Region 4 Soil	140	5	1.6	0.55
1,3-Dichlorobenzene	80	EPA Region 4 Soil	40	5	1.6	0.48
1,3-Dichloropropane	160000	EPA Region 4 Soil	80000	5	0.4	0.173
1,4-Dichlorobenzene	880	EPA Region 4 Soil	440	5	0.8	0.245
2-Butanone (MEK)	1000	EPA Region 4 Soil	500	20	12.8	3.89
2-Hexanone	360	EPA Region 4 Soil	180	20	12.8	4.89
4-Methyl-2-pentanone (MIBK)	443000	EPA Region 5 RCRA SO Ecological Screening Levels	222000	20	12.8	4.36
Acetone	1200	EPA Region 4 Soil	600	72	72	35.6
Benzene	120	EPA Region 4 Soil	60	5	0.4	0.151
Bromochloromethane	15000	2020 Nov EPA Resident Soil (THQ 0.1)	7500	5	5	2.46
Bromodichloromethane	290	2020 Nov EPA Resident Soil (THQ 0.1)	145	5	5	2.13
Bromoform	70	EPA Region 4 Soil	35	5.1	5.1	2.55

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C VOCs GW and SO)**
Concentration Level (if applicable):
Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Bromomethane	2	EPA Region 4 Soil	10	10	3.2	1.35
Carbon disulfide	5	EPA Region 4 Soil	5	5	5	1.66
Carbon tetrachloride	50	EPA Region 4 Soil	25	5	5	2.01
Chlorobenzene	2400	EPA Region 4 Soil	1200	5	5	2.06
Chloroethane	1400000	2020 Nov EPA Resident Soil (THQ 0.1)	700000	10	6.4	1.99
Chloroform	50	EPA Region 4 Soil	25	10	0.8	0.29
Chloromethane	10400	EPA Region 5 RCRA SO Ecological Screening Levels	5200	10	1.6	0.77
cis-1,2-Dichloroethene	40	EPA Region 4 Soil	20	5	0.8	0.201
cis-1,3-Dichloropropene	1	EPA Region 4 Soil	5	5	0.4	0.1
Cyclohexane	650000	2020 Nov EPA Resident Soil (THQ 0.1)	325000	5	5	1.76
Dibromochloromethane	2050	EPA Region 5 RCRA SO Ecological Screening Levels	1030	5	5	2.27
Dichlorodifluoromethane	8700	2020 Nov EPA Resident Soil (THQ 0.1)	4350	10	6.4	2.74
Ethylbenzene	270	EPA Region 4 Soil	135	5	0.8	0.305
Isopropylbenzene (Cumene)	40	EPA Region 4 Soil	20	5	5	2.41
m,p-Xylene	100	EPA Region 4 Soil	50	3.2	3.2	1.04
Methyl acetate	7800000	2020 Nov EPA Resident Soil (THQ 0.1)	3900000	8.5	8	2.75
Methyl tert-butyl ether (MTBE)	47000	2020 Nov EPA Resident Soil (THQ 0.1)	23500	20	6.4	2.11
Methylcyclohexane			25	5	1.6	0.42
Methylene chloride	210	EPA Region 4 Soil	105	5	3.2	1.6
o-Xylene	100	EPA Region 4 Soil	50	5	0.8	0.266
Styrene	1200	EPA Region 4 Soil	600	5	0.8	0.28
Tetrachloroethene (PCE)	60	EPA Region 4 Soil	30	5	5	1.91
Toluene	150	EPA Region 4 Soil	75	5	0.8	0.227

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C VOCs GW and SO)**
Concentration Level (if applicable):

Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
trans-1,2-Dichloroethene	40	EPA Region 4 Soil	20	5	0.8	0.39
trans-1,3-Dichloropropene	1	EPA Region 4 Soil	5	5	0.2	0.083
Trichlorofluoromethane	16400	EPA Region 4 Soil	8200	10	10	3.2
Vinyl chloride	30	EPA Region 4 Soil	15	5	3.2	1.34

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (SW8270D SVOCs TCL DNCB and DNP GW and SO)**

Concentration Level (if applicable): Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,2,4,5-Tetrachlorobenzene	180	EPA Region 4 Soil	330	330	67	31
1,4-Dioxane (p-Dioxane)	2050	EPA Region 5 RCRA SO Ecological Screening Levels	1030	330	330	120
2,2'-Oxybis(1-chloropropane)	19900	EPA Region 5 RCRA SO Ecological Screening Levels	9950	330	67	30
2,3,4,6-Tetrachlorophenol	40	EPA Region 4 Soil	330	330	83	22
2,4,5-Trichlorophenol	4000	EPA Region 4 Soil	2000	330	67	35
2,4,6-Trichlorophenol	6300	2020 Nov EPA Resident Soil (THQ 0.1)	3150	330	67	29
2,4-Dichlorophenol	50	EPA Region 4 Soil	330	330	67	35
2,4-Dimethylphenol	40	EPA Region 4 Soil	330	330	67	44
2,4-Dinitrochlorobenzene	600	Derived - See Appendix A.1 - Soil	660	660	330	150
2,4-Dinitrophenol	61	EPA Region 4 Soil	1700	1700	1300	830
2,4-Dinitrotoluene	1700	2020 Nov EPA Resident Soil (THQ 0.1)	850	330	83	49
2,6-Dinitrotoluene	360	2020 Nov EPA Resident Soil (THQ 0.1)	330	330	67	42
2-Chloronaphthalene	12.2	EPA Region 5 RCRA SO Ecological Screening Levels	330	330	67	35
2-Chlorophenol	60	EPA Region 4 Soil	330	330	67	40
2-Methylphenol (o-Cresol)	100	EPA Region 4 Soil	330	330	67	27
2-Nitroaniline	20	EPA Region 4 Soil	1700	1700	170	45
2-Nitrophenol	1600	EPA Region 5 RCRA SO Ecological Screening Levels	800	330	67	41
3,3'-Dichlorobenzidine	30	EPA Region 4 Soil	660	660	67	28
3-Nitroaniline			8500	1700	83	46

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (SW8270D SVOCs TCL DNCB and DNP GW and SO)**

Concentration Level (if applicable): Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
4,6-Dinitro-2-methylphenol	144	EPA Region 5 RCRA SO Ecological Screening Levels	1700	1700	330	170
4-Bromophenyl phenyl ether			1650	330	67	36
4-Chloro-3-methylphenol	7950	EPA Region 5 RCRA SO Ecological Screening Levels	3980	330	67	35
4-Chloroaniline	1000	EPA Region 4 Soil	660	660	83	52
4-Chlorophenyl phenyl ether			1650	330	67	44
4-Nitroaniline	21900	EPA Region 5 RCRA SO Ecological Screening Levels	11000	1700	83	49
4-Nitrophenol	5120	EPA Region 4 Soil	2560	1700	670	330
Acetophenone	300000	EPA Region 5 RCRA SO Ecological Screening Levels	150000	330	67	28
Atrazine	0.05	EPA Region 4 Soil	330	330	67	23
Benzaldehyde	170000	2020 Nov EPA Resident Soil (THQ 0.1)	85000	330	170	58
Benzyl butyl phthalate	590	EPA Region 4 Soil	330	330	67	26
Biphenyl (Diphenyl)	200	EPA Region 4 Soil	1700	1700	330	110
Bis(2-chloroethoxy) methane	302	EPA Region 5 RCRA SO Ecological Screening Levels	330	330	67	39
Bis(2-chloroethyl) ether (2-Chloroethyl ether)	230	2020 Nov EPA Resident Soil (THQ 0.1)	330	330	83	45
Bis(2-ethylhexyl) phthalate	20	EPA Region 4 Soil	330	330	67	29
Caprolactam	3100000	2020 Nov EPA Resident Soil (THQ 0.1)	1550000	330	170	66
Carbazole	70	EPA Region 4 Soil	330	330	67	30
Cresols, m- & p-	80	EPA Region 4 Soil	330	330	67	43

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (SW8270D SVOCs TCL DNCB and DNP GW and SO)**

Concentration Level (if applicable): Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Dibenzofuran	150	EPA Region 4 Soil	330	330	67	33
Diethyl phthalate	250	EPA Region 4 Soil	330	330	67	37
Dimethyl phthalate	350	EPA Region 4 Soil	330	330	67	34
Di-n-butyl phthalate	11	EPA Region 4 Soil	330	330	67	30
di-n-Octyl phthalate	910	EPA Region 4 Soil	455	330	67	29
Hexachlorobenzene	79	EPA Region 4 Soil	330	330	67	39
Hexachlorobutadiene	9	EPA Region 4 Soil	330	330	67	36
Hexachlorocyclopentadiene	1	EPA Region 4 Soil	330	330	67	41
Hexachloroethane	24	EPA Region 4 Soil	330	330	67	28
Isophorone	139000	EPA Region 5 RCRA SO Ecological Screening Levels	69500	330	67	33
Nitrobenzene	2200	EPA Region 4 Soil	1100	330	67	26
N-Nitrosodi-n-propylamine	78	2020 Nov EPA Resident Soil (THQ 0.1)	330	330	67	32
N-Nitrosodiphenylamine	545	EPA Region 4 Soil	330	330	67	33
Pentachlorophenol	1000	2020 Nov EPA Resident Soil (THQ 0.1)	1700	1700	670	330
Phenol	790	EPA Region 4 Soil	395	330	67	34

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) (SW8330A Explosives**
Concentration Level (if applicable): Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Picric acid	6.1	Derived - See Appendix A.1 - Soil	250	250	165	56.3

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Nitroaromatics and Nitramines by HPLC (SW8330B Explosives SO ISM)**
Concentration Level (if applicable):
Units: **µg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Picric acid	6.1	Derived - See Appendix A.1 - Soil	100	100	100	56.3

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Total Organic Carbon (SW9060A TOC SO Discrete)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Total Organic Carbon			20000	4000	2000	902

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #15: Project Action Limit and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: **Solid**
Analytical Group: **Cation-Exchange Capacity of Soils (Sodium Acetate) (SW9081 CEC SO)**
Concentration Level (if applicable):
Units: **mEq/100g**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Cation-Exchange Capacity			0.25	0.05	0	0

A blank in the Project Action Limit (PAL) column indicates that no PAL is available.

Project Quantitation Limit Goals (PQLG) are set to ½ the PAL, unless ½ the PAL is less than the laboratory-achievable LOQ. If ½ the PAL is less than the laboratory-achievable LOQ, the LOQ is selected as the PQLG. Analytes without PALs had the PQLG set to five times the LOQ.

LOQs, LODs, DLs are derived by the laboratories, are current as of when the information was requested for the UFP-QAPP, and should be considered representative.

PQLG, LOQ, LOD, and DL are highlighted yellow if the value does not meet the PAL.

QAPP Worksheet #17: Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

17.1 INTRODUCTION

This worksheet provides information on the field program for the Former NYOW Site. It builds on **Worksheet #11** and discusses the methods required to execute the field program, including the methodology and rationale for selecting and/or modifying actual sampling techniques, and locations and depths based on field conditions.

Phase 1 is a judgmental sampling design intended to provide sufficient information regarding the nature and extent of sampling efforts that will be completed in Phase 2. The physical boundaries of the Site were described in the CSM presented in **Worksheet #10**. Six months is anticipated for data collection, the timing of which is dependent on multiple factors including weather constraints, property access, laboratory capacity, etc. Contingencies are provided for any field conditions encountered that may impact the sampling design and/or schedule.

The field program worksheet includes:

- Site Reconnaissance (**Subsection 17.2**)
- Mobilization (**Subsection 17.3**)
- Surface Geophysical Surveys – AOC 3 and AOC 5 (**Subsection 17.4**)
- ISM Surface Soil Investigation (**Subsection 17.5**)
- Geoprobe® DPT Subsurface Investigation (**Subsection 17.6**)
- Water Level Measurements (**Subsection 17.7**)
- Sample Management (**Subsection 17.8**)
- Decontamination Procedures (**Subsection 17.9**)
- Management of Investigation-Derived Wastes (**Subsection 17.10**)
- Site Restoration (**Subsection 17.11**)
- References (**Subsection 17.12**)

The field activities listed above will be performed in accordance with Bluestone SOPs (see **Appendix B**).

Note that the DoD is not responsible for the investigation of mobilization of metals owing to the acid tanks that were remediated by NYSDEC. Therefore, no soil or groundwater sampling is planned associated with the three former acid tanks at AOC 1. However, DoD is responsible for the metals used during the picric acid manufacturing process, so the rest of AOC 1 will be assessed for metals outside the footprint of the former acid tanks and immediate groundwater.

17.2 SITE RECONNAISSANCE

Site reconnaissance activities further described below include obtaining access to private properties, wetland delineation and vernal pool identification, resource receptor survey, sampling location identification, overhead obstruction and subsurface utility mark-out, and vegetation removal. Initial reconnaissance will also include further review of aerial photographs and photographic documentation. Photographs of site conditions will be documented prior to, during, and after completion of field activities.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.2.1 Access to Private Property

Access to private property must be obtained in order to execute the field investigation. USACE will be responsible for obtaining rights-of-entry for all properties. Bluestone will provide a list of property owners to be contacted prior to the field activities. The list will include the mailing address and telephone number of the property owners (if available). Field work will commence once USACE establishes an access agreement with the owner(s) of each privately-owned parcel proposed for the field program. Bluestone will contact and coordinate with property owners to schedule sampling activities. During the field program, sampling crews will carry a hard copy of the signed access agreement for each parcel, as needed. Per USACE direction, a minimum of one full week (seven days) advanced verbal notice will be provided to the facility/property owners prior to mobilization.

Due to the intrusive nature of some field activities, it is possible that property owners may object to having a DPT rig on their property or may feel that the field team is trespassing. If USACE cannot secure rights-of-entry, Bluestone will obtain the contact information for several of the closest neighbors (if possible) and provide the information to USACE to establish rights-of-entry to the adjacent properties.

17.2.2 Wetland Delineation and Vernal Pool Identification

A wetlands delineation will be conducted for all four AOCs (1, 3, 4, and 5) to identify jurisdictional areas such as wetlands, streams, and vernal pools that may be impacted by site-related contamination. The wetlands delineation will be conducted in accordance with the USACE Wetlands Delineation Manual (1987) and the Regional Supplement to the USACE Wetland Delineation Manual: North Central and Northeast Region, Version 2.0 (USACE, 2012). Delineation activities will also incorporate requirements of The Freshwater Wetlands Act (6 NYCRR Part 663-665) (NYSDEC, 1997). Desktop research will be conducted first, followed by a field assessment and determination per applicable guidelines. Assessment criteria will include hydrology, hydric soils, and vegetation. Areas where assessment criteria are met will then be delineated. The delineation will be conducted in the appropriate season (i.e., springtime) for identification of vernal pools. The USACE Contracting Officer's Representative (COR)/PM will be notified if any permits are required.

The following paragraphs provide details of the wetlands delineation and vernal pools survey field activities and resulting deliverables.

Wetlands Delineation: The boundaries between wetland and upland areas within the Site will be flagged in the field. Boundary lines will be based upon the wetland indicators and criteria defined in the USACE Wetlands Delineation Manual (1987) and the Regional Supplement to the USACE Wetland Delineation Manual: North Central and Northeast Region, Version 2.0 (USACE, 2012).

A map of the Site illustrating the wetland boundaries and locations of wetland flags, and data collection points will be developed upon completion of the field delineation. Accompanying report documentation will include: USGS and National Wetland Inventory (NWI) maps showing the site location and any wetland areas; soil survey map showing the distribution of soils within the Site including the location of hydric soils; summary of findings and delineation rationale, including topography and drainage, soils, vegetation, and hydrology for the Site including any accompanying data; map of GPS wetland boundary location points, USACE site survey data forms, and photographs.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

Vernal Pools Survey: Two site visits to each vernal pool feature are tentatively scheduled in the April-May period of 2020. Timing of the vernal pool surveys will be refined to local climatic conditions (i.e., temperature and precipitation) and further confirmed by observations of regional experts and updated on the regional amphibian migrations. The first survey will be scheduled approximately two weeks following reports of full wood frog choruses to target wood frog egg mass identification and enumeration. The second survey will be scheduled approximately two weeks following the first survey to target salamander egg mass identification.

A survey and documentation of each feature will be completed according to the guidelines outlined in the Maine Association of Wetland Scientists Vernal Pool Technical Committee, Vernal Pool Survey Protocol and guidance from Identifying and Documenting Vernal Pools in New Hampshire, Third Edition 2016. The highwater line of each vernal pool depression will be mapped using handheld GPS and desktop ortho digitization. Records will include general site conditions, the identification and abundance of species observed in each feature, with particular attention focused on the obligate species and their egg masses.

Net sweeps will be conducted in each feature to determine presence of fairy shrimp (*Eubranchipus* spp.), amphibian larvae, and or invertebrates. Additionally, visual surveys will be conducted out to 50 feet from the depressional highwater line in the adjacent critical terrestrial habitat.

17.2.3 Resource Receptor Inventory

A description of habitats potentially affected by site-related constituents as well as flora and fauna present or reasonably expected to be present in these habitats have been identified from the following sources:

- Notes from a site visit by PDT members on 27 June 2018;
- New York Natural Heritage Database Search for the Town of Lysander – New York Nature Explorer (NYSDEC, 2018a);
- Habitat Management Plan for the Three Rivers Wildlife Management Area 2018-2027 (NYSDEC, 2018b);
- New England Wildlife: Habitat, Natural History, and Distribution (DeGraff and Yamasaki, 2001);
- Three Rivers Bird Conservation Area (NYSDEC, 2018c); and,
- US Fish and Wildlife National Wetlands Inventory (USFWS, 2018).

Following are descriptions of Three Rivers WMA, the terrestrial habitat observed during the PDT site visit, wetland habitats near the AOCs (which are potential groundwater discharge points) as identified in the National Wetlands Inventory Database, and additional wildlife and habitats of concern identified in the New York Natural Heritage Database for the town of Lysander.

A general list of flora and fauna that have been observed in and around the site or that are expected to occur in this region or local habitat based on available species distribution data are provided below. The species identified are meant only to represent species broadly typical of this habitat type and may not necessarily occur within any AOC because of regional and local factors that affect species distribution.

Receptors identified as possibly present in the literature surveys will be verified by observations made during the wetland delineation and vernal pool surveys, as well as incidental observations during other Phase 1 field investigations and discussions with the Three Rivers Wildlife Management Area staff.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

The results from multiple lines of evidence from Phase 1 will be incorporated into the CSM and more formal wildlife surveys will be performed during Phase 2, if appropriate.

Three Rivers WMA

The Three Rivers WMA comprises 3,597 acres of land that is actively managed for the preservation of wildlife and natural communities. According to the *Habitat Management Plan for Three Rivers WMA 2018-2027*, there are 64 acres of open water, 468 acres of natural wetland, 113 acres of impounded wetland, and 20 miles of rivers and streams at Three Rivers WMA (NYSDEC, 2018b). AOC 4 is entirely located within the Three Rivers WMA.

According to the *Habitat Management Plan for Three Rivers WMA 2018-2027*, the following avian and mammalian wildlife, typical of central New York, is present in the Three Rivers WMA: beaver, mink, muskrat, white-tailed deer, cottontail rabbit, bobolink (*Dolichonyx oryzivorus*), blue-winged warbler (*Vermivora cyanoptera*), northern harrier (*Circus cyaneus*), bald eagle (*Haliaeetus leucocephalus*), American woodcock (*Scolopax minor*), ruffed grouse, and wild turkey. Reptiles present on the Three Rivers WMA include: common ribbonsnake (*Thamnophis sauritus*), Jefferson salamander (*Ambystoma jeffersonianum*), and snapping turtle (*Chelydra serpentina*) (b).

The Three Rivers WMA is identified as a New York State Bird Conservation Area (NYSDEC, 2018b). There are several known species of birds that are listed as endangered, threatened, or of special concern that may be present at Three Rivers WMA (NYSDEC, 2018b, NYSDEC, 2018c), including:

- American bittern (*Botaurus lentiginosus),* special concern
- Bald eagle (*Haliaeetus leucocephalus*), threatened
- Cerulean warbler (*Dendroica cerulea)*, special concern
- Common nighthawk (*Chordeiles minor)*, special concern
- Common tern (*Sterna hirundo)*, threatened
- Cooper's hawk (*Accipiter cooperii),* special concern
- Eastern whip-poor-will (*Caprimulgus vociferus)*, special concern
- Golden-winged warbler (*Vermivora chrysoptera*), special concern
- Grasshopper sparrow (*Ammodramus savannarum)*, special concern
- Henslow's sparrow (*Ammodramus henslowii),* threatened
- Horned lark (*Eremophila alpestris)*, special concern
- Least bittern (*Ixobrychus exilis)*, threatened
- Northern goshawk (*Accipiter gentilis)*, special concern
- Northern harrier (*Circus cyaneus)*, threatened
- Osprey (*Pandion haliaetus*), special concern
- Peregrine falcon (*Falco peregrinus)*, endangered
- Pied-billed grebe (*Podilymbus podiceps)*, threatened
- Red-headed woodpecker (*Melanerpes erythrocephalus*), special concern
- Red-shouldered hawk (*Buteo lineatus)*, special concern
- Sedge wren (*Cistothorus platensis)*, threatened
- Sharp-skinned hawk (*Accipiter striatus)*, special concern
- Upland sandpiper (*Bartramia longicauda)*, threatened
- Vesper sparrow (*Pooecetes gramineus),* special concern

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

- Yellow-breasted chat (*Icteria virens*), special concern

Mammals of conservation concern at Three Rivers WMA include:

- Indiana bat (*Myotis sodalist),* endangered
- Northern long-eared bat (*Myotis septentrionalis),* threatened
- small-footed bat (*Myotis leibii),* special concern.

Amphibians and reptiles of special concern include:

- Jefferson salamander (*Ambystoma jeffersonianum)*
- wood turtle (*Clemmys insculpta)*.

The following discussion provides a description of terrestrial habitat observed during the PDT site visit, wetland habitats near the AOCs as identified in the National Wetlands Inventory Database and additional wildlife and habitats of concern identified in the New York Natural Heritage Database for the Town of Lysander.

Terrestrial Habitat

The terrestrial habitat discussions for AOCs 1 and 4 are provided below.

AOC 1 – Ammonium Picrate Area: The majority of the Ammonium Picrate Area terrestrial habitat is forested and is very similar to that described in the following Section for the Bunker Area. The major difference being a much less dense understory layer. In general, vegetation composition and expected wildlife are similar between the two areas. The Ammonium Picrate Area has maintained trails and recreational grassy areas interspersed throughout the site.

AOC 4 – Bunker Area: The bunkers are covered with grass and at most places are periodically managed to discourage the rooting of trees and shrubs on top of the bunkers. Across the road from the bunkers, a heavily vegetated drainage ditch runs parallel to the road in front of each bunker. Terrestrial habitat bordering Igloo Road and associated bunkers consists primarily of grassy fields and northern deciduous forest. The following provides a brief description of the primary terrestrial habitat location within the Bunker Area.

Grass Fields

- Located predominantly along the northern portion of the Bunker Area although smaller grass field patches occur around each of the bunkers. The extent of grass maintenance around the bunkers is limited.
- Dominant vegetation observed in the grass fields are the herbaceous plants including various grass species (e.g., sweetgrass, bluestem), broomsedge (*Andropogon virginicus*)*,* common milkweed (*Asclepias syriaca*), goldenrod (*Solidago spp*.), common ragweed (*Ambrosia artemisiifolia*), mullein (*Verbascum spp*.), asters (*Aster spp*.), oxeye daisy (*Leucanthemum vulgare*), rudbeckia (*Rudbeckia spp*.), crown vetch (*Securigera varia*), thistles (*Cirsium spp*.), and yarrow (*Achillea millefolium*) among others. Saplings, shrubs and vines of various sizes (though most appear to be early successional) include southern arrowwood (*Viburnum*

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

dentatum), maple leaf viburnum (*Viburnum acerifolium*), smooth sumac (*Rhus glabra*), pin cherry (*Prunus pensylvanica*), gray birch (*Betula populifolia*), red maple (*Acer rubrum*), and sweetgum (*Liquidambar styraciflua*).

- Avian species observed or likely to occur in the grass field habitat include, among others, American robin (*Turdus migratorius*), grey catbird (*Dumetella carolinensis*), field sparrow (*Spizella pusilla*) chipping sparrow (*Spizella passerina*), tree swallow (*Tachycineta bicolor*), common crow (*Corvus brachyrhynchos*), northern mockingbird (*Mimus polyglottos*), mourning dove (*Zenaida macroura*), and American kestrel (*Falco sparverius*).
- Mammals expected to occur in the grass field habitat include: deer mouse (*Peromyscus maniculatus*), white-footed mouse (*Peromyscus leucopus*), house mouse (*Mus musculus*), northern short-tailed shrew (*Blarina brevicauda*), eastern cottontail (*Sylvilagus floridanus*), woodchuck (*Marmota monax*), raccoon (*Procyon lotor*), and white-tailed deer (*Odocoileus virginianus*).

Woodlands

- Eastern deciduous forest borders the Bunker area to the north and east; second growth forest of similar composition is located between the two bunker rows except at the northern end. The southeastern site border is a mix of maintained grassy areas and deciduous forest.
- Characteristic trees and shrubs observed included red maple, quaking aspen (*Populus tremuloides*), gray birch, red oak (*Quercus rubra*), sweet gum, poplar (*Populus spp*.), sassafras (*Sassafras albidum*), pin cherry, black cherry (*Prunus serotina*), eastern white pine (*Pinus strobus*), arrowwood , New York ironweed (*Vernonia noveboracensis*), smooth sumac, maple leaf viburnum, greenbrier (*Smilax spp*.), and oriental bittersweet (*Celastrus orbiculatus*).
- Avian species observed or likely to occur in the deciduous forested habitat include: northern flicker (*Colaptes auratus*), downy woodpecker (*Picoides pubescens)*, red-bellied woodpecker (*Melanerpes carolinus),* wood thrush (*Hylocichla mustelina*), white-breasted nuthatch (*Sitta carolinensis*), eastern phoebe (*Sayornis phoebe*), warbler spp., great horned owl (*Bubo virginianus*), barred owl (*Strix varia*), wild turkey (*Meleagris gallopavo*), ruffed grouse (*Bonasa umbellus*), red-tailed hawk (*Buteo jamaicensis*), and sharp-shinned hawk (*Accipiter striatus*).
- Mammals expected to occur in the grass field habitat include: deer mouse, white-footed mouse, house mouse, northern short-tailed shrew (*Blarina brevicauda*), gray squirrel (*Sciuris carolinensis*), eastern chipmink (*Tamias striatus*), red fox (*Vulpes vulpes*), long-tailed weasel (*Mustela frenata*), raccoon, and white-tailed deer.

Wetland Habitat

According to the National Wetlands Inventory Map managed by the USFWS (2018), there are several freshwater forested/shrub wetland and freshwater emergent wetland areas in the western portion of the site (primarily in the undeveloped areas of Three Rivers WMA). The freshwater forested/shrub wetland category consists of woody wetlands, forested swamp, or shrub bogs. Freshwater emergent wetlands are described as herbaceous marsh, fen, swale, and wet meadow (USFWS, 2018). There are several small

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

freshwater ponds located within the Radisson Community. A site-specific Wetland Delineation and Vernal Pool Identification will be conducted as noted in **Section 17.2.2**.

New York Natural Heritage Database

The New York Nature Explorer is the New York Natural Heritage Database online tool for finding out about the animals, plants, and habitats in areas of interest. Users may choose a county, town, or watershed, or may specify their own location on a map, and receive a list of the animals, plants, and natural communities that have been found there. Users may also choose a specific animal, plant, or natural community type, and receive a list of the counties, towns, or watersheds where it has been found.

Included in Nature Explorer are the rare plants, rare animals, and significant natural communities (such as forests, wetlands, and other habitat types) documented by the Natural Heritage database; birds documented during the second NYS Breeding Bird Atlas from 2000 to 2005; and reptiles and amphibians documented during the NYS Herp Atlas from 1990 to 1999.

The NYSDEC Nature Explorer also identified two endangered plants in the Lysander Area (NYSDEC, 2018a) not listed the Three River WMA Management Plan (NYSDEC 2018b):

- Carey's Smartweed (*Persicaria careyi*), endangered
- Creeping Juniper (*Juniperus horizontalis*), endangered.

Neither of these species in expected in the AOCs under evaluation.

17.2.4 Selection of Sampling Locations

Prior to the field activities, Bluestone will flag proposed sampling locations. The number of samples and preliminary locations for surface soil, subsurface soil, and groundwater are discussed in **Worksheet #11** and shown on **Figures 11-1 through 11-5**. Based on conditions encountered in the field, it may be necessary to modify the primary sampling locations. Reasons for modifications include the following:

- Inability to secure right-of-entry from property owners (**Subsection 17.2.1**);
- Overhead obstruction or subsurface utilities (**Subsection 17.2.6**);
- Inaccessibility (**Subsections 17.2.2, 17.5, 17.6, 17.7, and 17.8**);
- DPT refusal before target depth could be reached to collect samples (**Subsections 17.7 and 17.8**); and,
- Other unexpected field conditions (**Subsections 17.2.2, 17.5, 17.6, 17.7, 17.8, and 17.9**).

17.2.5 Selection of Background Sampling Locations

Areas selected for background samples are provided in **Figure 11-5** in **Worksheet #11**. The primary purpose of background sampling is to evaluate surface and subsurface conditions outside the area of influence (i.e., topographically upgradient/upslope) of the historical DoD operations and any associated environmental impacts, in order to evaluate the degree of impact to the Site and downgradient areas caused by historical site use. Background sampling locations must be placed at a distance far enough away from the influence of DoD operations for each AOC so as not to be impacted by Site activities, and yet close enough to represent typical soil types and chemistry present at each AOC. In addition, the

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

upgradient groundwater samples will be located so as to be representative of the quality of groundwater flowing onto the site.

Proposed soil and groundwater sampling locations shown in **Figure 11-5** are tentative. The sample locations will be refined based on field conditions and Geoprobe® access. Following the compilation and interpretation of groundwater elevation data, background and upgradient (or cross gradient, if necessary) well locations will be identified, and these areas will be sampled. Other potential sources of non-DoD-related contamination will be considered (i.e., the golf course proximal to AOC 3) when selecting final upgradient well locations. Background soil samples will be collected at locations believed to be free of impact from DoD activity and BTVs will be calculated. Site investigation samples will be compared to the BTVs as applicable, to evaluate anthropogenic non-DoD contamination and site contamination.

Wherever possible, background samples for surface soil will be co-located with the subsurface soil samples. A total of 15 surface and 30 subsurface soil samples will be collected from each of the geologic/surficial units identified within the Radisson Community to represent background conditions in undeveloped land. Based on nature of constituents of concern (DNCB, 2,4-DNP, and picric acid) at AOC 4 soil and groundwater, no background sampling is necessary.

Background sample analyses will include TOC and grain size distribution (including hydrometer analysis), as these particular soil characteristics are most indicative of the degree of similarity when comparing soil types. Following preliminary testing, background samples that are determined to be an appropriate match to the respective AOC sample locations, will be accepted for chemical analysis, provided that laboratory holding times have not been exceeded. Background sampling will be conducted per AOC, to determine the presence of constituents (if any) in accordance with **Worksheet #11**. Any background soils that vary too widely in composition from the respective AOC soils will not be approved for chemical analysis. In the event of a delay due to laboratory backlog, the background samples will be submitted for chemical analysis to meet holding times, regardless of the status of grain size analyses. Subsequently, if the respective soil comparisons do not yield a 'match', the background sample will be discarded. Bluestone will attempt to obtain 15 viable background samples; however, no more than two rounds of sampling will be performed.

The three major surface geologic units at the Former NYOW Site are diamicton (Pd), silt and clay (Psc), and stratified sand, silt and gravel (Ps). The depositional environments and other geologic and hydrogeologic characteristics for these three units are detailed in **Worksheet #10, Sections 10.2.3 and 10.2.4**. Geologic maps presented in **Worksheet #10** include Glacial Geology in **Figure 10-8** (Muller and Cadwell, 1986); Surficial Geology in **Figure 10-9** (Pair, 2014); and Bedrock Geology in **Figure 10-10** (Rickard and Fisher, 1970).

Proposed background sample locations are presented in **Figure 11-5**; however, actual sample locations may vary based on rights-of-entry and/or conditions encountered in the field. For example, samples to be collected along roadways will be placed a minimum of 10 feet from paved areas, to avoid residual impacts from the asphalt (if any).

A total of nine (9), three per applicable AOC (1, 3, and 5) upgradient groundwater samples will be collected. Preliminary locations of these samples are shown in **Worksheet #11**, **Figure 11-5**.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.2.6 Overhead Obstruction and Subsurface Utility Mark-out

The FTL or designee and the DPT subcontractor will walk to each flagged soil boring location to document potential overhead obstructions. Currently, high power transmission lines cross overhead at AOC 1. If any proposed DPT locations are proximal to or beneath power lines, Bluestone will contact the local electric utility to determine the voltage and adhere to the requirements of OSHA Standards 29 CFR 1926.1408 for the minimum distance the equipment must be kept from the lines.

Prior to any intrusive activities on the Site, subsurface utility clearance will be conducted to ensure that no buried utilities are damaged. An initial call to "Dig Safely New York" (1-800-962-7962, or locally at 811) will be placed to identify any publicly-owned utility lines in the area (note that a request can be submitted online at https://www.digsafelynewyork.com/). In addition, a third-party utility locator will then conduct a series of geophysical field tests to identify any subsurface utilities located on private property (i.e., electrical, gas, communication, water, sewer, steam, etc.). The private utility survey will verify and mark underground utilities located proximal to proposed sampling locations, and will outline safe zones to advance soil borings, in case one or more boreholes must be offset during the progression of work.

Field Procedures for these activities are detailed in:

- SOP No. 06 – Field Documentation;
- SOP No. 08 – Electromagnetic Induction (EM)31 Survey;
- SOP No. 09 – Ground-Penetrating Radar (GPR) Survey; and,
- SOP No. 18 – Utility Clearance.

17.2.7 Vegetation Clearance

Any necessary vegetation and/or tree clearing will be approved in advance by USACE and the property owner. Clearing activities will be conducted by a subcontractor under the supervision of the Bluestone field team. If possible, disturbed areas on private or public properties will be restored to the previous condition.

17.3 MOBILIZATION

17.3.1 Field Staging Area

A field staging area will be established in a central location approved by USACE. The staging area will be large enough to accommodate portable sanitary facilities (and service access), a drive-on pad for the decontamination of drilling and sampling equipment, a second drive-on pad for invasive species rinse water, staging of DPT equipment and supplies, and vehicle parking. A Conex box or lockable trailer will be leased for the long-term storage of equipment and supplies. In addition, a secured chain-link fenced area will be constructed for the staging of investigation derived wastes (IDW) and trash. The decontamination pad will be constructed with water-proof materials and cinder blocks (or similar), for the containment of decontamination fluids. Any fluids that collect in the bottom of the pad will be pumped into 55-gallon drums, as needed, and labeled appropriately. In addition, the secure fenced area will be used to stage drums containing impacted drill cuttings, if applicable, pending proper waste characterization and disposal.

At the completion of the field activities, all decontamination pad materials and DPT equipment, supplies, and vehicles will be removed from the staging area, unless otherwise instructed by USACE. If possible, the

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

staging area will be restored to its original condition. Bluestone personnel will be on Site to oversee and document the site restoration field activities.

17.3.2 Invasive Species Mitigation

To mitigate the spread of invasive species, all vehicles that enter or exit AOC 4 will be rinsed with potable water (no soaps or detergents) prior to entering or leaving AOC 4. The rinse water will be managed separate from equipment decontamination IDW using a dedicated containment pad located at the staging area. All rinse water will be managed and disposed in accordance with applicable State regulations.

Additionally, all field staff will be trained on the identification and significance of the swallow-wort as an invasive species, and on procedures to avoid its spread.

17.3.3 Field Equipment and Supplies

Bluestone will obtain the necessary equipment and field supplies for each part of the Phase 1 field investigation, including purchased supplies and rental items. Calibration records (as applicable) will be maintained daily, and each piece of equipment will be calibrated at the start of each workday, and again during work activities if needed. Equipment items requiring daily calibration include the water quality multi-parameter meter (YSI Pro Plus, or similar) and the PID (MiniRAE® 3000, or similar). In addition, equipment will be rotated as needed for factory calibrations and cleaning. All equipment will be inspected prior to use.

17.3.4 Field Planning Meetings

Prior to field activities, each field team member will review the project plans (QAPP and APP/SSHP) and participate in a field planning meeting conducted by the Field Team Leader or designee to become familiar with the history of the Site, health and safety requirements, roles and responsibilities, field procedures, field data collection and management procedures, sample nomenclature, communication procedures, and related quality control (QC) requirements. At the start of each workday, the SSHO will conduct a brief health and safety tailgate meeting for all field personnel, and again upon the arrival of new field personnel and/or approved visitors. Supplemental meetings may be conducted as required by any changes in site conditions, work activities, or to review field operation procedures.

Field Procedures for these activities are detailed in:

- SOP No. 02 – Equipment Calibration and Maintenance; and,
- SOP No. 06 – Field Documentation.

17.4 SURFACE GEOPHYSICAL SURVEYS – AOC 3 AND AOC 5

Geophysical survey activities will be conducted at AOC 3 and AOC 5 to guide and/or confirm the proposed soil and groundwater sampling locations. Geophysical surveys will be conducted by a qualified firm, and will consist of electromagnetic induction methods (e.g., Geonics® EM-31 and/or EM-61) and ground-penetrating radar (GPR). Bluestone will provide oversight during the survey and will document initial findings. The proposed geophysical transects are based on interpretations of aerial photographs conducted by the AGC (AGC, 2019), as presented in **Worksheets #10 and #11**.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

The geophysical surveys will be conducted in accordance with EM 1110-1-1802 Geophysical Exploration for Engineering and Environmental Investigations (USACE, 1995) and ASTM Method D6429 *Standard Guide for Selecting Surface Geophysical Methods* (ASTM, 2011). Location control will be provided using differential Global Positioning System (GPS), to ensure data are compatible with Geographic Information System mapping programs. The data will be recorded in the instrument's memory and transferred onto a laptop computer in the field. Data will be contoured and overlain on site base maps.

Field Procedures for these activities are detailed in:

- SOP No. 04 – Global Positioning Survey (GPS);
- SOP No. 06 – Field Documentation;
- SOP No. 08 –EM-31 Survey; and,
- SOP No. 09 – Ground-Penetrating Radar (GPR) Survey.

17.5 ISM SURFACE SOIL INVESTIGATION

17.5.1 Introduction and Rationale for ISM

ISM will be completed for the investigation of surface soils in AOC 4, in accordance with the Interstate Technology and Regulatory Council (ITRC) guidance document, *Incremental Sampling Methodology* (2012) and applicable updates, pending publication in Fall 2020; and USEPA's *Standard Operating Procedure for Incremental Sampling Methodology for Soil*, dated 15 December, 2015. ISM is a structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of mean contaminant concentrations in a volume of soil targeted for sampling (ITRC, 2012). As part of the ISM approach, a DU represents the smallest volume of soil for which a characterization, risk-based, or remedial decision is made. DUs are based on project-specific needs and site-specific DQOs.

17.5.2 Sampling Approach

A total of 12 DUs will be evaluated for AOC 4, one DU at each FUDS-eligible bunker. Each DU will be comprised of a one-half acre grid that begins at the roadway and includes the bunker, bunker aprons, and surrounding cleared area. Each DU grid will be comprised of 30 roughly equal segments, from which one sample increment will be collected per ISM sample. For QC purposes, ISM samples will be collected in triplicate sample sets at each DU, for a total of 36 ISM samples. Corresponding soil increments will be distinguished by a designated color of pin flag, and will be collected from a consistent location within each grid segment. Each ISM sample will be comprised of 30 soil increments composited into one representative bulk sample (90 increments per DU, 1,080 soil increments total). Sampling grids will be horizontally located in the field using a Trimble® GPS unit.

17.5.3 Sampling Methodology

ISM surface sample increments will be collected using a dedicated volumetric sampling device (LaMotte 1055 Soil Sampler, or similar), from the 0 to 1 ft bgs interval. The triplicate sample sets will be organized as follows:

- Primary Sample (first path of travel)
- Duplicate Sample (second path of travel); and,

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

- Triplicate Sample (third path of travel).

In order to comply with laboratory standards, each composited ISM sample should be a maximum volume approximately 1,000 grams. As such, the sampling tool must be conducive to collecting a volume of 30 to 35 grams of soil per increment. A digital scale will be used to ensure that appropriate soil volumes are collected. Soil increments will be weighed and transferred into the laboratory-prepared plastic bags for homogenization by the laboratory prior to chemical analysis.

Bluestone will document general descriptions of soil, and any environmental impacts observed during sampling. Sampling equipment will be decontaminated between sample sets, in accordance with Bluestone's SOP No. 05 – Decontamination of Field Equipment. ISM sampling is detailed in Bluestone's SOP No. 21 – Incremental Sampling Methodology.

Field Procedures for these activities are detailed in:

- SOP No. 05 – Decontamination of Field Equipment
- SOP 06 – Field Documentation
- SOP No. 13 – Sample Packing and Shipping
- SOP No. 16 – Surface Soil Sampling
- SOP No. 21 – Incremental Sampling Methodology (ISM)
- Subsection 17.9 – Decontamination Procedures

17.6 GEOPROBE® DIRECT-PUSH TECHNOLOGY (DPT) SUBSURFACE INVESTIGATION

A DPT subcontractor will be used for the collection of surface and subsurface soil samples, and also groundwater samples. Since the objective of sampling at depth is to evaluate separate zones of potential impacts, a Geoprobe® brand Dual Tube Sampling System will be used to collect continuous soil cores. Based on the local geology and estimated target depths, this system is the most efficient and cost-effective method to evaluate the quality of discrete soil and water-bearing zone(s) from ground surface to DPT refusal. The Dual Tube System allows for geologic logging, discrete soil and groundwater sample collection, and measurement of groundwater levels simultaneously.

The Dual Tube Sampling System allows for the collection of continuous soil cores in both the saturated and unsaturated zones by employing two separate barrels. The large-diameter barrel functions as the outer casing and receives the driving force of the hammer. The small-diameter barrel holds the sample line in place as the casing is advanced and is then retracted to retrieve the filled acetate sleeve (Macro-Cores®). In turn, the inner core may be recovered without the threat of cross-contamination. Following the collection of soil samples, the borehole can be converted into a temporary groundwater well.

The diamicton/till material beneath portions of AOCs 1 and 5 may not be conducive to advancement of a dual barrel. Other issues that may occur with the Dual Tube Sampling System include insufficient soil volumes available from the narrow inner barrel for sampling, insufficient groundwater volumes, and/or incomplete groundwater elevation data. Based on subsurface conditions observed in the field, the approach can be modified to standard DPT soil borings (single 2 to 3-inch barrel lined with Macro-Core® sleeves) to achieve project objectives. Groundwater elevation data and groundwater sampling details are discussed further in **Section 17.6.4**.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.6.1 Geologic Logging of Soil Cores

Soils will be logged by an experienced glacial geologist in general accordance with the American Society for Testing and Materials (ASTM) Standard 2488-17-E1, *Standard Practice for the Description and Identification of Soils (Visual-Manual Procedures)* (ASTM, 2017a), and ASTM Method D2487-17, *Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)* (ASTM, 2017b), and ASTM Method D6286-19, *Standard Guide for Selection of Drilling and Direct Push Methods for Geotechnical and Environmental Subsurface Site Characterization* (ASTM, 2019). Descriptions of soil cores will include media type (grain size), degree of sorting, color, particle shape, consistency [soft to hard], degree of cementation/cohesion, moisture, interpreted geologic formation (i.e., till, lacustrine, outwash, etc.), and other observations (i.e., odors, staining, visual impacts, etc.). Soil cores will be screened every six inches for the presence of organic vapors using a PID. Any observations made by field personnel with respect to visual impacts (i.e., elevated PID measurements, staining, odors, etc.) will be documented in the field logbook and conveyed by field personnel to the Bluestone PM at the end of each workday. Any major issues or findings that may alter the progression of field work will be conveyed by the Bluestone PM to the USACE PM immediately along with proposed solutions, and Bluestone field personnel will await direction from the Bluestone PM and USACE personnel on how to proceed.

17.6.2 Surface Soil Sampling

Surface soil samples will be collected as both discrete grab and ISM samples during the Phase 1 Investigation. Locations of surface soil samples are shown in **Worksheet #11, Figures 11-1 through 11-5**. Discrete surface soil samples will be collected at AOCs 1, 3, 4, and 5; ISM surface samples will be collected at AOC 4 only, to assess the nature and extent of potential impacts and for representative background locations, as applicable.

Discrete grab surface soil samples will be collected from the 0 to 1 ft bgs interval in the Macro-Core® sleeve. If an insufficient volume of soil is available for the required analyses, additional soil will be collected from around the borehole using a disposable plastic scoop. Soils will be homogenized by hand inside a Ziploc® bag and transferred directly to the sampling jars.

Bluestone will not collect soil samples in areas where building debris is observed at the ground surface. Areas containing building debris will be documented in writing and photographed.

Field Procedures for these activities are detailed in:

- SOP No. 02 – Equipment Calibration and Maintenance
- SOP No. 05 – Decontamination of Field Equipment
- SOP 06 – Field Documentation
- SOP 07 – Field Screening Methodology
- SOP No. 10 – Storage and Sampling of IDW
- SOP No. 11 – Geologic Logging
- SOP No. 13 – Sample Packing and Shipping
- SOP No. 16 – Surface Soil Sampling
- Subsection 17.9 – Decontamination Procedures

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.6.3 Subsurface Soil Sampling

Subsurface soil samples will be co-located with the surface soil samples. The subsurface soil analytical data will be used to delineate the nature and extent of constituents at depth in each AOC, and to determine if residual constituents may be impacting local groundwater quality. Preliminary locations for DPT subsurface soil sampling are shown in **Worksheet #11, Figures 11-1 through 11-15**.

Soil borings will be advanced in 5-ft lengths until refusal or to a specified target depth is encountered and logged continuously by an experienced glacial geologist, as described above. If shallow refusal is encountered (i.e., less than 5 ft bgs), three attempts will be made within 10 feet of the original location to attain the targeted depth. If all three attempts meet shallow refusal, installation of the boring will be stopped, and the drilling methodology will be reassessed during Phase 2. The entire length of core will be logged and photographed prior to sample collection. The depth intervals of soils samples will be selected by the onsite glacial geologist. One subsurface sample will be collected from the 1 to 5 ft bgs interval and one from the 5 to 10 ft bgs interval.

For VOC analysis of subsurface soils, a TerraCore® kit will be used to collect the soil from the mid-point of the core (unless visual impacts are observed, at which point the sample will be collected from the impacted interval). Additional discrete (grab) samples will be collected in intervals where visual impacts are observed, if any (i.e., elevated PID measurements, staining, and/or odors)), from the 12-inch interval centered along the observed impacts. If no visual impacts are observed, the remainder of recovered soils from 1 to 5 ft and from 5 to 10 ft bgs will be homogenized inside a Ziploc® bag and transferred into the sampling jars.

Depending on subsurface conditions, DPT refusal may occur at very shallow depths, owing to the variability of glacial deposits, or the presence of anthropogenic debris beneath the surface. If refusal is encountered within the first 5 ft bgs, the material will be recovered, and a surface soil sample will be collected. If the remaining soil core is insufficient to collect a full sample, the soil will be discarded and the DPT rig will be set up at a location 5 to 10 ft away from the first borehole in any direction and the process will be repeated. If there are three unsuccessful attempts to advance the core barrel to a depth where sufficient subsurface soil can be collected, the location will be sampled for surface soil (0 to 1 ft bgs) and if possible, one subsurface sample between 1 ft bgs and the depth of refusal. The field geologist will examine the DPT shoe for indications of weathered bedrock vs. debris obstruction or cobble/boulder.

Field Procedures for these activities are detailed in:

- SOP No. 02 – Equipment Calibration and Maintenance
- SOP No. 05 – Decontamination of Field Equipment
- SOP 06 – Field Documentation
- SOP 07 – Field Screening Methodology
- SOP No. 10 – Storage and Sampling of IDW
- SOP No. 13 – Sample Packing and Shipping
- SOP No. 15 – Subsurface Soil Sampling
- Subsection 17.9 – Decontamination Procedures
- Worksheet 11 – Project/Data Quality Objectives

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.6.4 Groundwater Sampling

Groundwater samples will be collected in conjunction with the DPT soil investigation. As described in **Section 17.6**, the objective is to collect discrete overburden groundwater samples at AOCs 3, 4, and 5, and from each water-bearing zone from ground surface to DPT refusal at AOC 1. At refusal, and upon completion of logging and soil sampling, each borehole will be converted into a temporary monitoring well, from which a groundwater sample will be collected. In AOC 1, if any additional water-bearing zones are identified in shallower intervals (as evident in soil cores), each additional zone will be sampled by advancing a separate soil boring and constructing a temporary well at each target depth. The temporary wells will be placed as a well cluster (within a 5-ft radius around the primary soil boring) and constructed with 1-inch screened PVC casing. A sand pack will be placed in the annulus around the well, to two feet above the screen, followed by two feet of bentonite chips or pellets, and hydrated to provide a seal. Well casings will be cut to grade and capped/covered for the duration of use. The flush-mount finish is intended to eliminate drawing attention (and potential tampering) and creating a safety hazard to surrounding animal life and/or local residents.

Discrete groundwater samples will be collected using a peristaltic pump provided that hydraulic head is within an acceptable range (typically no more than 30 to 35 ft bgs). If hydraulic head exceeds 30 to 35 feet, a submersible bladder pump will be used to collect samples. Field parameters (i.e., pH, temperature, DO, specific conductivity, turbidity, and oxidation-reduction [ORP]) will be recorded at the time of sample collection using properly calibrated instruments. Standard Hydrogen Electrode (SHE) corrections for ORP field measurements will be completed at the end of the sampling event, for reporting purposes. Due to the potential for elevated turbidity in the grab groundwater samples, aliquots for metal and inorganic constituents will be field filtered when filling the sample bottles. Each filter will be used once and discarded. Aliquots for VOC and SVOC analysis will be collected first but will not be field filtered. The temporary wells will be left in place for the duration of the DPT investigation in order to obtain a synoptic round of water levels. Once each temporary well is surveyed and monitoring activities are complete, the wells will be properly abandoned in accordance with NYSDEC Policy CP-43 (NYSDEC 2009).

Locations of the DPT borings (and corresponding groundwater samples) are shown in **Worksheet #11, Figures 11-1 through 11-5**.

Field Procedures for these activities are detailed in:

- SOP No. 02 – Equipment Calibration and Maintenance
- SOP No. 05 – Decontamination of Field Equipment
- SOP No. 06 – Field Documentation
- SOP No. 07 – Field Screening Methodology
- SOP No. 10 – Storage and Sampling of IDW
- SOP No. 12 – Low-Flow Groundwater Sampling and Sampling with Bailer
- SOP No. 13 – Sample Packing and Shipping
- Subsection 17.9 – Decontamination Procedures
- Worksheet 11 – Project/Data Quality Objectives.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.7 WATER LEVEL MEASUREMENTS

Since the installation of permanent groundwater monitoring wells was eliminated from Phase 1 activities, groundwater elevation data will be obtained from the temporary monitoring wells installed in conjunction with the advancement of soil borings. The temporary wells will be left in place for the duration of the DPT investigation in order to obtain a synoptic round of water levels at each AOC. Depth-to-water measurements will be collected from ground surface using a Solinst® Model 122 Interface Meter (in case LNAPL is encountered). A licensed surveyor will locate each of the temporary wells and record respective ground surface and measuring point elevations.

The objective for obtaining groundwater elevation data is to refine/determine the hydraulic gradient at each AOC, and subsequently identify potential discharge areas such as wetlands or small streams, in addition to potential constituent migration pathways. The hydraulic gradients in AOC 3 and AOC 5 are currently unknown.

Once each temporary well is surveyed and monitoring activities are complete, the wells will be properly abandoned in accordance with NYSDEC Policy CP-43 (NYSDEC 2009).

Field Procedures for these activities are detailed in:

- SOP No. 01 – Abandonment of Monitoring Wells and Piezometers;
- SOP No. 02 – Equipment Calibration and Maintenance;
- SOP No. 03 – Design and Installation of Monitoring Wells and Well Development;
- SOP No. 04 – GPS Survey;
- SOP No. 05 – Decontamination of Field Equipment;
- SOP 06 – Field Documentation;
- SOP 19 – Groundwater Elevation Monitoring; and,
- Subsection 17.9 – Decontamination Procedures.

17.8 SAMPLE MANAGEMENT

17.8.1 Sample Nomenclature

Each sample will be assigned a unique sample identification number that appears on all sample labels, chains of custody (COCs), field logbooks, and all other applicable documentation forms. **Worksheet 18, Section 18.2** provides further details on sample labeling.

17.8.2 Sample Tracking

The COC serves as physical evidence of sample custody over the life of the sample batch. Field personnel will initiate a COC at the time of sample collection. All custody transfers of the sample batch will be recorded on the COC by the individual relinquishing and the receiver of the samples, and signed and dated, and time stamped at the time of transfer. Each cooler is assigned a separate COC, on which only the samples packed in that cooler are listed. After completing the COC, the original will be enclosed in a plastic bag and taped to the inside lid of the cooler.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

Field Procedures for these activities are detailed in:

- SOP No. 06 – Field Documentation; and,
- SOP No. 13 – Sample Packing and Shipping.

17.9 DECONTAMINATION PROCEDURES

17.9.1 Decontamination Procedure

The required decontamination procedures for small sampling equipment are as follows, and must be completed after every sample:

1. Wash and scrub with low-phosphate detergent;
2. Rinse with tapwater;
3. Lightly spray with appropriate solvent (i.e., methanol for organics, nitric acid [10%] for inorganics);
4. Rinse with de-ionized water;
5. Place small equipment on clean plastic sheeting and allow to air dry; and,
6. Wrap in aluminum foil for transport.

The required decontamination procedures for large sampling equipment are as follows:

1. Transport the DPT rig and/or rods and shoes to the decontamination pad located in the staging area;
2. Steam clean the barrel and rods and shoes between soil boring locations;
3. Steam clean the DPT rig between AOCs; and,
4. Decontamination fluids will be pumped into 55-gallon drums for later characterization and disposal.

In addition, large equipment such as the DPT rig will be decontaminated at the end of the field effort. It should be noted that the Geoprobe® rig must be equipped with multiple barrels, rods and shoes, so that drilling can proceed without frequent interruption.

17.9.2 Decontamination Equipment

- Steam cleaner
- Distilled/de-ionized water
- Potable water
- Solvent sprays – methanol and nitric acid (10%)
- Polyethylene sheeting
- Cinder blocks or similar
- Portable generator
- 5-gallon pails
- 55-gallon drums
- Utility knife
- Brush

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

- Non-phosphate detergent

Field Procedures for these activities are detailed in:

- SOP No. 05 – Decontamination of Field Equipment; and,
- SOP No. 10 – Storage and Sampling of IDW.

17.10 MANAGEMENT OF INVESTIGATION-DERIVED WASTES

IDW generated during the investigation, including soil cuttings, groundwater sampling purge water (Phase 2 only), decontamination fluids, and other sampling equipment etc. will be containerized separately and stored on secondary containment pads in the designated field storage area. Waste management procedures for IDW are based on the requirements specified in Title 40 of the CFR, Part 262 (40 CFR 262) *Standards Applicable to Generators of Hazardous Waste*, all applicable State regulations (including but not limited to 6 NYCRR Part 360, 6 NYCRR Part 370-374 and 376), and industry best management practices.

Field Procedures for these activities are detailed in:

- SOP No. 06 – Field Documentation;
- SOP No. 10 – Storage and Sampling of IDW; and,
- SOP No. 13 – Sample Packing and Shipping.

17.11 SITE RESTORATION

Bluestone will procure a subcontractor for the removal and proper disposal of all waste media (i.e., soil cuttings, decontamination fluids, secondary containment materials, disposable sampling supplies and equipment). Representative soil and wastewater samples will be collected and analyzed to characterize the IDW. General trash such as gloves, food wrappers, Macro-Core® sleeves that were not in contact with impacted soil, etc. will be placed in trash bags and discarded with municipal wastes.

If necessary, a landscape contractor will be procured to restore to the extent possible, any areas disturbed or damaged during site setup or sampling activities to pre-existing conditions. NYSDEC's list of approved seeds and seeding rates will be used in restoration efforts. A photographic log will be maintained for each AOC to document conditions before sampling and after restoration.

Field Procedures for these activities are detailed in:

- SOP No. 02 – Equipment Calibration and Maintenance;
- SOP No. 05 – Decontamination of Field Equipment;
- SOP No. 06 – Field Documentation;
- SOP No. 10 – Storage and Sampling of IDW;
- SOP No. 13 – Sample Packing and Shipping; and,
- Subsection 17.9 – Decontamination Procedures.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

17.12 REFERENCES

AGC, 2019. NYOW – Baldwinsville, New York, Historical Photo Analysis. Draft. Prepared for USACE New England District.

American Society for Testing and Materials (ASTM), 2011. ASTM Method D6429-99(2011)e1. Standard Guide for Selecting Surface Geophysical Methods.

ASTM, 2017a. ASTM Method D2488-17e1. Standard Practice for the Description and Identification of Soils (Visual-Manual Procedures).

ASTM, 2017b. ASTM Method D2487-17. Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System).

ASTM, 2019. ASTM Method D6286-19. Standard Guide for Selection of Drilling and Direct Push Methods for Geotechnical and Environmental Subsurface Site Characterization.

DeGraff, R.M. and Yamasaki, M., 2001. New England Wildlife: Habitat, Natural History, and Distribution.

Maine Association of Wetland Scientists Vernal Pool Technical Committee. 2014. Vernal Pool Survey Protocol, April.

Muller, E.H. and Cadwell, D.H., 1986. Surficial Geologic Map of New York, Finger Lakes Sheet, New York Geologic Survey, Education Department, Albany NY.

New Hampshire Fish and Game Department. 2016. Identifying and Documenting Vernal Pools in New Hampshire, Third Edition. Nongame and Endangered Wildlife Program. Marchand, Michael (Editor).

NYSDEC, 1997. The Freshwater Wetlands Act. 6 NYCRR Part 663-665.

NYSDEC, 2018a. New York Nature Explorer Town Results Report, Lysander, New York. www.dec.ny.gov/natureexplorer.

NYSDEC, 2018b. Habitat Management Plan for Three Rivers Wildlife Management Area 2018-2027. Prepared by the Division of Fish and Wildlife, Bureau of Wildlife, March 2018. https://www.dec.ny.gov/docs/wildlife_pdf/yfithreeriversh mp.pdf

NYSDEC, 2018c. Three Rivers Bird Conservation Area. https://www.dec.ny.gov/animals/ 32035.html.

NYSDEC, 2009. Policy CP-43: Groundwater Monitoring Well Decommissioning Policy, NYSDEC, November 2009.

Pair, Donald L., 2014. Surficial Geology of the Baldwinsville Quadrangle, Onondaga County, New York. New York State Museum, New York State Geological Survey.

Rickard, L.V., 1969. Stratigraphy of the Upper Silurian Salina Group. University of the State of New York, State Education Department, Albany, NY.

QAPP Worksheet # 17: Sampling Design and Rationale, Continued

USACE, 2012. Regional Supplement to the USACE Wetland Delineation Manual: North Central and Northeast Region, Version 2.0

USACE, 1995. Geophysical Exploration for Engineering and Environmental Investigations. EM 1110-1-1802.

USACE, 1987. *Wetlands Delineation Manual*. Wetlands Research Program Technical Report Y-87-1.

USEPA, 2013. Science and Ecosystem Support Division Operating Procedure – Pore Water Sampling. SESDPROC-513-R2.

USFWS, 2018. National Wetlands Inventory, Wetlands Mapper. www.fws.gov/wetlands/data/Mapper.html.

QAPP Worksheet #18: Sampling Locations and Methods
(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

18.1 SAMPLING LOCATIONS

Samples will be collected at four AOCs and three background areas. Proposed sampling locations are shown on **Worksheet 11, Figures 11-1 through 11-5**. The field sampling will include:

- Up to 64 DPT groundwater samples (including 9 background samples);
- Up to 303 subsurface soil samples (including 90 background samples);
- Up to 124 surface soil samples (at 0 to 1 ft bgs, including 45 background samples); and
- Up to 36 ISM surface soil samples (12 DUs times 3 replicates collected at 0 to 1 ft bgs).

18.2 SAMPLE LABELING

Each sample container will be labeled with the project name, unique sample identification number, analysis method number, date and time collected, preservative (if any), and will be initialed by the sampler. All samples will be assigned a unique sample identifier (alpha-numeric code). The purpose of the numbering system is to assist in the tracking of samples and to facilitate retrieval of analytical results. The sampling numbers will be used on the sample labels, sample tracking forms, COC forms, field logs, and all other applicable documentation.

For the NYOW Phase 1 field work, the field sample identifiers will follow the "Recommended Naming Conventions for Locations, Field Samples, and Library File Nomenclature" – Appendix B of the most recent FUDSChem Database User's Manual in force when the Location Definition Information (LDI) tables are uploaded and Event Planning is completed in FUDSChem. Example sample IDs based on the 30 April 2020 User's Manual are presented below.

Sample	Example Nomenclature
Surface Soil, AOC 1, DPT Location 2 (1st depth interval, 0-1 ft bgs)	NYOW-01-PH002-A
ISM Sample, AOC 4, DU/Bunker 1, Surface Soil, Replicate #1	NYOW-04-DU001-SL-A
Subsurface Soil, AOC 3, DPT Location 7, 3rd depth interval	NYOW-03-PH007-C
Groundwater from temporary well, AOC 4, well #3 (not part of a well cluster)	NYOW-04-WL003
Groundwater from temporary well, AOC 1, well #4, second well in cluster	NYOW-01-WL004B
IDW, Aqueous, 3rd Sample	NYOW-IDWA003
IDW, Solid, 2nd Sample	NYOW-IDWS002

Where:

- NYOW = New York Ordnance Works
- PH = Direct push technology
- SL = Surface location (surface soil)
- WL = Well

Discrete sample field duplicates will be collected as blind to the laboratory. For the NYOW Phase 1 field work, the sample numbers will be in the following format: Location Type-DUP-Sequence_Date. For example, the second field duplicate collected from a borehole on 9/30/20 would be PH-DUP-002_200930.

QAPP Worksheet #18: Sampling Locations and Methods, Continued

The time noted on the COC will be midnight. The primary sample with which the duplicate is associated along with the actual collection time of the duplicate sample will be noted in the field log.

Trip blanks, equipment blanks, and other blanks not tied to specific locations, will be given unique names every event. For the NYOW Phase 1 field work, the sample numbers will be in the following format: Blank Type-Number_YYMMDD. Where:

Blank Type is an abbreviation indicating the type of blank as follows.

- EB Equipment Rinseate Blank
- TB Trip Blank

For example, the first equipment rinseate blank collected on 9/30/20 would be EB-01_200930. The equipment rinsed and sampling team which used the equipment will be noted in the field log so that equipment rinseate blanks may be associated with the appropriate field samples.

A detailed map will be maintained in the field log noting the location of each sample location. The depth bgs will be recorded in the field notes. Sample packaging, custody, and transportation are discussed in **SOP #13**.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times
(UFP-QAPP Manual Section 3.1.2.2)
(EPA 2106-G-05 Section 2.3.2)

Laboratory: Eurofins TestAmerica, Denver

Ship samples to:

Eurofins TestAmerica, Denver
Attn: Sample Receiving
4955 Yarrow Street
Arvada, CO 80002-4517

POC: Patrick McEntee, Patrick.McEntee@testamericainc.com, 303-736-0107

List of Required Accreditations/Certifications: DoD ELAP, version 5.3 (see **Appendix C**)

Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
VOCs	SO	8260C/ DV-MS-0010	10/31/2021	3, 40 mL VOA vials –Terra Core Plus 1, 40 mL unpreserved VOA vial to be submitted for moisture content	DI water/frozen or Methanol; Cool ≤ 6 °C	14 days (if samples are preserved/frozen within 48 hours of sampling)		21 days
PAHs	SO	8270D-SIM/ DV-MS-0002	10/31/2021	1, 8 oz, wide-mouth jar	Cool ≤ 6°C	14 days	40 days	21 calendar days
Picric Acid	SO (discrete)	8330A/ DV-LC-0002	10/31/2021	1, 8 oz, wide-mouth jar (8330A)	Cool ≤ 6°C	14 days	40 days	21 calendar days

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
Metals	SO	6010C/ DV-MT-0021	10/31/2021	1, 4 oz, glass jar	Cool ≤ 6°C	180 days		21 calendar days
Metals	SO	6020A/ DV-MT-0022	10/31/2021	1, 4 oz, glass jar	Cool ≤ 6°C	180 days		21 calendar days
Mercury	SO	7471B/ DV-MT-0016	10/31/2021	1,4 oz, glass jar	Cool ≤ 6°C	28 days		21 calendar days
PCBs	SO	8082A/DV-GC-0021	10/31/2021	1, 8 oz, glass jar	Cool ≤ 6°C	1 year	40 days	21 calendar days
TOC	SO	9060A/ DV-WC-0048	10/31/2021	1, 4oz glass jar	Cool ≤ 6°C	28 days		21 calendar days
Picric Acid	ISM SO	8330A/ DV-LC-0002	10/31/2021	1 gallon Ziploc bag (please double-bag)	Cool ≤ 6°C	14 days	40 days	21 calendar days
Picric Acid	IDW-S	8330A/ DV-LC-0002	10/31/2021	1, 8 oz, wide-mouth jar (8330A)	Cool ≤ 6°C	14 days	40 days	21 calendar days
PCBs	IDW-S	8082A/DV-GC-0021	10/31/2021	1, 8 oz, glass jar	Cool ≤ 6°C	1 year	40 days	21 calendar days
Reactivity – Cyanide	IDW-S	9012/ DV-WC-0083	10/31/2021	1, 4 oz, glass jar	Cool ≤ 6°C	14 days		21 calendar days
Reactivity – Sulfide	IDW-S	9034/DV-WC-0091	10/31/2021	1, 4 oz, glass jar	Cool ≤ 6 °C	7 days		21 calendar days
Toxicity Characteristic Leaching Procedure (TCLP) Metals	IDW-S	1311/ DV-IP-0012	10/31/2021	1, 32oz glass jar	Cool 4 ± 2°C; Preservation not added until after leaching	180 days		21 calendar days

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
TCLP VOCs	IDW-S	1311/ DV-IP-0012	10/31/2021	1, 4oz jar -Teflon lined lids – no headspace	Cool 4 ± 2°C; Preservation not added until after leaching	14 days		21 calendar days
TCLP SVOCs	IDW-S	1311/ DV-IP-0012	10/31/2021	1, 32oz glass jar	Cool 4 ± 2°C; Preservation not added until after leaching	14 days		21 calendar days
pH	IDW-S	9045/ DV-WC-0001	10/31/2021	1, 4 oz, glass jar	Cool ≤ 6°C	28 days		21 calendar days
VOCs	GW	8260C/ DV-MS-0010	10/31/2021	3, 40 mL glass VOA Vials	Cool ≤ 6 °C; adjust pH < 2; HCl	14 days - preserved 7 days - unpreserved		21 calendar days
PAHs	GW	8270D-SIM/ DV-MS-0002	10/31/2021	2, 1 liter, amber	Cool ≤ 6°C	7 days	40 days	21 calendar days
Picric Acid	GW	8330B/ DV-LC-0002	10/31/2021	2, 500 mL, amber	Cool ≤ 6°C	7 days	40 days	21 calendar days
Metals	GW	6010C/ DV-MT-0021	10/31/2021	1, 250 mL, HDPE	HNO3, pH < 2	180 days		21 calendar days
Metals	GW	6020A/ DV-MT-0022	10/31/2021	1, 250 mL, HDPE	HNO3, pH < 2	180 days		21 calendar days
Mercury	GW	7470A/DV-MT-0017	10/31/2021	1, 250 mL, HDPE	HNO3, pH < 2	28 days		21 calendar days

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
PCBs	GW	8082A/DV-GC-0021	10/31/2021	1, 1000 mL, amber glass jar	Cool ≤ 6°C	1 year	40 days	21 calendar days
pH	IDW-AQ	9040/ DV-WC-0031	10/31/2021	1, 4oz, glass or plastic jar	None	None		21 calendar days
Flashpoint	IDW-AQ	1010/ DV-WC-0075	10/31/2021	1, 500 mL, amber	Cool 0 to 6°C	No requirement		21 calendar days
Picric Acid	IDW-AQ	8330B/ DV-LC-0002	10/31/2021	2, 500 mL, amber	Cool ≤ 6°C	7 days	40 days	21 calendar days
Reactivity – Cyanide	IDW-AQ	9012/ DV-WC-0083	10/31/2021	1, 500 mL, HDPE	NaOH, pH > 12; Cool ≤ 6 °C	14 days		21 calendar days
Reactivity – Sulfide	IDW-AQ	9034/DV-WC-0091	10/31/2021	1, 250 mL HDPE	NaOH/Zn Acetate pH>9 Cool ≤ 6 °C	7 days		21 calendar days

Notes:
[1]GW = groundwater, IDW = investigative derived waste (solid[S]/aqueous[AQ]), ISM = incremental sampling methodology, and SO = soil
[2]All laboratory SOPs are presented in **Appendix C**.
[3]Containers may be combined for analyses of the same sample. The laboratories will coordinate containers which can be combined.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Laboratory: Eurofins TestAmerica, Corpus Christi

Ship samples to:

Eurofins TestAmerica, Denver
Attn: Sample Receiving
4955 Yarrow Street
Arvada, CO 80002-4517

POC: Patrick McEntee, Patrick.McEntee@testamericainc.com, 303-736-0107

List of Required Accreditations/Certifications: N/A

Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
CEC	SO	9081/6010B CC-ATM-M020/CC-ATM-M005	N/A	1, 1000 mL HDPE or glass	None	6 months		21 calendar days

Notes:
[1] SO = soil.
[2]All laboratory SOPs are presented in **Appendix D**.
[3]Containers may be combined for analyses of the same sample. The laboratories will coordinate containers which can be combined.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Laboratory: Eurofins TestAmerica, Savannah

Ship samples to:

Eurofins TestAmerica, Denver
Attn: Sample Receiving
4955 Yarrow Street
Arvada, CO 80002-4517

POC: Patrick McEntee, Patrick.McEntee@testamericainc.com, 303-736-0107

List of Required Accreditations/Certifications: DoD ELAP, version 5.3 (see **Appendix C**)

Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
SVOCs (TCL, DNCB, and 2,4-DNP)	SO	8270D/ SA-SM-033	9/22/2022	8 oz, glass jar	Cool 0 to 6°C	14 days	40 days	21 calendar days
SVOCs (TCL, DNCB, and 2,4-DNP)	ISM SO	8270D/ SA-SM-033	9/22/2022	1 gallon Ziploc bag (please double-bag)	Cool 0 to 6°C	14 days	40 days	21 calendar days
Ignitability	IDW-S	1030/ SA-GE-140	9/22/2022	8 oz, glass	Cool 0 to 6°C	None		21 calendar days

Notes:
[1]GW = groundwater, IDW = investigative derived waste (solid[S]/aqueous[AQ]), ISM = incremental sampling methodology, and SO = soil
[2]All laboratory SOPs are presented in **Appendix D**.
[3]Containers may be combined for analyses of the same sample. The laboratories will coordinate containers which can be combined.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Laboratory: Eurofins TestAmerica, Savannah
Ship samples to:

Eurofins TestAmerica Savannah
Attention Sample Control
5102 LaRoche Avenue
Savannah, GA 31404-6019
Main Phone: 912-354-7858

POC: Patrick McEntee, Patrick.McEntee@testamericainc.com, 303-736-0107

List of Required Accreditations/Certifications: DoD ELAP, version 5.3 (see **Appendix C**)

Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
SVOCs (TCL, DNCB, and 2,4-DNP)	GW	8270D/ SA-SM-033	9/22/2022	2, 1 liter, amber, glass	Cool 0 to 6°C	7 days	40 days	21 calendar days

Notes:
[1]GW = groundwater, IDW = investigative derived waste (solid[S]/aqueous[AQ]), and SO = soil
[2]All laboratory SOPs are presented in **Appendix D**.
[3]Containers may be combined for analyses of the same sample. The laboratories will coordinate containers which can be combined.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Laboratory: Eurofins TestAmerica, Seattle

Ship samples to:

Eurofins TestAmerica, Denver
Attn: Sample Receiving
4955 Yarrow Street
Arvada, CO 80002-4517

POC: Patrick McEntee, Patrick.McEntee@testamericainc.com, 303-736-0107

List of Required Accreditations/Certifications: DoD ELAP, version 5.1.1 (see **Appendix C**)

Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix[1]	Method/ SOP[2]	Accreditation Expiration Date	Container(s) (number, size, and type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
Grain Size	SO	ASTM D422/ TA-WC-0183	N/A	1, 16oz glass jar	Cool 0 to 6°C	6 months		21 calendar days

Notes:
[1]SO = soil
[2]All laboratory SOPs are presented in **Appendix D**.
[3]Containers may be combined for analyses of the same sample. The laboratories will coordinate containers which can be combined.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Continued

Laboratory: MicroVision Labs, Inc.

Ship samples to:

MicroVision Labs, Inc.
Attn: Sample Receiving
187 Billerica Road
Chelmsford, MA 01824

POC: John Knowles, 978-315-5768

List of Required Accreditations/Certifications: ISO/IEC 17025:2017 (see **Appendix C**)

Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix[1]	Method/ SOP	Accreditation Expiration Date	Container(s) (number, size, and type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
Coal Ash	SO	Proprietary	8/31/2021	16 oz, glass jar	NA	NA	NA	21 calendar days

Notes:
[1]SO= soil

QAPP Worksheet #20: Field Quality Control Summary
(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

Matrix	Analyte/ Analyte Group	Field Samples	Field Duplicate	Matrix Spike	Matrix Spike Duplicate or Matrix Duplicate	Equipment Rinsate Blanks	Trip Blank		Total # of Analyses
Discrete Surface Soil	TOC	69	7	4	4	0	0		84
	Grain Size	69	7	0	0	0	0		76
	CEC	69	7	0	0	0	0		76
	Select SVOCs (DNCB and 2,4-DNP)	54	6	3	3	1 per day of sampling	0		66+ Blanks
	Select Explosives (Picric Acid)	54	6	3	3	1 per day of sampling	0		66+ Blanks
	TAL Metals	108	11	6	6	1 per day of sampling	0		131+ Blanks
	Target Compound List (TCL) SVOCs	31	4	2	2	1 per day of sampling	0		39+ Blanks
	PAHs	86	9	5	5	1 per day of sampling	0		105+ Blanks
	PCBs	22	3	2	2	1 per day of sampling	0		29+ Blanks
	Coal Ash	10	1	0	0	0	0	0	11
ISM Surface Soil	Select SVOCs (DNCB and 2,4-DNP)	36	0	4	0	1 per day of sampling	0		40+ Blanks
	Select Explosives (Picric Acid)	36	0	4	0	1 per day of sampling	0		40+ Blanks

QAPP Worksheet #20: Field Quality Control Summary, Continued

Matrix	Analyte/ Analyte Group	Field Samples	Field Duplicate	Matrix Spike	Matrix Spike Duplicate or Matrix Duplicate	Equipment Rinsate Blanks	Trip Blank		Total # of Analyses
Subsurface Soil	TOC	130	13	7	7	0	0		157
	Grain Size	130	13	0	0	0	0		143
	CEC	130	13	0	0	0	0		143
	Select SVOCs (DNCB and 2,4-DNP)	161	17	9	9	1 per day of sampling	0		196+ Blanks
	Select Explosives (Picric Acid)	161	17	9	9	1 per day of sampling	0		196+ Blanks
	TAL Metals	200	20	10	10	1 per day of sampling	0		240+ Blanks
	TCL VOCs	28	3	2	2	1 per day of sampling	1 per cooler of samples		35+ Blanks
	TCL SVOCs	66	7	4	4	1 per day of sampling	0		81+ Blanks
	PAHs	156	16	8	8	1 per day of sampling	0		188+ Blanks
	PCBs	12	3	2	2	1 per day of sampling	0		29+ Blanks
Groundwater (DPT samples)	Select SVOCs (DNCB and 2,4-DNP)	43	5	3	3	1 per day of sampling	0		54+ Blanks
	Select Explosives (Picric Acid)	43	5	3	3	1 per day of sampling	0		54+ Blanks

QAPP Worksheet #20: Field Quality Control Summary, Continued

Matrix	Analyte/ Analyte Group	Field Samples	Field Duplicate	Matrix Spike	Matrix Spike Duplicate or Matrix Duplicate	Equipment Rinsate Blanks	Trip Blank		Total # of Analyses
	TAL Metals–Dissolved	48	5	3	3	1 per day of sampling	0		59+ Blanks
	TCL VOCs	10	1	1	1	1 per day of sampling	1 per cooler of samples		13+ Blanks
	TCL SVOCs	19	2	1	1	1 per day of sampling	0		23+ Blanks
	PAHs	28	3	2	2	1 per day of sampling	0		35+ Blanks
	PCBs	9	1	1	1	1 per day of sampling	0		12+ Blanks

Notes:
All solid samples to be analyzed for % moisture.
If a sample is being analyzed for VOCs only, a separate vial without water or methanol preservatives must be submitted for moisture analysis.
Equipment Blanks = 1 per day of sampling for reused equipment only.
IDW samples not included (up to seven aqueous and four solid, i.e., one per medium per AOC and background area).

QAPP Worksheet #21: Field SOPs
(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

Field SOPs are located in **Appendix B:**

SOP # or Reference	Title, Revision, Date, and URL (if available)	Originating Organization	SOP option or Equipment Type (if SOP provides different options)	Modified For Project? Y/N	Comments
01	Abandonment of Monitoring Wells and Piezometers	Bluestone	N/A	N	
02	Equipment Calibration and Maintenance	Bluestone	N/A	N	
03	Design and Installation of Monitoring Wells and Well Development	Bluestone	N/A	N	
04	Global Positioning Survey (GPS)	Bluestone	N/A	N	
05	Decontamination of Field Equipment	Bluestone	N/A	N	
06	Field Documentation	Bluestone	N/A	N	
07	Field Screening Soils Using a Photo-ionization detector (PID)	Bluestone	PID	N	
08	Electromagnetic (EM) 31 Geophysical Survey	Bluestone	GPR	N	
09	Ground-Penetrating Radar (GPR) Geophysical Survey	Bluestone	N/A	N	
10	Storage and Sampling of Investigation-Derived Wastes (IDW)	Bluestone	N/A	N	
11	Geologic Logging	Bluestone		N	
12	Low-Flow Groundwater Sampling and Sampling with a Bailer	Bluestone	Bailer, Bladder, Peristaltic Pump	N	
13	Sample Packaging and Shipping	Bluestone	N/A	N	
15	Subsurface Soil Sampling	Bluestone	N/A	N	
16	Surface Soil Sampling	Bluestone	MacroCore sleeves or disposable spoons	N	
18	Utility Clearance	Bluestone	N/A	N	

QAPP Worksheet #21: Field SOPs, Continued

SOP # or Reference	Title, Revision, Date, and URL (if available)	Originating Organization	SOP option or Equipment Type (if SOP provides different options)	Modified For Project? Y/N	Comments
19	Groundwater Elevation Monitoring	Bluestone	Electronic Water Level Indicator	N	
21	Incremental Sampling Methodology	Bluestone	N/A	N	
3.0	U.S. Environmental Protection Agency: Samplers Guide (2014)	EPA	N/A	N	Incorporated by reference. Not included in Appendix B.
Method D2488-17e1	Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)	ASTM	N/A	N	
Method D2487-17	Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)	ASTM	N/A	N	

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
MiniRAE® plus Classic (PGM-76) Toxic Gas Monitor - 11.7 electron volt (eV) lamp MultiRAE® plus PID Toxic Gas Monitor - 11.7 eV lamp	Calibration performed at the start of each workday and checked at the end of each day. Maintain as needed in field; factory-calibrated or replaced by supplier as needed.	Manufacturer specifications	FTL	Calibrate am, check pm	± 10% of the calibrated value	Manually zero the meter by performing a 'fresh air' calibration; recalibrate with gas (isobutylene); return/replace the meter for service as necessary
YSI-Pro Plus digital sampling system with flow-through cell	Calibration performed at the start of each workday and checked at the end of each workday. Meter must be factory-calibrated and serviced prior to rental delivery, and again as needed. Replacement probes and calibration fluids must be provided by the vendor.	Manufacturer specifications	FTL	Calibrate am Check pm	pH: ± 0.05 Specific Conductivity: ±5 micro Siemens (µS) DO ± 0.02 parts per million (ppm) Temp.: ± 0.30C	Recalibrate or service as necessary
Solinst® 101 P7 Water Level Meter (or similar)	None		FTL	Check meter battery daily before each use		Return to rental company for replacement

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection, Continued

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
EOS Arrow 100	Pre-field data set-up; post field data differential correction Maintenance: Charge batteries daily before use; keep unit away from extreme heat or cold. Keep unit clean. If not able to calibrate see service manual.	Manufacturer specifications	FTL; Environmental Sampler	Daily, before each use; at all sampling points for maintenance	Manufacturers specifications	Manufacturers specifications
Natural Gamma (Downhole Geophysics) ACTive real-time downhole CT unit (or similar)	Calibrated by manufacturer		Subcontractor personnel	As needed	0-100 kilo counts per second	Recalibrate or service as necessary

QAPP Worksheet #23: Analytical SOPs
(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

Analytical SOPs are located in **Appendix D:**

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
CC-ATM-M005	Trace Metals Analysis by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) EPA SW-846 Methods 6010B and 200.7	Definitive	Solid/Metals	ICP-AES	N
CC-ATM-M020	Cation-Exchange Capacity (CEC) of Soils SW-846 Method 9081 and LDNR Method 29-B	Definitive	Solid/Metals	N/A	N
CC-GLO-008	Receipt, Log-in, and Storage of Environmental Samples	N/A	Sample Handling	N/A	N
CC-GLO-WI002	Test America Corpus Christi Sample Acceptance Policy	N/A	Sample Handling	N/A	N
DV-GC-0021	PCBs by GC/ Electron Capture Detector (ECD) [SW846 Methods 8082 and 8082A]	Definitive	Solid & Water/PCBs	GC/ECD	N
DV-HS-0003	Characterization of Waste	N/A	Sample Handling	N/A	N
DV-HS-0005	Excess Sample Material Management	N/A	Sample Handling	N/A	N
DV-IP-0010	Acid Digestion of Aqueous Samples for Metals Analysis by Inductively Coupled Plasma (ICP)	Preparation	Water/Metals	N/A	N
DV-IP-0012	TCLP and Synthetic Precipitation Leaching Procedure (SPLP) [SW846 1311 and 1312]	Preparation	Solid/Organics & Metals	N/A	N
DV-IP-0014	Acid Digestion of Aqueous Samples for Analysis by ICP-MS (SW-846 3005A, 3020A, and EPA 200.8)	Preparation	Water/Metals	N/A	N
DV-IP-0015	Acid Digestion of Solids (EPA 3050B)	Preparation	Solid/Metals	N/A	N

QAPP Worksheet #23: Analytical SOPs, Continued

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
DV-LC-0002	Nitroaromatic and Nitroamine Explosive Compounds by High Performance Liquid Chromatography (HPLC) (SW846 8330A & 8330B)	Definitive	Solid & Water /Explosives	HPLC	N
DV-MS-0002	Polynuclear Aromatic Hydrocarbons by Gas Chromatograph/Mass Spectroscopy (GC/MS) Selected Ion Monitoring (SIM) [SW 846 Method 8270C and 8270D]	Definitive	Solid & Water/PAHs	GC/MS	N
DV-MS-0010	Determination of Volatile Organics by GC/MS (SW846 8260C and EPA 624)	Definitive	Solid & Water/Volatiles	GC/MS	N
DV-MT-0016	Mercury in Solids by Cold Vapor Atomic Absorption (CVAA)	Definitive	Solid/Mercury	CVAA	N
DV-MT-0017	Mercury in Water by CVAA [SW 7470A]	Definitive	Water/Mercury	CVAA	N
DV-MT-0021	ICP Analysis for Trace Elements by SW-846 Method 6010C	Definitive	Solid & Water/Metals	ICP	N
DV-MT-0022	Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A	Definitive	Solid & Water/Metals	ICP-MS	N
DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series	Preparation	Water/Organics	N/A	N
DV-OP-0007	Concentration and Clean-up of Organic Extracts (SW-846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 series)	Preparation	Solid & Water/Organics	N/A	N
DV-OP-0013	Multi-incremental Sub-sampling from Soils and Sediments (ASTM D 6323)	Preparation	Solid/Organics and Metals	N/A	N
DV-OP-0015	Microwave Extraction of Solid Samples [SW-846 3546]	Preparation	Solid/Organics	N/A	N

QAPP Worksheet #23: Analytical SOPs, Continued

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
DV-OP-0017	Solid Phase Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Water Samples (SW-846 3535A)	Preparation	Water/Explosives	N/A	N
DV-OP-0018	Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Soil Samples (SW-846 8330A & 8330B)	Preparation	Solid/Explosives	N/A	N
DV-QA-0003	Sample Management and Chain of Custody	N/A	Sample Handling	N/A	N
DV-QA-0014	Selecting and Using Balances	N/A	Maintenance	Balances	N
DV-QA-0036	Sub-out Work Sample Management and Chain of Custody	N/A	Sample Handling	N/A	N
DV-WC-0001	Soil and Waste pH [SW 9045C & SW 9045D]	Definitive	Solid/pH	Probe & Meter	N
DV-WC-0023	Percent Moisture in Soils and Wastes [ASTM D2216, CLP ILM05.3]	Definitive	Solid/Percent Moisture	N/A	N
DV-WC-0031	Manual pH [SM 4500-H+ B, SW 9040B & SW 9040C}	Definitive	Water/pH	Probe & Meter	N
DV-WC-0048	Carbon in Soil (TOC, TC, TIC) [SW846 9060, 9060A)	Definitive	Solid/TOC	TOC Instruments	N
DV-WC-0075	Flash Point by Automatic Pensky-Martens Closed Cup Apparatus [SW 1010A, ASTM D93]	Definitive	Water/Flash point	Pensky-Martens Closed Cup Apparatus	N
DV-WC-0083	Total and Amenable Cyanide by SM 4500-CN B, 4500-CN C, 4500-CN E, 4500-CN G, SW-846 9012A, 9012B, and Weak Acid Dissociable Cyanide by 4500-Cn I	Definitive	Solid & Water/Cyanide	Colorimetric	N
DV-WC-0091	Acid-Soluble and Acid-Insoluble Sulfides: Distillation and Titration [SW 9030B/SW 9034]	Definitive	Solid & Water/Sulfide	Distillation/ Titration	N

QAPP Worksheet #23: Analytical SOPs, Continued

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
SA-CU-001	Sample Receipt	N/A	Sample Handling	N/A	N
SA-EX-030	Liquid Extraction Procedure: Continuous Liquid-Liquid (Methods: EPA 3520C, EPA 3520C_LVI, and EPA 600-series)	Preparation	Water/Organics	N/A	N
SA-EX-040	Microwave and Sonication Soil Extraction Procedures (Methods: EPA 3546 and EPA 3550C)	Preparation	Solid/Organics	N/A	N
SA-GE-140	Ignitability	Definitive	Solid/Ignitability	N/A	N
SA-SM-033	Semi-Volatile Compounds by GC/MS (Methods: EPA 625, EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270D_LL, EPA 8270E_LL, EPA 8270D_LL_PAH, and EPA 8270E_LL_PAH)	Definitive	Solid & Water/Semi-Volatiles	GC/MS	N
Savannah – EHS Manual	Addendum to the Environmental Health and Safety Manual	N/A	Sample Handling	N/A	N
TA-EHS-0036	Laboratory Waste Management and Disposal	N/A	Sample Handling	N/A	N
TA-QA-0001	Sample Receipt and Log-in	N/A	Sample Handling	N/A	N
TA-QA-0002	Chain of Custody, Internal Sample Transfer, Storage, and Security	N/A	Sample Handling	N/A	N
TA-WC-0183	Particle Size Analysis of Soils [Methods ASTM D422-63, D7928, D6913]	Definitive	Solid/Grain Size	N/A	N

QAPP Worksheet #24: Analytical Instrument Calibration
(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

QAPP Worksheet #24-1: Analytical Instrument Calibration – ICP/AES for Metals (6010C)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
ICP/AES Metals (6010C)	Linear Dynamic Range (LDR) or High-level Check Standard		At initial set-up and checked every six months with a high standard at the upper limit of the range.	Within ±10% of true value.	Dilute samples within the calibration range or re-establish/verify the LDR.	Analyst/ Section Supervisor	DV-MT-0021
	Initial Calibration (ICAL) for all analytes	Minimum one high standard and a calibration blank.	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$	Correct problem, then repeat ICAL.		
	Initial Calibration Verification (ICV)		Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ±10% of true value.	Correct problem. Rerun ICV. If that fails repeat ICAL. No samples shall be analyzed until the second-source calibration verification is successful.		
	Initial and Continuing		Immediately after the ICV and	Absolute values of all analytes must be < ½	Correct any problems and repeat ICV/ICB		

QAPP Worksheet #24-1: Analytical Instrument Calibration – ICP/AES for Metals (6010C), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
	Calibration Blank (ICB/CCB)		immediately after every Continuing Calibration Verification (CCV)	LOQ or <1/10 the amount measured in any sample. Non-detects associated with positive blank infractions may be reported. Sample results >10x LOQ associated with negative blanks may be reported.	analysis. If that fails rerun ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCVs. CCB failures due to carryover may not require an ICAL.		
	CCV		After every 10 field samples and at end of analysis sequence.	All reported analytes within ±10% of true value.	Recalibrate and reanalyze all affected samples since the last acceptable CCV. or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If		

QAPP Worksheet #24-1: Analytical Instrument Calibration – ICP/AES for Metals (6010C), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
					either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		
	Low-level Calibration Check Standard (LLCCV)	LLCCV ≤LOQ	Daily at the beginning of the run.	All reported analytes within ±20% of true value.	Correct problem and repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.		
	Interference Check Solutions (ICS)		After ICAL and prior to sample analysis.	ICS-A: absolute value of concentration for all non-spiked analytes < ½ LOQ (except verified trace impurities from one of the spiked analytes) ICS-AB: recovery within ±20 % true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze affected samples. If corrective action fails, apply Q-flag to all results for specific analytes in all samples associated with the failed ICS.		

QAPP Worksheet #24-2: Analytical Instrument Calibration – ICP/MS for Metals (6020A)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
ICP/MS Metals (6020A)	LDR or High-level Check Standard		At initial set-up and checked every six months with a high standard at the upper limit of the range.	Within ±10% of true value.	Dilute samples within the calibration range or re-establish/verify the LDR.	Analyst/ Section Supervisor	DV-MT-0022
	Tuning		Prior to ICAL	Mass calibration ≤ 0.1 atomic mass unit (amu) from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify. No samples shall be analyzed without a valid tune.		
	ICAL for all analytes	Minimum one high standard and a calibration blank.	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$	Correct problem, then repeat ICAL.		
	ICV		Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ±10% of true value.	Correct problem. Rerun ICV. If that fails repeat ICAL. No samples shall be analyzed until the second-source		

QAPP Worksheet #24-2: Analytical Instrument Calibration – ICP/MS for Metals (6020A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
					calibration verification is successful.		
	Initial and Continuing Calibration Blank (ICB/CCB)		Immediately after the ICV and immediately after every CCV	Absolute values of all analytes must be < ½ LOQ or <1/10 the amount measured in any sample. Non-detects associated with positive blank infractions may be reported. Sample results >10x LOQ associated with negative blanks may be reported.	Correct any problems and repeat ICV/ICB analysis. If that fails rerun ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCVs. CCB failures due to carryover may not require an ICAL.		
	CCV		After every 10 field samples and at end of analysis sequence.	All reported analytes within ±10% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take		

QAPP Worksheet #24-2: Analytical Instrument Calibration – ICP/MS for Metals (6020A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
					corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Or Recalibrate and reanalyze all affected samples since the last acceptable CCV		
	LLCCV	LLCCV ≤LOQ	Daily at the beginning of the run.	All reported analytes within ±20% of true value.	Correct problem and repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.		
	ICS		After ICAL and prior to sample analysis.	ICS-A: absolute value of concentration for all non-spiked analytes < ½ LOQ (except verified trace impurities from one of the spiked analytes)	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze affected samples.		

QAPP Worksheet #24-2: Analytical Instrument Calibration – ICP/MS for Metals (6020A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
				ICS-AB: recovery within ±20 % true value.			

QAPP Worksheet #24-3: Analytical Instrument Calibration – CVAA for Mercury (7470A and 7471B)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/Position Responsible for Corrective Action	SOP Reference
CVAA Spectro-photometer; Mercury	ICAL	Minimum five standards and calibration blank	Daily initial calibration prior to sample analysis.	$r^2 \geq 0.99$	Correct problem then repeat initial calibration.	Analyst	DV-MT-0016 and DV-MT-0017
	ICB/CCB		Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).		
	ICV		Run second-source standard once after each ICAL and prior to sample analysis.	Analytes within +10% of expected value	Correct problem. Rerun ICV. If that fails, rerun ICAL.		

QAPP Worksheet #24-3: Analytical Instrument Calibration – CVAA for Mercury (7470A and 7471B), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/Position Responsible for Corrective Action	SOP Reference
	LLCCV	LLCCV ≤LOQ	Daily at the beginning of the run.	All reported analytes within ±20% of true value.	Correct problem and repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.		
	CCV		After every 10 field samples, and at the end of the analysis sequence	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.		

QAPP Worksheet #24-4: Analytical Instrument Calibration – GC/ECD for PCBs (8082A)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/Position Responsible for Corrective Action	SOP Reference
Gas Chromatograph/Electron Capture Detector (GC/ECD); PCBs	ICAL	Minimum five-point calibration for all analytes for linear and six levels for quadratic.	Initial calibration prior to sample analysis and after ICV or CCV failure.	Acceptance Criteria options: 1. Relative standard deviation (RSD) for each analyte ≤ 20%. 2. Linear least squares regression: $r^2 \geq 0.99$ 3. Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (quadratic).	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration Quantitation for multicomponent analytes, such as Aroclors, must be performed using a 5-point calibration.	Analyst	DV-GC-0021
	Establish retention time (RT) window positions		Once per ICAL and at the beginning of the analytical sequence, for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA		

QAPP Worksheet #24-4: Analytical Instrument Calibration – GC/ECD for PCBs (8082A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/Position Responsible for Corrective Action	SOP Reference
	Retention Time Window Width		Perform 72-hour study at method set-up and after major maintenance (e.g., column change) to calculate the RT window width for each analyte and surrogate.	RT width is ± 3 times the standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA		
	ICV		Second source standard, immediately following ICAL	CCV	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.		
			Before sample analysis, after every 10 field	All reported analytes and surrogates	Immediately analyze two additional consecutive CCVs. If		

QAPP Worksheet #24-4: Analytical Instrument Calibration – GC/ECD for PCBs (8082A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/Position Responsible for Corrective Action	SOP Reference
			samples, and at the end of the analysis sequence with the exception of CCVs for Pesticide multicomponent analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	within established RT windows. All project analytes within ± 20% of the expected value from the ICAL	both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze once all affected samples since the last acceptable CCV. If CCV still fails, consult client before reporting. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.		

QAPP Worksheet #24-5: Analytical Instrument Calibration – GC/MS for VOCs (8260C)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS (8260C)	Tune Check		Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Analyst/ Section Supervisor	DV-MS-0010
	ICAL for all analytes (including surrogates)	Minimum five-point initial calibration for linear or six-point for quadratic; lowest concentration standard at or below the reporting limit.	At instrument set-up, prior to sample analysis and when CCV fails.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤15% Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.		

QAPP Worksheet #24-5: Analytical Instrument Calibration – GC/MS for VOCs (8260C), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
	RT window position establishment		Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA		
	Evaluation of Relative Retention Times (RRT)		With each sample.	RRT of each reported analyte within ±0.06 RRT units.	Correct problem, then rerun ICAL.		
	ICV		Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes and surrogates within ±20% of the true value.	Correct problem. Rerun ICV, if that fails repeat ICAL		
	CCV		Daily before sample analysis; after every 12 hours of analysis time; at the end of the analytical batch	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ±	recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass,		

QAPP Worksheet #24-5: Analytical Instrument Calibration – GC/MS for VOCs (8260C), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
				50% for end of analytical batch CCV.	samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

QAPP Worksheet #24-6: Analytical Instrument Calibration – GC/MS for SVOCs (8270D)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS (8270D)	Tune Check		Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of decafluorotriphenylphos-phine (DFTPP) from method.	Retune instrument and verify.	Analyst/ Section Supervisor	SA-SM-033
	Performance Check		At the beginning of each 12-hour period, prior to sample analysis.	Degradation ≤20% for DDT. Benzidine and Pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat the performance checks.		
	ICAL (for all analytes and surrogates)	Minimum five levels for linear and six levels for quadratic. Lowest concentration standard at or near the reporting limit.	At instrument set-up, prior to sample analysis and after ICV or CCV failure	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.		

QAPP Worksheet #24-6: Analytical Instrument Calibration – GC/MS for SVOCs (8270D), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
				(quadratic) for each analyte: $r^2 \geq 0.99$.			
	RT window position establishment		Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA		
	Evaluation of RRT		With each sample.	RRT of each reported analyte within ±0.06 RRT units.	Correct problem, then rerun ICAL.		
	ICV		Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun verification. If still fails, repeat initial calibration.		
	CCV		Daily before sample analysis; after every 12 hours of analysis	All reported analytes and surrogates within ± 20% of true value All reported analytes	Recalibrate, and reanalyze all affected samples since the last acceptable CCV;		

QAPP Worksheet #24-6: Analytical Instrument Calibration – GC/MS for SVOCs (8270D), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
			time; and at the end of the analytical batch run.	and surrogates within ± 50% for end of analytical batch CCV.	or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

QAPP Worksheet #24-7: Analytical Instrument Calibration – GC/MS for PAHs (8270D-SIM)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS SIM Mode for PAHs (8270D-SIM)	Tune Check		Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method 8270D. Tune check may be acquired as a full scan.	Retune instrument and verify.	Analyst/Section Supervisor	DV-MS-0002
	Performance Check		At the beginning of each 12-hour period, prior to sample analysis.	Degradation ≤ 20% for DDT.	Correct problem, then repeat the performance checks.		
	ICAL	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit.	At instrument set-up, prior to sample analysis.	Each analyte must meet one of the options below: Option 1: RSD for each analyte ≤ 20%. If pentachlorophenol is a target analyte, an RSD of ≤ 40% allowed. Option 2: linear least	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.		

QAPP Worksheet #24-7: Analytical Instrument Calibration – GC/MS for PAHs (8270D-SIM), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
				squares regression for each analyte: $r^2 \geq 0.99$;			
	RT window position establishment		Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA		
	Evaluation of RRT		With each sample.	RRT of each reported analyte within ±0.06 RRT units of the mean RRT of the calibration standards. RRT may be updated based on the daily CCV.	Correct problem, then rerun ICAL.		
	ICV		Once after each ICAL, analysis of a second source standard	All reported analytes within ±20% of true value.	Correct problem and verify second source standard. Rerun		

QAPP Worksheet #24-7: Analytical Instrument Calibration – GC/MS for PAHs (8270D-SIM), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
			prior to sample analysis.		verification. If still fails, repeat initial calibration.		
	CCV	Concentration the same as the mid-point calibration standard (or lower).	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and		

QAPP Worksheet #24-7: Analytical Instrument Calibration – GC/MS for PAHs (8270D-SIM), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
					re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

QAPP Worksheet #24-8: Analytical Instrument Calibration – HPLC for Picric Acid (8330A)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
HPLC Picric Acid (8330A)	ICAL	Minimum five levels for linear and six levels for quadratic. No samples shall be analyzed until ICAL has passed.	At instrument set-up and after ICV or CCV failure, prior to sample analysis. Perform instrument re-calibration once per year minimum.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Analyst/Lab Manager	DV-LC-0002
	RT window position establishment		Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA		

QAPP Worksheet #24-8: Analytical Instrument Calibration – HPLC for Picric Acid (8330A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
	RT window width		At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times the standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA		
	ICV		Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes ±15% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.		
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within ± 15% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV Or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and		

QAPP Worksheet #24-8: Analytical Instrument Calibration – HPLC for Picric Acid (8330A), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
					re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

QAPP Worksheet #24-9: Analytical Instrument Calibration – HPLC for Picric Acid (8330B)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
HPLC Picric Acid (8330B)	ICAL	Minimum five levels for linear and six levels for quadratic. No samples shall be analyzed until ICAL has passed.	At instrument set-up and after ICV or CCV failure, prior to sample analysis. Perform instrument re-calibration once per year minimum.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Analyst/Lab Manager	DV-LC-0002
	Retention RT window position establishment		Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA		

QAPP Worksheet #24-9: Analytical Instrument Calibration – HPLC for Picric Acid (8330B), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
	RT window width		At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times the standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA		
	ICV		Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes ±20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.		
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within ± 20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV Or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and		

QAPP Worksheet #24-9: Analytical Instrument Calibration – HPLC for Picric Acid (8330B), Continued

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
					re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

QAPP Worksheet #24-10: Analytical Instrument Calibration – TOC Analyzer for TOC (9060A)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
Shimadzu TOC-V TOC analyzer (9060A)	ICAL	Per manufacturer's instructions, with a 5-point standard curve with 40% carbon standard.	Annually, or as needed. based on the instrument performance and maintenance.	The absolute value of the correlation coefficient (r) must be 0.995 or greater.	Correct problem, then repeat ICAL.	Lab Manager/ Analyst	DV-WC-0048
	ICV	Per manufacturer's instructions, with a low and/or high standard	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Within ± 10% of true value.	If it is outside the acceptance limits, check the equipment and standards, correct any problems, and then recalibrate.		
	CCV	Per manufacturer's instructions, with a low and/or high standard	The calibration is checked at the beginning of an analytical sequence (ICV), after every ten samples (CCV), and at the end of the sequence (CCV)	Within ± 10% of true value.	If it is outside the acceptance limits, check the equipment and standards, correct any problems, recalibrate, and rerun all samples analyzed since the last successful CCV.		

QAPP Worksheet #24-10: Analytical Instrument Calibration – TOC Analyzer for TOC (9060A)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
			by measuring a CCV standard.				
	ICB/CCB		System cleanliness is checked at the beginning of an analytical sequence (ICB), after every ten samples (CCB), and at the end of the sequence (CCB) by analyzing a blank.	Absolute values of all analytes must be < LOQ or <1/10 the amount measured in any sample. Non-detects associated with positive blank infractions may be reported. Sample results >10x LOQ associated with negative blanks may be reported.	If the blank result is greater than the LOQ, check for carry-over from high level samples, clean the system, recalibrate, and rerun all samples analyzed since the last successful CCB.		

QAPP Worksheet #24-11: Analytical Instrument Calibration – Hydrometer for Grain Size (ASTM D422)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/Position Responsible for Corrective Action	SOP Reference
Hydrometer	Verification of hydrometer		Before each use	Reading of DI water must be 1.000 ± 0.0005	Select a different hydrometer if verification cannot be completed	Analyst	TA-WC-0183

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/ECD	Change septum, clean injection port, change or clip column, install new liner, replace column, filters and seals	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	Quality Assurance Manual – Section 20
GC/MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	Analyst	Quality Assurance Manual – Section 20
	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection, Continued

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS	Replace sleeve, septum, splitless disc, and syringe; clip guard column; clean sources; maintain vacuum pumps	Detector signals and chromato-gram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	SA-SM-033
HPLC	Replace columns, DAD flow cell windows and ball-valve cartridges as needed, clean/ change filters, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	Quality Assurance Manual – Section 20
ICP	Replace pump windings and gas tanks, check standard and sample flow	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Analyst	Quality Assurance Manual – Section 20

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection, Continued

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
ICP/MS	Replace disposables, clean/change nebulizer, torch, and cones	Tuning	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Recalibrate	Analyst	Quality Assurance Manual – Section 20
CVAA	Replace disposables, flush lines, check lamp current and gas flow	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	Quality Assurance Manual – Section 20
TOC Instruments	Replace scrubbers	Preventative maintenance	Preventative Maintenance	As needed	None	Replace scrubbers	Analyst	DV-WC-0048
Balances	Clean balance pan and check if level	Calibration check with Class "S" traceable weight check	Instrument performance and accuracy	Daily or when used	See SOP DV-QA-0014 – Section 5	Recalibrate, unplug balance and restart Field Service if inoperable or does not meet calibration criteria	Analyst	SOP DV-QA-0014

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal
(UFP-QAPP Manual Section 3.3)
(EPA 2106-G-05 Section 2.3.3)

Sampling Organization: Bluestone

Laboratory: Test America/Eurofins and MicroVision Laboratories, Inc.

Method of sample delivery (shipper/carrier): FedEx or UPS

Number of days from reporting until sample disposal: Minimum of 30 days following analysis. After 30 days, shorter of holding time and approval of Data Validation Report in FUDSChem.

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP reference
Sample labeling	FTL	Bluestone SOPNo. 06
Chain-of-custody form completion	FTL/Sample Manager	Bluestone SOP No. 06
Packaging	FTL/Sample Manager	Bluestone SOP No. 13
Shipping coordination	FTL/Sample Manager	Bluestone SOP No. 13

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action – ISM Processing

Matrix: Soil ISM Approach
Analytical Groups: SVOCs (DNCB and 2,4-DNP only) and Explosives
Analytical Method/SOP: Incremental Soil DV-OP-0013

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Sample Preparation Processing	Each ISM sample, PT, Laboratory Control Sample (LCS), and Blank. Entire sample must be dried, ground, and sieved unless otherwise specified. See note below. Note that field samples collected using ISM approach submitted for analysis by 8270D will have aliquots taken for DNCB and 2,4-DNP analysis & moisture content prior to drying. See table below.	Refer to method specific tables for blank and other QC acceptance criteria. Where method specific tables require the use of DoD/DOE QSM Appendix C limits, those limits are not applicable to analysis after preparation using Table B-23 and laboratories shall develop in-house acceptance criteria. Laboratories shall develop in-house surrogate acceptance criteria.	Refer to method specific tables for respective corrective actions.	Refer to method specific tables for respective corrective actions.	Per DoD QSM, version 5.3, Table B-23
Soil Drying Procedure	Each sample, LCS, and Method Blank	Laboratory must have a procedure to determine when the sample is dry to constant	Not Applicable (NA)	Analyst / Section Supervisor	Per DoD, QSM, version 5.3, Table B-3

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action – ISM Processing, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
	Note that field samples collected using ISM approach submitted for analysis by 8270D will have aliquots taken for DNCB and 2,4-DNP analysis & moisture content prior to drying. See table below.	mass. Entire sample must be air dried at room temperature.			
Soil Sieving Procedure	Each sample, LCS, and Method Blank Note that this procedure does not pertain to SVOCs (DNCB and 2,4-DNP).	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Collect and weigh any portion unable to pass through the sieve.	NA	Analyst / Section Supervisor	Per DoD, QSM, version 5.3, Table B-3
Soil Grinding Procedure	Initial demonstration at start up and any time major equipment is changed or when a reduction in the number or time of grinding cycles occurs. Each required sample, LCS, Blank, and Matrix Spike sample.	Initial demonstration of grinding equipment: The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA	Analyst / Section Supervisor	Per DoD, QSM, version 5.3, Table B-3

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action – ISM Processing, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
	Note that this procedure does not pertain to SVOCs (DNCB and 2,4-DNP).				
Subsampling	Each required ISM sample, LCS, blank and matrix spike sample. All ISM sample types must be incrementally subsampled after processing. Note that this procedure does not pertain to SVOCs (DNCB and 2,4-DNP).	Entire sample is mixed and spread out evenly on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth and breadth to obtain the appropriate subsample size.	NA	Analyst / Section Supervisor	Per DoD, QSM, version 5.3, Table B-3 and B-23
Grinding Blanks	One per batch of samples. The Grinding Blank must be processed after the LCS (if ground). Or After a client sample with known contamination. Or At the end of the batch.	No reported analytes must be detected > ½ LOQ	Blank results must be reported and the affected samples must be flagged accordingly if blank criteria are not met. If required, reprep and reanalyze Method Blank and all QC samples and field samples processed	Analyst / Section Supervisor	Per DoD, QSM, version 5.3, Table B-3

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action – ISM Processing, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
			with the contaminated blank. If any individual Grinding blank is found to exceed the acceptance criteria, apply B-flag to the samples following that blank.		
ISM Laboratory Replicates	At the subsampling step, performed on one ISM sample per batch. Cannot be performed on any sample identified as a blank (e.g., Field Blank, Method Blank, Grinding Blank) or other QC. Sample triplicates are taken from a sample expected to contain the highest levels of analytes of concern.	The Relative Standard Deviation (RSD) for all three results above the LOQ must not exceed 20%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken. If reported per the client, apply J-flag to all samples within that batch is acceptance criteria are not met and explain in the case narrative.	Analyst / Section Supervisor	Per DoD, QSM, version 5.3, Tables B-3 and B-23

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action – ISM Processing, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Additional QC elements such as surrogates, internal standards, matrix spikes, etc. to be performed at the rates and in manner specified by the incremental sample preparations and analytical methods.					

The field team will not be performing ISM subsampling. Subsampling, as well as grinding, will be done in the laboratory according to the following schedule:

Analyte Group	Method	Dry/Subsample?*	Grind?
DNCB and 2,4-DNP	8270D	N	N
Picric Acid	8330B	Y	Y
*Moisture content (ASTM D2216) must be determined for analyses not being dried/subsampled.			

QAPP Worksheet #28-2: Analytical Quality Control and Corrective Action – 6010C

Matrix: Solid and Aqueous
Analytical Group: Metals
Analytical Method/SOP Reference: EPA 6010C/SOP DV-MT-0021

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss relative percent difference (RPD) exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines (aqueous only).	Field personnel Data Validators	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater
Method Blank	1/ Preparatory Batch (20 samples)	No Target Compounds > ½ LOQ or greater than 1/10 the amount measured in any sample or 1/10 the	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Non-detects associated with positive blank infractions may be reported. Sample	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-2: Analytical Quality Control and Corrective Action – 6010C, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		regulatory limit (whichever is greater).	results >10X the LOQ associated with negative blanks may be reported.		
Laboratory Control Sample/Laboratory Control Spike Duplicate (LCS/LCSD)	1/Preparatory Batch (20 samples)	Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: between LCS and LCSD ≤20%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative	Analyst / Section Supervisor	Same as QC Acceptance Limits.

QAPP Worksheet #28-2: Analytical Quality Control and Corrective Action – 6010C, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
MS/MSD	1 per batch of 20 or fewer samples	Recovery: Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between MS and MSD ≤ 20%	If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error. For Sample/Matrix Duplicate criteria only apply to analytes whose concentration in the sample is greater than or equal to the Limit of Quantitation (LOQ).	Analyst / Section Supervisor	Same as QC Acceptance Limits.
Dilution test	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations >50 x LOQ. Use along with MS/MSD and post digestion spike data to confirm matrix effects	Five-fold dilution must agree within ± 10% of the original determination	If dilution test fails analyze post digestion spike.	Analyst / Section Supervisor	Same as QC Acceptance Limits.

QAPP Worksheet #28-2: Analytical Quality Control and Corrective Action – 6010C, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Post digestion spike addition	When MS/MSD fails and analyte concentration < 50 x LOQ	Recovery within 80-120% of expected results	For specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits.
Method of Standard Additions	When dilution test or PDS fails and if required by the project	NA	Document use of MSA in Case Narrative	Analyst / Section Supervisor	N/A

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-3: Analytical Quality Control and Corrective Action – 6020A

Matrix: Aqueous and Solid
Analytical Group: Metals
Analytical Method/SOP Reference: EPA 6020A/SOP DV-MT-0022

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines (aqueous only).	Field personnel Data Validators	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater
Method Blank	1/ Preparatory Batch (20 samples)	No Target Compounds > ½ LOQ or greater than 1/10 the amount measured in any sample or 1/10 the	Correct problem. If required, reprep and reanalyze MB and all QC samples and field	Analyst / Section Supervisor	Same as QC Acceptance Criteria

QAPP Worksheet #28-3: Analytical Quality Control and Corrective Action – 6020A, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		regulatory limit (whichever is greater).	samples processed with the contaminated blank.		
LCS/LCSD	1/Preparatory Batch (20 samples)	Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between MS and MSD ≤ 20%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative	Analyst / Section Supervisor	Same as QC Acceptance Limits
MS/MSD	1 pair/ Preparatory Batch (20 samples)	Recovery: Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between MS and MSD ≤ 20% For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.	If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-3: Analytical Quality Control and Corrective Action – 6020A, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Dilution test	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations >50 x LOQ. Use along with MS/MSD or PDS data to confirm matrix effects.	Five-fold dilution must agree within ± 10% of the original determination	If dilution test fails analyze post digestion spike.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Post digestion spike addition	When MS/MSD fails or analyte concentration of all samples < 50 x LOQ.	Recovery within 80-120% of expected results	For specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Method of Standard Additions	When dilution test or post digestion spike fails and if required by the project.	NA	Document use of MSA in the Case Narrative	Analyst / Section Supervisor	N/A
Internal Standards	Every field sample, standard and QC sample.	IS intensity in the samples within 30 to 120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5- fold dilutions until criteria is met. For failed QC samples,	Analyst	Same as QC Acceptance Limits

QAPP Worksheet #28-3: Analytical Quality Control and Corrective Action – 6020A, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
			correct problem, and rerun all associated failed field samples.		

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-4: Analytical Quality Control and Corrective Action – 7470A/7471B

Matrix: Groundwater (7470A) and soil (7471B)
Analytical Group: Mercury
Analytical Method/SOP Reference: EPA 7470A and 7471B/SOP DV-MT-0017 and DV-MT-0016

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines (aqueous only).	Field personnel Data Validators	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.
MB	1/Preparatory Batch (20 samples)	No Target Compounds > ½ LOQ or greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem then re-prep and analyze method blank and all QC and field samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative.	Analyst	Same as QC Acceptance Limits

QAPP Worksheet #28-4: Analytical Quality Control and Corrective Action – 7470A/7471B, Continued

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
LCS	1/Preparatory Batch (20 samples)	%R aqueous = 82-119 %R solid = 80-124	Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.	Analyst	Same as QC Acceptance Limits
MSD	1 per 20 samples	RPD ≤20%	Examine project DQOs with PM. Evaluate data to determine source of difference between results.	Analyst	Same as QC Acceptance Limits
MS	1/Preparatory Batch (20 samples)	%R aqueous = 82-119 %R solid = 80-124	If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken.	Analyst	Same as QC Acceptance Limits

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-5: Analytical Quality Control and Corrective Action – 8082A

Matrix: Soil and Aqueous QC
Analytical Group: PCBs
Analytical Method/SOP Reference: EPA 8082A/SOP DV-GC-0021

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidance (aqueous only).	Field personnel Data Validators	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.
MB	1/Batch (20 samples)	No Target Compounds> ½ LOQ or > 1/10 the amount in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst	Same as QC Acceptance Limits
LCS	1/Batch (20 samples)	Analyte-specific %R. See **Worksheet 12**.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed	Analyst	Same as QC Acceptance Limits

QAPP Worksheet #28-5: Analytical Quality Control and Corrective Action – 8082A, Continued

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
			analytes if sufficient sample material is available.		
LCSD	1 per 20 samples	RPD ≤ 30%	If above control limits and analyte(s) not detected, data may be reported with qualifiers and discussed in the Case Narrative. Otherwise, the entire batch must be re-prepared and reanalyzed.	Analyst	Same as QC Acceptance Limits
MS	1/Batch (20 samples)	Analyte-specific %R. See **Worksheet 12**.	Examine the project-specific requirements. Contact the client as to additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. Explain in the case narrative. The MS is for matrix evaluation only. If MS falls outside limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Analyst	Same as QC Acceptance Limits

QAPP Worksheet #28-5: Analytical Quality Control and Corrective Action – 8082A, Continued

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
MSD	1 per 20 samples	RPD ≤ 30%	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst	Same as QC Acceptance Limits
Surrogate Compounds	All field samples and QC samples	%Rs Tetrachloro-m-xylene – Aqueous = 25-120; Solid = 44-130 Decachlorobiphenyl – Aqueous = 30-136; Solid = 59-130	Correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative. Apply Q-flag to all associated analytes if acceptance criteria are not met. Explain in the case narrative. All surrogates analyzed must be reported.	Analyst	Same as QC Acceptance Limits
Second Column Confirmation	All positive results	Calibration and QC criteria for the confirmation analysis are the same as for the	Report the result from the primary column. Apply J-flag if RPD > 40% in data validation.	Analyst	Same as initial or primary column analysis.

QAPP Worksheet #28-5: Analytical Quality Control and Corrective Action – 8082A, Continued

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
		primary column analysis. The RPD between results for the primary and secondary columns must be ≤ 40%.			

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-6: Analytical Quality Control and Corrective Action – 8260C

Matrix: Aqueous and Solid
Analytical Group: Volatile Organics
Analytical Method/SOP Reference: EPA 8260C/SOP DV-MS-0010

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blanks	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines (aqueous only).	Field personnel Data Validators	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater
Trip Blanks	1 per cooler	N/A	Qualify data according to validation guidelines.	Data Validators	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater

QAPP Worksheet #28-6: Analytical Quality Control and Corrective Action – 8260C, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Internal Standards	Each calibration standard, sample and QC sample	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Method Blank	One per preparatory batch (20 samples)	No Target Analytes > ½ LOQ or > 1/10 the amount in any sample or 1/10 the regulatory limit (whichever is greater). No common lab contaminants >LOQ.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst / Section Supervisor	Same as QC Acceptance Limits
LCS/ LCS Duplicate	One per preparatory batch (20 samples)	Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between LCS and LCSD ≤ 20%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-6: Analytical Quality Control and Corrective Action – 8260C, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Matrix Spike/Matrix Spike Duplicate	One MS/MSD per preparatory batch (20 samples)	Recovery: Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between MS and MSD ≤ 20%	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Surrogates	Every field and QC sample	Surrogate-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for surrogate-specific limits.	Correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference is present, the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-7: Analytical Quality Control and Corrective Action – 8270D

Matrix: Aqueous and Solid
Analytical Group: Semivolatile Organics
Analytical Method/SOP Reference: EPA 8270D/SOP SA-SM-033 (Savannah)

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines (aqueous only).	Field personnel Data Validators	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater
Internal Standard (ISTD)	Each calibration standard, sample and QC sample	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-7: Analytical Quality Control and Corrective Action – 8270D, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch (20 samples)	No Target Compounds> ½ LOQ and > 1/10 the amount in any sample or 1/10 the regulatory limit (whichever is greater). No common lab contaminants >LOQ.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst / Section Supervisor	Same as QC Acceptance Limits
LCS/LCSD	One per preparatory batch (20 samples)	Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD: RPD between LCS and LCSD ≤20%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Matrix Spike / Matrix Spike Duplicate (MS/MSD)	One MS/MSD per preparatory batch (20 samples)	Recovery: Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD: RPD between MS and MSD ≤ 20%	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-7: Analytical Quality Control and Corrective Action – 8270D, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Surrogate	Every field and QC sample	Surrogate-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for surrogate-specific limits.	Correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference is present, the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-8: Analytical Quality Control and Corrective Action – 8270D-SIM

Matrix: Aqueous and Solid
Analytical Group: Semivolatile Organics - Polynuclear Aromatic Hydrocarbons
Analytical Method/SOP Reference: EPA 8270D/SOP DV-MS-0002

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples and RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines (aqueous only).	Field personnel Data Validators	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater
Internal Standards	Each calibration standard, sample and QC sample	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-8: Analytical Quality Control and Corrective Action – 8270D-SIM, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch (20 samples)	No Target Compounds > ½ LOQ or > 1/10 the amount in any sample or 1/10 the regulatory limit (whichever is greater)	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst / Section Supervisor	Same as QC Acceptance Limits
LCS/LCSD	One per preparatory batch (20 samples)	Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between LCS and LCSD ≤ 40% for all analytes	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Matrix Spike/Matrix Spike Duplicate	One MS/MSD per preparatory batch (20 samples)	Recovery: Analyte-specific (DoD QSM V.5.3 established) recovery limits. See **Worksheet 12** for analyte-specific limits. RPD: RPD between MS and MSD ≤ 40% for all analytes (between MS/MSD or sample and MD).	Determine root cause; J flag analytes in parent sample if acceptance criteria not met. Discuss in narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Surrogates	Every field and QC sample	PAH Analysis: Deuterated monitoring compounds (DMC) required for PAH target	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-8: Analytical Quality Control and Corrective Action – 8270D-SIM, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		analytes: Fluoranthene-d_{10} and 2-Methylnaphthylene-d_{10} Minimum relative response factor for PAH DMCs is 0.40 Surrogate-specific (DoD QSM V.5.3 established) recovery limits. or current in-house limits if no QSM limits published or 50-150% until limits can be established. See **Worksheet 12** for surrogate-specific limits.	interference is present, notify the client, report with narrative comment. Otherwise, reextract and reanalyze.		
Characteristic ions for MS confirmation	Minimum 3 ions.	The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities must be > 0. Confirmation requires S/N ratio of ≥ 3 for each quant and confirmation ion.	No data can be reported without MS confirmation.	Analyst / Section Supervisor	Same as QC Acceptance Limits

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-9: Analytical Quality Control and Corrective Action – 8330A

Matrix: Solid
Analytical Group: Explosives
Analytical Method/SOP Reference: EPA 8330A/SOP DV-LC-0002

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤50% for solid samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible.	Field personnel	No target analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater
Method Blank	1/Batch (20 samples)	No Target Compounds > ½ LOQ or > 1/10 the amount in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst / Section Supervisor	No Target Compounds>1/2 LOQ or > 1/10 the amount in any sample or 1/10 the regulatory limit

QAPP Worksheet #28-9: Analytical Quality Control and Corrective Action – 8330A, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
					(whichever is greater).
LCS/LCSD	1/Batch (20 samples)	QSM limits (if available) or current in-house limits if no QSM limits published. See **Worksheet 12** for analyte-specific limits. RPD: RPD between LCS and LCSD ≤ 30%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. If insufficient sample, then data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Matrix Spike/Matrix Spike Duplicate	1/Batch (20 samples)	QSM limits (if available) or current in-house limits if no QSM limits published. See **Worksheet 12** for analyte-specific limits. RPD: RPD between MS and MSD ≤ 30%	For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. Explain in the case narrative. The MS is for matrix evaluation only. If MS falls outside MS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-9: Analytical Quality Control and Corrective Action – 8330A, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Surrogates	Every field sample and QC sample	QSM limits (if available) or current in-house limits if no QSM limits published. See **Worksheet 12** for analyte-specific limits.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative. Apply Q-flag to all associated analytes if acceptance criteria are not met. Explain in the case narrative. All surrogates analyzed must be reported.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Second-column confirmation	All positive results	Calibration and QC criteria for the confirmation analysis are the same as for the primary column analysis. The RPD between results for the primary and secondary columns must be ≤ 40%.	Apply J-flag if RPD > 40% and discuss in case narrative. Use project-specific reporting requirements if available; otherwise use method requirements if available; otherwise report the result from the primary column.	Analyst / Section Supervisor	Same as for initial or primary column analysis The RPD between results for the primary and

QAPP Worksheet #28-9: Analytical Quality Control and Corrective Action – 8330A, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
					secondary columns must be ≤ 40%.

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-10: Analytical Quality Control and Corrective Action – 8330B

Matrix: Aqueous
Analytical Group: Explosives – Picric Acid
Analytical Method/SOP Reference: EPA 8330B/SOP DV-LC-0002

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤30% for aqueous samples when at least one result is greater than the LOQ
Equipment Blank	1 per day	N/A	Clean equipment carefully or use disposable sampling equipment where possible. Qualify data according to validation guidelines.	Field personnel Data Validators	No target analytes detected >1/2 LOQ or > $1/10^{th}$ the amount measured in any sample, or $1/10^{th}$ the regulatory limit, whichever is greater
Method Blank	1/Preparatory Batch (20 samples)	No Target Compounds> ½ LOQ or > 1/10 the amount in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-10: Analytical Quality Control and Corrective Action – 8330B, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
LCS	1/Preparatory Batch (20 samples)	QSM limits (if available) or current in-house limits if no QSM limits published. See **Worksheet 12** for analyte-specific limits.	Correct problem. If required, reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. If insufficient sample, then data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Matrix Spike/Matrix Spike Duplicate	1/Preparatory Batch (20 samples)	QSM limits (if available) or current in-house limits if no QSM limits published. See **Worksheet 12** for analyte-specific limits. RPD: $\leq$ 20% For sample/MD: % recovery and RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.	For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. Explain in the case narrative. The MS is for matrix evaluation only. If MS falls outside MS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-10: Analytical Quality Control and Corrective Action – 8330B, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Surrogates	Every field sample and QC samples	QSM limits (if available) or current in-house limits if no QSM limits published. See **Worksheet 12** for surrogate-specific limits.	For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and failures must be discussed in the case narrative. Apply Q-flag to all associated analytes if acceptance criteria are not met. Explain in the case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits
Confirmation of positive results	All results > DL must be confirmed Confirmation not required if LC/MS or LC/MS/MS used for primary analysis.	Calibration and QC criteria are the same for the confirmation analysis as for initial or primary column analysis. Results between primary and second column RPD $\leq$ 40%.	Report from both columns. Apply J-flag if RPD > 40%; discuss in case narrative.	Analyst / Section Supervisor	Same as QC Acceptance Limits

QAPP Worksheet #28-10: Analytical Quality Control and Corrective Action – 8330B, Continued

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		Confirmation column must be capable of resolving all of the analytes of interest and must have a different retention time order relative to the primary column. Report from primary column unless project directs otherwise.			

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-11: Analytical Quality Control and Corrective Action – 9060A

Matrix: Solid
Analytical Group: Total Organic Carbon
Analytical Method/SOP Reference: EPA 9060A/DV-WC-0048

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤50% for solid samples when at least one result is greater than the LOQ
MB	1/ Batch (20 samples)	No target analytes ≥ ½ LOQ.	Rinse boats thoroughly and re-run method blank	Lab Manager / Analyst	No target analytes ≥ ½ LOQ.
LCS	1/ Batch (20 samples)	Recovery 46% - 130% of true value	Reprep LCS and re-analyze.	Lab Manager / Analyst	Same as QC Acceptance Limits.
LCSD	1 per batch of 20 or fewer samples	RPD ≤ 20%	Reprep LCS and re-analyze.	Analyst / Section Supervisor	Same as QC Acceptance Limits.
MS	1/ Batch (20 samples)	Recovery 46% - 130% of true value	Reprep MS and re-analyze.	Lab Manager / Analyst	Same as QC Acceptance Limits.
MSD	1 per batch of 20 or fewer samples	RPD ≤ 20%	Reprep MS and re-analyze.	Analyst / Section Supervisor	Same as QC Acceptance Limits.
Sample Duplicate (SD)	1/ Batch (20 samples)	RPD ≤ 50%	Reanalysis of duplicate and associated samples	Lab Manager / Analyst	Same as QC Acceptance Limits.

QAPP Worksheet #28-11: Analytical Quality Control and Corrective Action – 9060A, Continued

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-12: Analytical Quality Control and Corrective Action – 9081

Matrix: Solid
Analytical Group: CEC
Analytical Method/SOP Reference: EPA 9081/CC-ATM-M020

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples	N/A	Discuss RPD exceptions in the Phase 1 Field Report. Impacts will be considered in the refinement of the CSM.	End-Data Users	RPD ≤50% for solid samples when at least one result is greater than the LOQ
MB	One per preparation batch (20 samples)	No Target Analytes > ½ RL (LOQ)	Correct problem, then re-prep and reanalyze all samples and QC in the affected analytical batch.	Analyst	Same as QC Acceptance Limits
Laboratory Duplicate	One per every 10 project samples	≤ 20% RPD	Report.	Analyst	Same as QC Acceptance Limits

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #28-13: Analytical Quality Control and Corrective Action – Grain Size

Matrix: Solid
Analytical Group: Grain Size
Analytical Method/SOP: ASTM D422/TA-WC-0183

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
Field Duplicate	1 per 10 field samples	RPD ≤ 50%	Examine project DQOs with PM. Evaluate data to determine source of difference between results.	Analyst	Same as QC Acceptance Limits
Laboratory Duplicate	1 per 20 samples	RPD ≤ 20%	Reanalysis of duplicate and associated samples	Analyst	Same as QC Acceptance Limits

Notes:

1. This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2. SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #29: Project Documents and Records
(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

Sample Collection and Field Records			
Record	**Generation**	**Verification**	**Storage Location/Archival**
Field Logbook or Data Collection Sheets (field forms)	Field Sampler or FTL or Designee	RI TM	Project File
Field SOPs	FTL or Designee	RI TM	Project File
Chain-of-Custody Forms	Sample Manager or Designee	FTL or Designee	Project File
Air Bills	Sample Manager or Designee	FTL or Designee	Project File
Sample Tracking Forms	Sample Manager or Designee	FTL or Designee	Project File
Daily Quality Control (QC) Reports	FTL or Designee	RI TM or Designee	Project File
Deviations from Scope of Work - Field Change Notification Forms	FTL or Designee	RI TM or Designee	Project File
Corrective Action Reports	FTL	FTL or Designee	Project File
Correspondence	FTL	FTL or Designee	Project File
Soil Boring Logs /Groundwater Elevation Logs	FTL or Designee	Field Geologist or Designee	Project File
Water Quality Data Logs (field parameters)	FTL or Designee	Field Geologist or Designee	Project File
Equipment Calibration and Maintenance Logs	FTL or Designee	Field Scientist or Designee	Project File
Photographs	FTL or Designee	Field Scientist or Designee	Project File

QAPP Worksheet #29: Project Documents and Records, Continued

Sample Collection and Field Records			
Field Equipment Inspection and Maintenance records	FTL or Designee	Field Geologist or Designee	Project File
Monitoring Well Construction Diagrams	FTL or Designee	Field Geologist or Designee	Project File
Sample Disposal Logs and Waste Manifests	FTL or Designee	Field Scientist or Designee	Project File

Project Assessments			
Record	**Generation**	**Verification**	**Storage Location/Archival**
Audit Plans and Reports	Auditor	QA Manager or Designee	Project File
Field Audit Checklists	Auditor	QA Manager or Designee	Project File
Corrective Action Report	QA Manager	FTL	Project File
Self-Assessment Checklist	FTL or Designee	QA Specialist	Project File
Data Verification Checklists	FTL or Designee	ASC	Project File
Data Validation Report	Data validator	Chemist	Project File
Data Usability Assessment Report	ASC or Designee	Chemist	Project File

QAPP Worksheet #29: Project Documents and Records, Continued

Laboratory Records			
Record	**Generation**	**Verification**	**Storage Location/Archival**
Bid Sheets, Scopes of Work	RI TM or Designee	Technical Reviewer and Procurement Specialist	Procurement File
Subcontract Laboratory Certifications	Laboratory QA Officer	Chemist or QA Specialist	Procurement File
Subcontract Laboratory QA Plans	Laboratory QA Officer	Chemist or QA Specialist	Procurement File
Laboratory SOPs	Laboratory QA Officer	Chemist or QA Specialist	Procurement File

QAPP Worksheet #29: Project Documents and Records, Continued

Laboratory Data Deliverables				
Record	**Organics**	**Metals**	**Wet Chemistry**	**Other**
Narrative	X	X	X	X
COC Form	X	X	X	X
Summary Results	X	X	X	X
Analytical sample results	X	X	X	X
QC Results	X	X	X	X
Chromatograms	X	NA	NA	TBD
Sample Preparation Log	X	X	X	X
Sample Run Log	X	X	X	X
Tentatively Identified Compounds (TICs)	X	NA	NA	NA
Raw Data	X	X	X	X

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action
(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Daily Field Work Report	FTL Bluestone	Daily during field activities	NA	Daily Field Work Report	Daily during field activities
ELAP Accreditation	TA-Denver (A2LA) TA-Savannah (ANAB) TA-Seattle (ANAB)	Annually	TA – Denver (October 2021) TA – Savannah (September 2022) TA – Seattle (January 2022)	Certification	TA – Denver (October 2021) TA – Savannah (September 2022) TA – Seattle (January 2022)
Data Review	Bluestone	Once	28 business days after receipt of data	Validation Report	28 business days after receipt of data
NELAP (NY Certification)	NY State Department of Health	Annually	TA – Denver (April 2021) TA-Savannah (April 2021)	Certification	TA – Denver (April 2021) TA-Savannah (April 2021)
DoD/ISO 17025	TA Denver - L-A-B for DoD	Every two years	TA Denver - May 2021	Corrective Action Plan	Due 30 days after receiving final audit report
	TA Savannah – ANAB TA Seattle – ANAB	Annually	TA Savannah - June 2021 TA Seattle - January 2022		
ISO/IEC 17025:2017	MicroVision – John Knowles	Every two years	August 2021	Corrective Action Plan	Due 30 days after receiving final audit report

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action, Continued

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Internal Laboratory Audit	TA Denver - Roxanne Sullivan TA Savannah – Kim Chamberlain TA Seattle – Terri Torres	Annually	TA Denver - April 2021 TA Savannah -November 2021 TA Seattle – August 2021	Written Audit Report	30 Days after audit
Corrective Action Report	Michael Badeau, Bluestone	As needed	---	Written Report	---

Notes:

The QA Manager (QAM) will determine the need for any field or office audits. If the FTL requests self-assessments in lieu of the project audit, the QAM will review and approve or reject the self-assessments being considered.

Field auditors are selected based on level of experience and technical specialty. Office audits are performed by trained and approved QA Staff members.

Findings and deviations from plans will require corrective actions which will be documented and discussed appropriately. The USACE will be notified by the PM.

QAPP Worksheet #34: Data Verification and Validation Inputs
(UFP-QAPP Manual Section 5.2.1 and Table 9)
(EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (Completeness)	Validation (Conformance to Specifications)
Planning Documents/Records			
1	Approved QAPP	X	
2	Contract/PWS	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
Field Records			
5	Field logbooks/Daily Reports	X	
6	Equipment calibration records	X	
7	COC Forms	X	
8	Sampling diagrams/surveys/sampling figures	X	
9	Drilling, boring logs, geophysical logs	X	
10	Relevant Correspondence	X	
11	Change orders/deviations	X	
12	Field audit reports, if audited	X	
13	Field corrective action reports	X	
14	Photographs	X	
15	Groundwater sampling forms	X	
16	Well development forms	X	
17	Water level forms (stored in logbook)	X	
Analytical Data Package[1]			
18	Cover sheet (laboratory identifying information)	X	X
19	Case narrative	X	X
20	Internal laboratory COC	X	
21	Sample receipt records	X	X
22	Sample chronology (i.e. dates and times of receipt, preparation, & analysis)	X	
23	Communication records	X	X
24	LOD/LOQ establishment and verification	X	
25	Standards Traceability	X	
26	Instrument calibration records	X	
27	Definition of laboratory qualifiers	X	X
28	Results reporting forms	X	X
29	QC sample results	X	X
30	Corrective action reports	X	X
31	Raw data	X	X
32	Electronic data deliverable (EDD)	X	X
33	Air Bills	X	X

QAPP Worksheet #34: Data Verification and Validation Inputs, Continued

34	Telephone Logs	X	X

Notes:

1.The items to be reviewed during the validation process are determined by the validation stage as specified in **Worksheet #36**.

QAPP Worksheet #35: Data Verification Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbook/Daily Reports	Field SOPs (**Appendix B**), Daily Reports, Field Corrective Action Reports	Verify that records are present and complete for each day of field activities, and daily reports are submitted. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that field instruments were calibrated appropriately, and field monitoring was performed as required, and results are documented.	Daily – FTL/PM At conclusion of field activities - Project QC Staff
COC Forms	Field SOPs (**Appendix B),** QAPP, and field logbook	Verify the completeness of COC records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily – FTL At conclusion of field activities – Project QC Staff
Laboratory Deliverable	QAPP, eQAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the COCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present. Determine potential impacts from noted/approved deviations in regard to Project Quality Objectives (PQOs). With the exception of IDW samples, Grain Size, and Methods for which the laboratories are not DoD-certified (CEC and Coal Ash Microscopy), analytical data will be verified through FUDSChem, a web-based data review program.	Before release – Laboratory QAM Upon receipt – Bluestone Validator

QAPP Worksheet #35: Data Verification Procedures, Continued

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
		IDW samples will not be validated but the data package will be verified for completeness and that limits for expected disposal requirements are met.	
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Project QC Staff

QAPP Worksheet #36a: Data Validation Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Validation Code and Label Identifier Table:

Validation Code	Validation Label	Description/Reference
S1VE	Stage 1 Validation Electronic	EPA 540-R-08-005
S1VM	Stage 1 Validation Manual	EPA 540-R-08-005
S1VEM	Stage 1 Validation Electronic and Manual	EPA 540-R-08-005
S2aVE	Stage 2a Validation Electronic	EPA 540-R-08-005
S2aVM	Stage 2a Validation Manual	EPA 540-R-08-005
S2aVEM	Stage 2a Validation Electronic and Manual	EPA 540-R-08-005
S2bVE	Stage 2b Validation Electronic	EPA 540-R-08-005
S2bVM	Stage 2b Validation Manual	EPA 540-R-08-005
S2bVEM	Stage 2b Validation Electronic and Manual	EPA 540-R-08-005
S3VE	Stage 3 Validation Electronic	EPA 540-R-08-005
S3VM	Stage 3 Validation Manual	EPA 540-R-08-005
S3VEM	Stage 3 Validation Electronic and Manual	EPA 540-R-08-005
S4VE	Stage 4 Validation Electronic	EPA 540-R-08-005
S4VM	Stage 4 Validation Manual	EPA 540-R-08-005
S4VEM	Stage 4 Validation Electronic and Manual	EPA 540-R-08-005
NV	Not Validated	EPA 540-R-08-005

Data Validation Qualifiers

The following data qualifiers will be applied during DV by a third party. Potential impacts on project DQOs will be discussed in the data usability report.

U The analyte was not detected and was reported as less than the LOD (practical quantitation limit [PQL] for non-DoD QSM analyses). The LOD (or PQL) has been adjusted for any dilution or concentration of the sample.

J The associated numerical value is an estimated quantity.

J+ The associated numerical value is an estimated quantity that may be biased high.

J- The associated numerical value is an estimated quantity that may be biased low.

UJ The undetected result is estimated due to QC sample considerations.

X The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a project chemist), but exclusion of the data is recommended.

QAPP Worksheet #36b: Data Validation Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Data Validator: FUDSCHEM/Emily Strake

Analytical Group/Method:	**Grain Size – Solid – ASTM D422**	**Metals – Aqueous and Solid – SW-846 6010C**
Data deliverable requirements:	Level 4 data package (pdf) uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.
Analytical specifications:	**Worksheets 24 and 28**, SOP TA-WC-0183	**Worksheets 24 and 28**, SOP DV-MT-0021
Measurement performance criteria:	**Worksheet 12**	W**orksheet 12**
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R-2017-001, January 2017 (USEPA 2017b). Analyses not provided for in USEPA 2017b will be reviewed based upon analytical method specific criteria.	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B Measurement Performance Indicators (MPIs). Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R-2017-001, January 2017 (USEPA 2017b). Analyses not provided for in USEPA 2017b will be reviewed based upon DoD QSM 5.1 requirements and analytical method specific criteria.
Validation code (*see attached table):	S1VM	S2bVEM
Electronic validation program/version:	Not Applicable	FUDSChem

QAPP Worksheet #36b: Data Validation Procedures

Data Validator: FUDSCHEM/Emily Strake

Analytical Group/Method:	Metals – Aqueous and Solid – SW-846 6020A	Mercury – Aqueous & Solid – SW-846 7470A /7471B
Data deliverable requirements:	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.
Analytical specifications:	**Worksheets 24 and 28**, SOP DV-MT-0022	**Worksheets 24 and 28**, SOPs DV-MT-0017/DV-MT-0016
Measurement performance criteria:	**Worksheet 12**	**Worksheet 12**
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B Measurement Performance Indicators (MPIs). Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R-2017-001, January 2017 (USEPA 2017b). Analyses not provided for in USEPA 2017b will be reviewed based upon DoD QSM 5.1 requirements and analytical method specific criteria.	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B Measurement Performance Indicators (MPIs). Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R-2017-001, January 2017 (USEPA 2017b). Analyses not provided for in USEPA 2017b will be reviewed based upon DoD QSM 5.1 requirements and analytical method specific criteria.
Validation code (*see attached table):	S2bVEM	S2bVEM
Electronic validation program/version:	FUDSChem	FUDSCHEM

QAPP Worksheet #36b: Data Validation Procedures

Data Validator: FUDSCHEM/Emily Strake

Analytical Group/Method:	**PCBs - Aqueous and Solid - SW-846 8082A**	**CEC – Solid – SW-846 9081**
Data deliverable requirements:	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.	Level 4 data package (pdf) uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.
Analytical specifications:	**Worksheets 24 and 28**, SOP DV-GC-0021	**Worksheets 24 and 28**, SOP CC-ATM-M020
Measurement performance criteria:	**Worksheet 12**	**Worksheet 12**
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B MPIs. Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Organic Superfund Methods Data Review, USEPA-540-R-2017-002, January 2017(USEPA 2017c).	Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R-2017-001, January 2017 (USEPA 2017b). Analyses not provided for in USEPA 2017b will be reviewed based upon DoD QSM 5.1 requirements and analytical method specific criteria.
Validation code (*see attached table):	S2bVEM	S1VM
Electronic validation program/version:	FUDSCHEM	Not Applicable

QAPP Worksheet #36b: Data Validation Procedures

Data Validator: FUDSCHEM/Emily Strake

Analytical Group/Method:	**VOCs – Aqueous & Solid – SW-846 8260C**	**SVOCs – Aqueous & Solid – SW-846 8270D**
Data deliverable requirements:	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.
Analytical specifications:	**Worksheets 24 and 28**, SOP DV-MS-0010	**Worksheets 24 and 28**, SOP SA-SM-033
Measurement performance criteria:	**Worksheet 12**	**Worksheet 12**
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B MPIs. Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), DoD Data Validation Guidelines Module 1, 08/03/2018 (EDQW, 2018) and USEPA National Functional Guidelines for Organic Superfund Methods Data Review, USEPA-540-R-2017-002, January 2017(USEPA 2017c).	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B MPIs. Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), DoD Data Validation Guidelines Module 1, 08/03/2018 (EDQW, 2018), and USEPA National Functional Guidelines for Organic Superfund Methods Data Review, USEPA-540-R-2017-002, January 2017 (USEPA 2017c).
Validation code (*see attached table):	S2bVEM	S2bVEM
Electronic validation program/version:	FUDSCHEM	FUDSCHEM

QAPP Worksheet #36b: Data Validation Procedures

Data Validator: FUDSCHEM/Emily Strake

Analytical Group/Method:	PAHs – Aqueous & Solid – SW-846 8270D-SIM	Picric Acid – Aqueous & Solid – SW-846 8330A/B
Data deliverable requirements:	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.
Analytical specifications:	**Worksheets 24 and 28**, SOP DV-MS-0002	**Worksheets 24 and 28**, SOP DV-LC-0002
Measurement performance criteria:	**Worksheet 12**	**Worksheet 12**
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B MPIs. Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), DoD Data Validation Guidelines Module 1, 08/03/2018 (EDQW, 2018), and USEPA National Functional Guidelines for Organic Superfund Methods Data Review, USEPA-540-R-2017-002, January 2017 (USEPA 2017c).	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B MPIs. Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Organic Superfund Methods Data Review, USEPA-540-R-2017-002, January 2017 (USEPA 2017c). Analyses not provided for in USEPA 2017c will be reviewed based upon DoD QSM 5.1 requirements and analytical method specific criteria.
Validation code (*see attached table):	S2bVEM	S2bVEM
Electronic validation program/version:	FUDSCHEM	FUDSCHEM

QAPP Worksheet #36b: Data Validation Procedures

Data Validator: FUDSCHEM/Emily Strake

Analytical Group/Method:	**TOC – Solid – SW-846 9060A**
Data deliverable requirements:	SEDD 2a, version 5.2 with three tier reporting, Level 4 data package (pdf) and uploaded by the laboratory without errors to FUDSChem within 3 weeks of sample date.
Analytical specifications:	**Worksheets 24 and 28**, SOP DV-WC-0048
Measurement performance criteria:	**Worksheet 12**
Percent of data packages to be validated:	100%
Percent of raw data reviewed:	0%
Percent of results to be recalculated:	0%
Validation procedure:	FUDSCHEM electronic validation with manual verification of flagged data and evaluation of additional 2B MPIs. Validation will be completed using project established QA/QC limits, DoD General Data Validation Guidelines (EDQW, 2019), and USEPA National Functional Guidelines for Organic Superfund Methods Data Review, USEPA-540-R-2017-002, January 2017 (USEPA 2017c). Analyses not provided for in USEPA 2017c will be reviewed based upon DoD QSM 5.1 requirements and analytical method specific criteria.
Validation code (*see attached table):	S2bVEM
Electronic validation program/version:	FUDSCHEM

QAPP Worksheet #37: Data Usability Assessment
(UFP-QAPP Manual Section 5.2.3 including Table 12)
(EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)

Identify personnel (organization and position/title) responsible for participating in the data usability assessment:

- Project Manager
- Task Leader
- FTL
- Risk Assessor
- Geologist/Hydrogeologist
- Project Engineer
- Project Chemist
- Data Validator
- Project QA Specialist
- Data Manager
- Data Coordinator
- Data Specialist

Step 1	Review the project's objectives and sampling design Review the key outputs defined during systematic planning (i.e., PQOs or DQOs and method performance criteria [MPCs]) to make sure they are still applicable. Review the sampling design for consistency with stated objectives. This provides the context for interpreting the data in subsequent steps.
Step 2	Review the data verification and DV outputs Review available QA reports. Perform basic calculations and summarize the data (using graphs, maps, tables, etc.). Look for patterns, trends, and anomalies (i.e., unexpected results). Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, non-compliant proficiency testing [PT] sample results, and SOP deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.
Step 3	Verify the assumptions of the selected statistical method If statistics will be used for the project, verify whether underlying assumptions for selected statistical methods (if documented in the QAPP) are valid. Common assumptions include the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, etc. Depending on the robustness of the statistical method, minor deviations from assumptions usually are not critical to statistical analysis and data interpretation. If serious deviations from assumptions are discovered, then another statistical method may need to be selected.
Step 4	Implement the statistical method Implement the specified statistical procedures for analyzing the data and review underlying assumptions. Consider the consequences for selecting the incorrect alternative; for estimation projects (e.g., establishing a boundary for surface soil impacts), consider the tolerance for uncertainty in measurements.

QAPP Worksheet #37: Data Usability Assessment, Continued

Step 5	Document data usability and draw conclusions Determine if the data can be used as intended, considering implications of deviations and corrective actions. Discuss Data Quality Indicators (DQIs) (see Usability Assessment details below). Assess the performance of the sampling design and Identify limitations on data use. Update the CSM and document conclusions.

The usability assessment will be documented as follows:

The Data Usability Assessment will be performed by Bluestone and a recommendation will be made to the PDT. The Project Manager will be responsible for information in the Data Usability Assessment and will also be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. Note that the Data Usability Assessment will be conducted on validated or reviewed data. After the Data Usability Assessment has been performed, data deemed appropriate for use will then be used in the subsequent project evaluation. The results of the Data Usability Assessment will be presented in the project measurement report such as the Risk Assessment, RI or other report. Tables will be prepared of the samples and analytical parameters, field and trip blank results, field duplicates comparison, and completeness of the data set. The following items will be assessed, and conclusions drawn based on their results.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

Precision – Results of laboratory duplicates will be assessed during DV and data will be qualified according to the DV procedures cited on **Worksheet #36**. Field duplicates will be assessed by matrix using the RPD for each pair of results reported above the LOQ for organic and inorganic analyses respectively. Absolute difference will be used for low results as described in **Worksheet #28**. A discussion summarizing the results of laboratory and field precision and any limitations on the use of the data will be described in the Data Usability Report.

Field duplicates – Field duplicates will be evaluated against the MPCs outlined in **Worksheet #12**. The PM will review the extent of exceedance of the field duplicate criteria. For groundwater, the sample results will be flagged according to the DV protocol. For soils, the exceedances will be compared with the field lithological logs and grain size results, if available. Based on this review, the project manager will determine whether the exceedance is due to inherent soil heterogeneity or the result of sample handling in the field or laboratory. This information will be included in the data assessment report. As an added measure, the FTL will be asked to inspect the soil coning and quartering procedures and re-train staff if needed. The data assessor will review the DV report. If the field duplicate comparison is not included, it will be performed by the assessor.

Accuracy/Bias Contamination – Laboratory blank results will be assessed as part of DV. During the DV process the validator will qualify the data following the procedures listed on **Worksheet #36**. A discussion summarizing the results of laboratory accuracy and bias based on contamination will be presented and limitations on the use of the data will be described.

QAPP Worksheet #37: Data Usability Assessment, Continued

Accuracy/Bias – The results for all matrix spike and surrogate standard spike analyses will be evaluated based on the requirements listed in **Worksheets #12 and #28**. A discussion will follow, summarizing accuracy and bias. Any conclusions about the accuracy and bias of the analyses will be drawn and any limitations on the use of the data will be described.

Sensitivity – Data results will be compared to criteria provided on **Worksheet #15**. A discussion summarizing any conclusions about sensitivity of the analyses will be presented and any limitations on the use of the data will be described.

Representativeness – Representativeness is achieved through adherence to sampling and analytical procedures described in this QAPP and compliance with stipulated sample holding times. After evaluation of relative compliance with specified procedures and holding times, conclusions about data representativeness will be drawn and any limitations on the use of data will be described. DV narratives will also be reviewed and any conclusions about the representativeness of the data set will be discussed in the Data Usability Report. Although biased sampling is being conducted, it does not preclude evaluating the representativeness parameter.

Comparability – Study results will be used in conjunction with existing data to make qualitative and quantitative assessments of the data to be used to produce the Site reports.

Reconciliation – The DQIs presented in **Worksheet #12** will be examined to determine if the MPCs were met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of major impacts observed from DV, DQIs and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. As a result of the quality determined, the usability of the data for each analysis will be established. After the combined usability of the data from all analyses for an objective is determined, it will be concluded if the DQIs were met and whether project goals were achieved. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

Completeness – A completeness check will be done on all data generated. The number of planned samples and samples with non-rejected results will be used to calculate the completeness of the obtained data set for each analytical group, with a goal of 90% completeness. The field completeness will be calculated by the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in this document. The equation for field completeness is:

$$\%\ Field\ Completeness = \frac{Number\ of\ Samples\ Received\ by\ Laboraties}{Total\ Number\ of\ Samples\ Planned\ to\ be\ Collected}\ x\ 100$$

The analytical completeness will be calculated by the ratio of total valid analytical data results (including estimated values) to the total number of analytical results requested on samples submitted for analysis. Valid analytical data results are defined as those that were not rejected during DV, due to a significant quality assurance/quality control problem. The equation for analytical completeness is:

QAPP Worksheet #37: Data Usability Assessment, Continued

$$\%\ Analytical\ Completeness = \frac{Total\ Valid\ Analytical\ Data}{Analytical\ Data\ Obtained}\ x\ 100$$

FIGURES

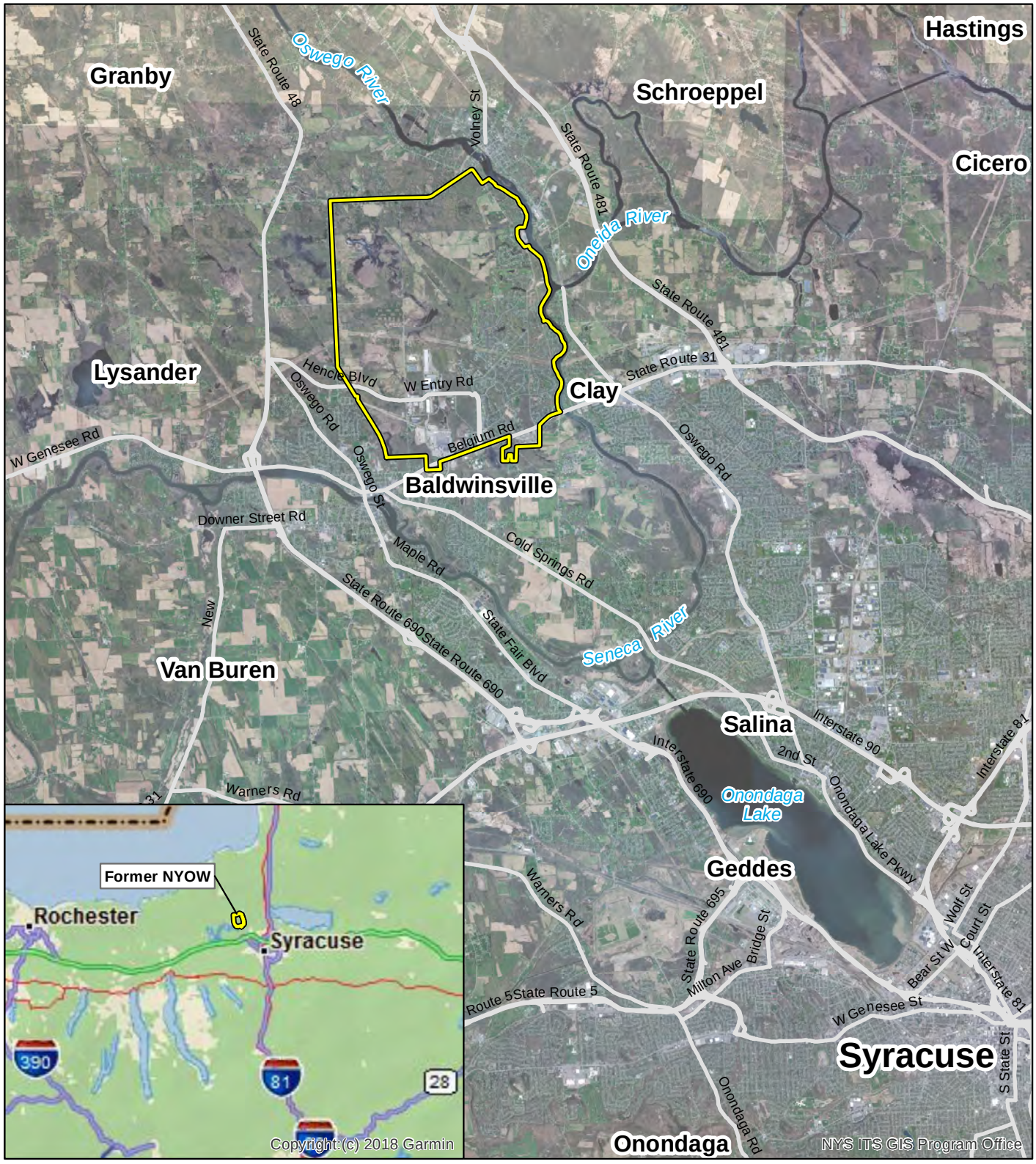


Figure 10-1: Former NYOW - Regional Map

Legend
- Former NYOW
- Major Roads

N
W E
S

0 0.5 1 2 3 4 Miles

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator

Additional Sources: NYS GIS Clearinghouse; U.S. Army Corps of Engineers

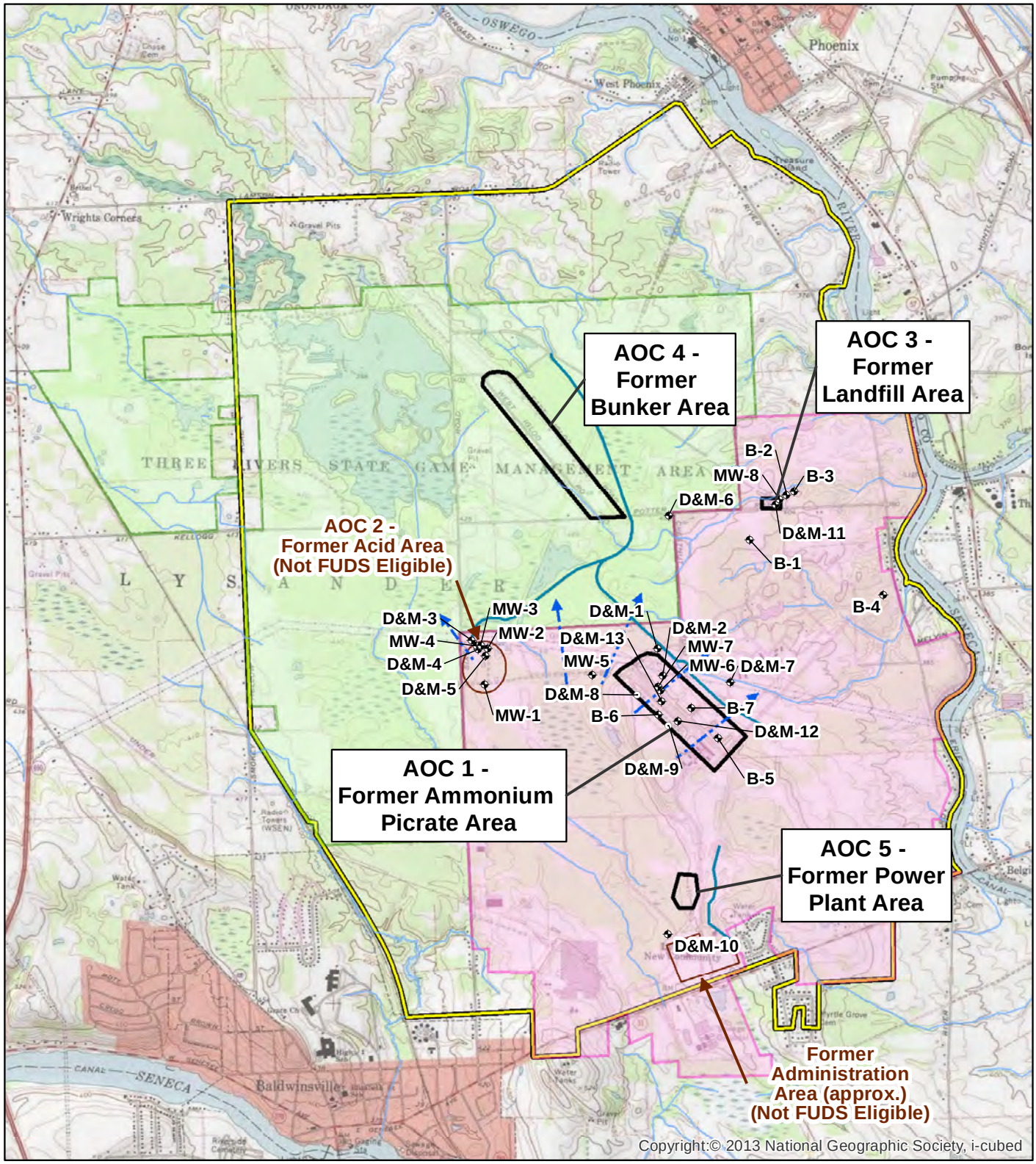


Figure 10-2: Former NYOW - Areas of Concern

Legend*
- Monitoring Well (D&M, 1981, M&E, 1990)
- Soil Boring (D&M, 1981)
- Former Wastewater Ditch
- Stream
- Inferred Groundwater Flow Direction
- Area of Concern (approx.)
- Radisson Community
- Three Rivers Wildlife Management Area
- Formerly Used Defense Site - NYOW

N, W, E, S

0 0.25 0.5 0.75 1 Miles

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator

Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; RFC, undated; USACE, 2019; USGS, 2019.

*Note: Site feature locations are approximate.

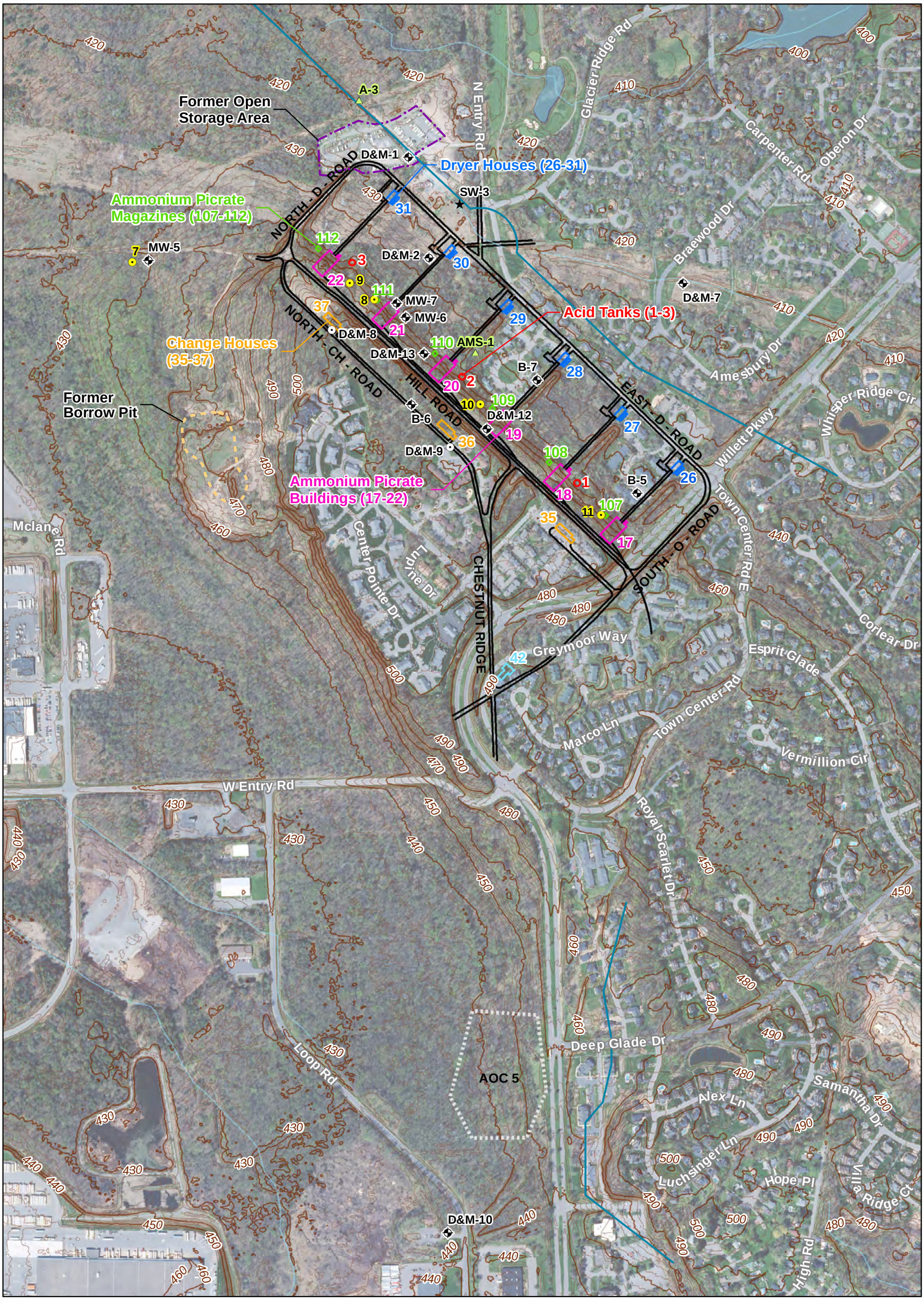


Figure 10-3: Former NYOW - AOC 1 - Former Ammonium Picrate Area Historical Features and Previous Sampling Locations

Legend*

- Grab Sample (D&M, 1981)
- Monitoring Well (D&M, 1981, M&E, 1990)
- Soil Boring (D&M, 1981)
- Soil Sample (M&E, 1990)
- Surface Water Sample (M&E, 1990)
- AOC 5 (approx.)
- Former Borrow Pit
- Former Open Storage Area
- Former Wastewater Ditch
- Water Body
- Stream
- 10' Contour
- 2' Contour
- Historical Site Feature
- Former Acid Tank (Not FUDS-Eligible)
- Ammonium Picrate Building
- Ammonium Picrate Magazine
- Change House
- Dryer House
- Laboratory Building

N W E S

0 300 600 900 1,200 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; NYS GIS, 2012 and 2019; O'Brien & Gere, 1993; RFC, undated; USGS, 2019.

*Note:
- Site feature locations are approximate.
- Existing road names are shown in white while historic road names are shown in black.

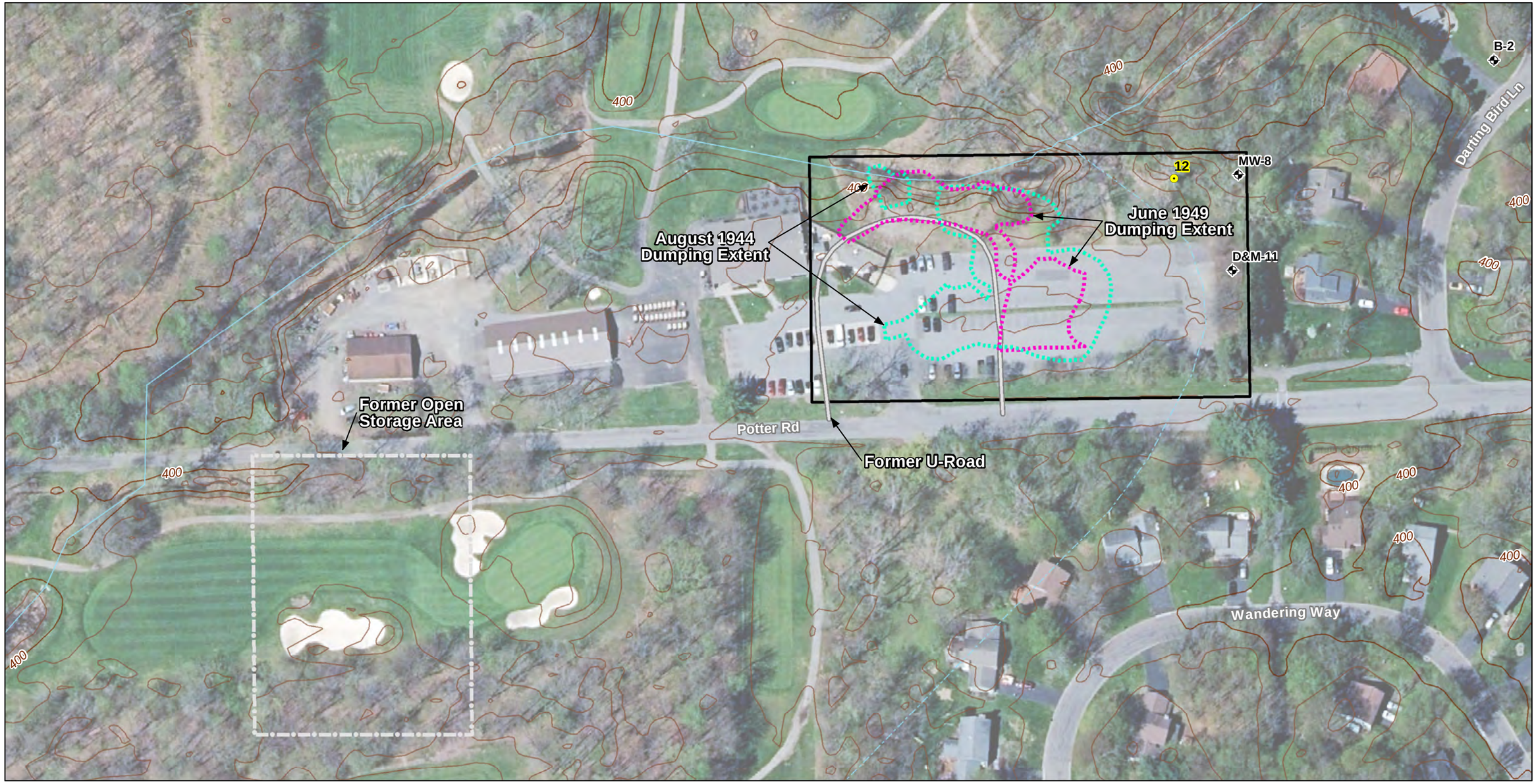


Figure 10-4: Former NYOW - AOC 3 - Former Landfill Area
Previous Sampling Locations

Legend*

- Monitoring Well (D&M, 1981, M&E, 1990)
- Soil Sample (M&E, 1990)
- Historic Drainage
- Stream
- Former Landfill Area of Concern (approx.)
- 10' Contour
- 2' Contour

Historic Site Features (USACE)

- August 1944 Dumping Extent
- June 1949 Dumping Extent
- Former Open Storage Area
- Former U-Road

N W E S

0 50 100 150 200 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; NYS GIS, 2012 and 2019; USACE, 2019; USGS, 2019.

*Note: Site feature locations are approximate.

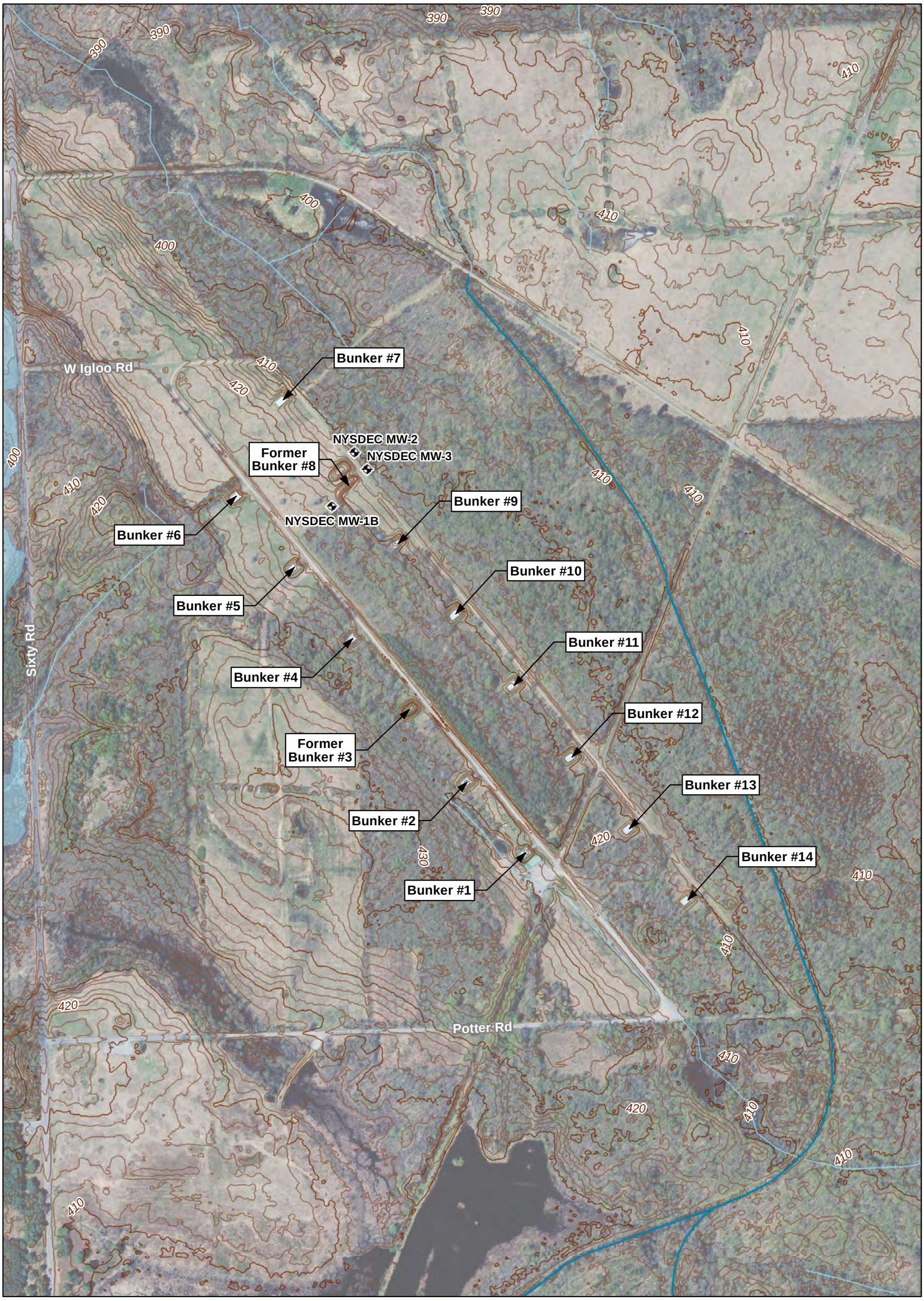


Figure 10-5: Former NYOW - AOC 4 - Former Bunker Area
Previous Sampling Locations

Legend*

- Monitoring Well (NYSDEC, 1990)
- Former Wastewater Ditch
- Stream
- Water Body
- 10' Contour
- 2' Contour

Note: Former Bunkers #3 and #8 are not FUDS eligible.

N W E S

0 250 500 750 1,000 Feet

Scale is apporximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: NYSDEC, 1990; NYS GIS, 2012 and 20019; RFC, undated; USGS, 2019.

*Note: Site feature locations are approximate.

Figure 10-6: Former NYOW - AOC 5 - Former Power Plant Area
Historical Features and Previous Sampling Locations

Legend*

- Monitoring Well (D&M, 1981, M&E, 1990)
- Former Wastewater Ditch (D&M, 1981)
- Stream
- Area of Concern (approx.)
- 10' Contour
- 2' Contour

Historic Site Features (USACE)

- AST
- Coal Storage (Eroded)
- Boiler House
- Electric Substation
- Possible Dump
- Pipeline
- Coal Aggregate Bins (CABs) (Approx.)
- Railroad
- Road
- Road - Track
- Drainage Ditch

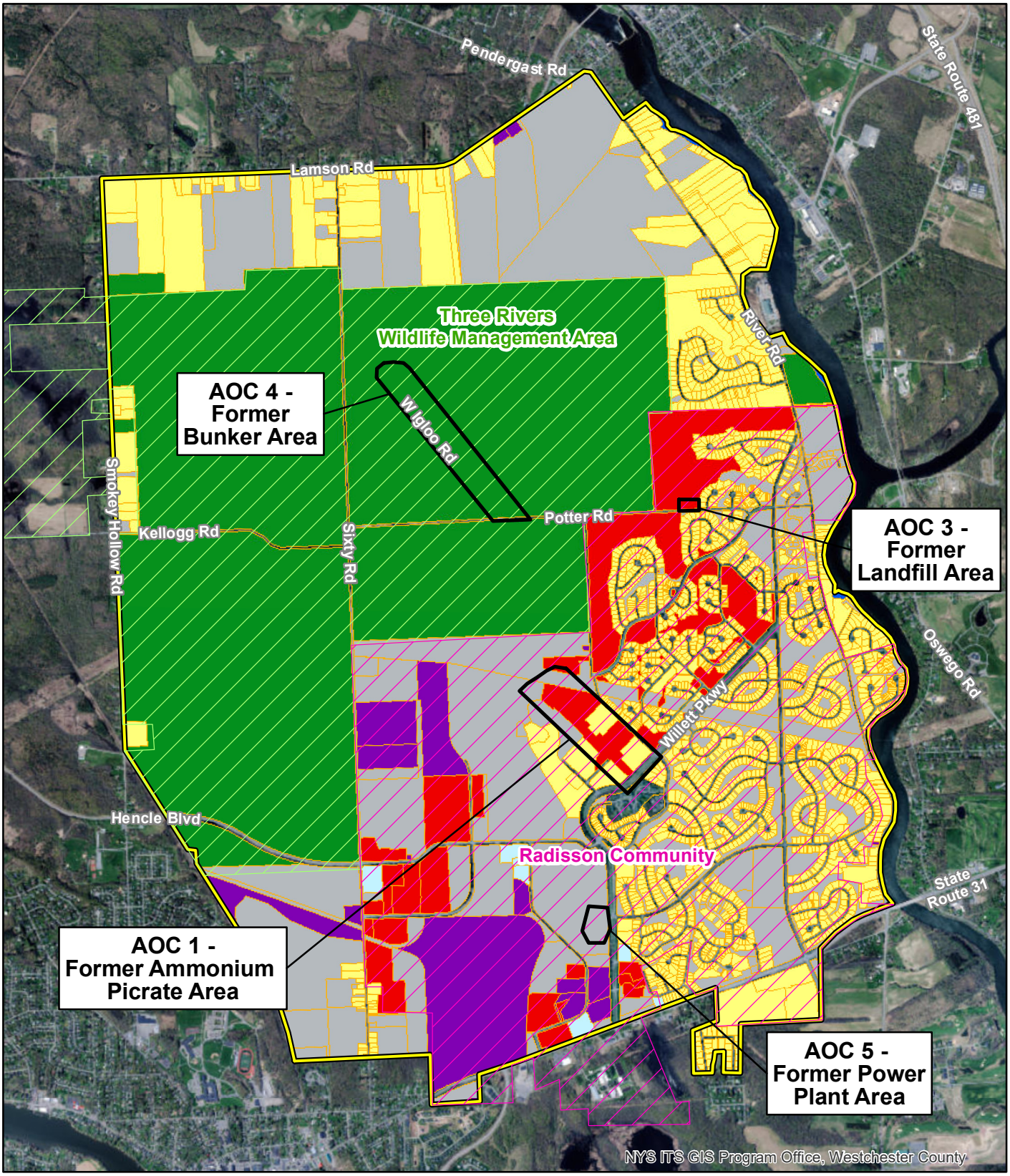
N W E S

0 75 150 225 300 600 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; NYS GIS, 2012 and 2019; Pair, 2014; RFC, undated; USACE, 2019.

*Note:
- Site feature locations are approximate.
- Existing road names are shown in white.



Legend*
Land Use (OCWA, 2019) [1]

- Parcel Boundary

Parcel Land Use

- Commercial
- Industrial/Utility
- Parks/Open Space
- Public Service
- Residential
- Vacant/Undeveloped
- Water

- Area of Concern (approx.)
- Three Rivers Wildlife Management Area
- Radisson Community
- Former NYOW

Figure 10-7: Current Land Use (Onondaga County Water Authority, 2019)

N
W E
S

0 1,500 3,000 6,000 Feet

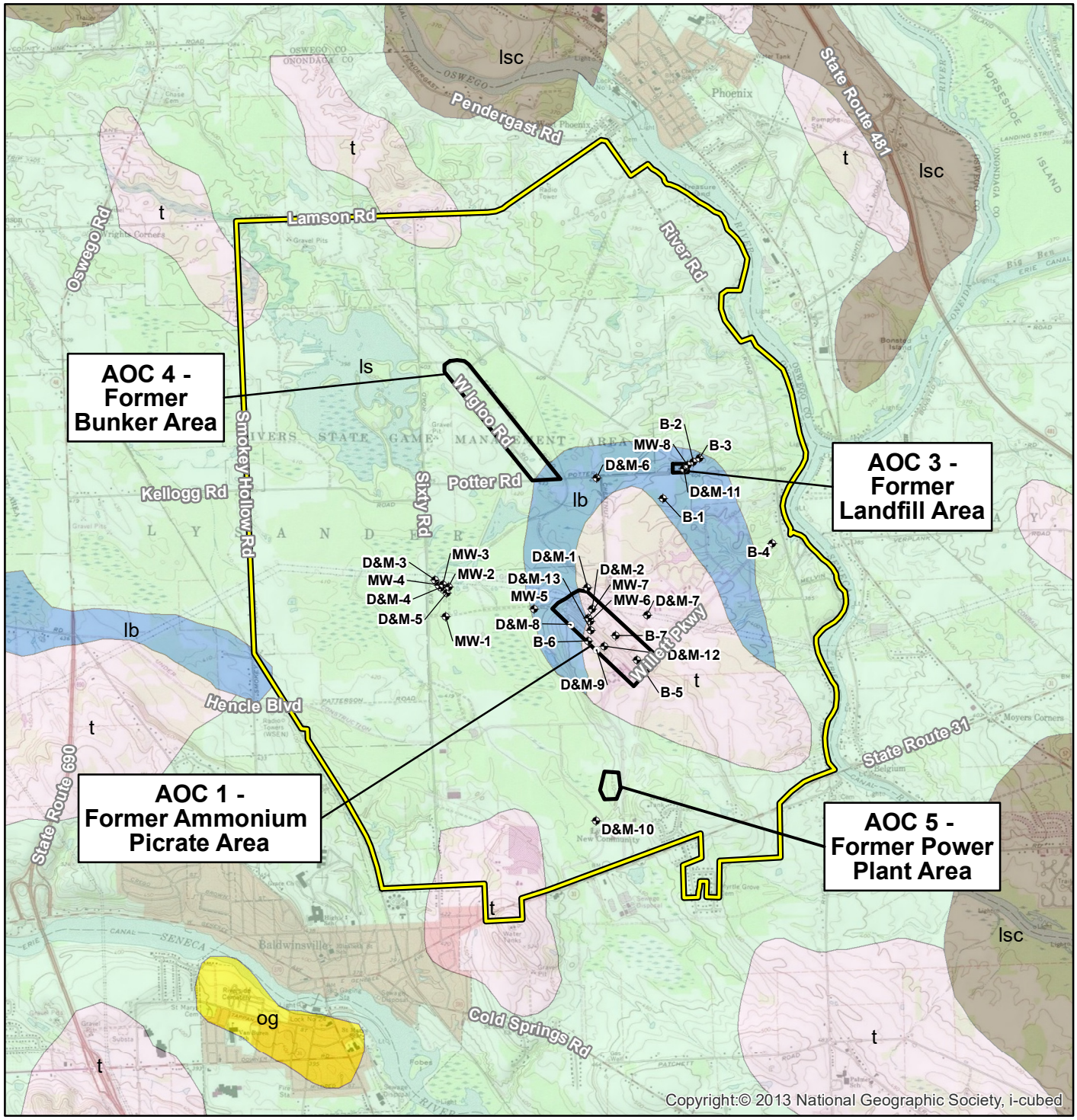
Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N. Projection: Transverse Mercator

[1] Land Use is Syracuse-Onondaga County GIS on the Web's "Parcel Land Use" (source: Onondaga County Parcel File by Onondaga County Water Authority [OCWA], last updated in 2019), downloaded as a PDF on 7/31/20 from www.fsihost.com/onondaga.

Additional Sources: USACE, 2019.

*Note: Site feature locations are approximate.



Lacustrine beach (lb) - Generally well-sorted sand and gravel, stratified, permeable and well-drained, deposited at a lake shoreline, generally non-calcareous, wave-winnowed lag gravel in isolated drumlin localities, thickness variable (2-10 meters).
Lacustrine sand (ls) - Sand deposits associated with large bodies of water, generally a near-shore deposit or near a sand source, well-sorted, stratified, generally quartz sand, thickness variable (2-20 meters).
Lacustrine silt and clay (lsc) - Generally laminated clay and silt deposited in proglacial lakes, generally calcareous, potential land instability, thickness variable (up to 50 meters).
Outwash sand and gravel (og) - Coarse to fine gravel with sand, proglacial fluvial deposition, well-rounded and stratified, generally finer texture away from ice border, thickness variable (2-20 meters).
Till (t) - Variable texture (e.g. clay, silt-clay, boulder clay), usually poorly sorted diamict, deposition beneath glacier ice, generally calcareous, relatively impermeable (loamy matrix), variable clast content - ranging from abundant well-rounded diverse lithologies in valley tills to relatively angular, more limited lithologies in upland tills, potential land instability on steep slopes, thickness variable (1-50 meters).

Legend*

Glacial Geology (Muller and Cadwell, 1986)

- lb Lacustrine beach
- ls Lacustrine sand
- lsc Lacustrine silt and clay
- og Outwash sand and gravel
- t Till

- Monitoring Well (D&M, 1981, M&E, 1990)
- Soil Boring (D&M, 1981)
- Area of Concern (approx.)
- Former NYOW

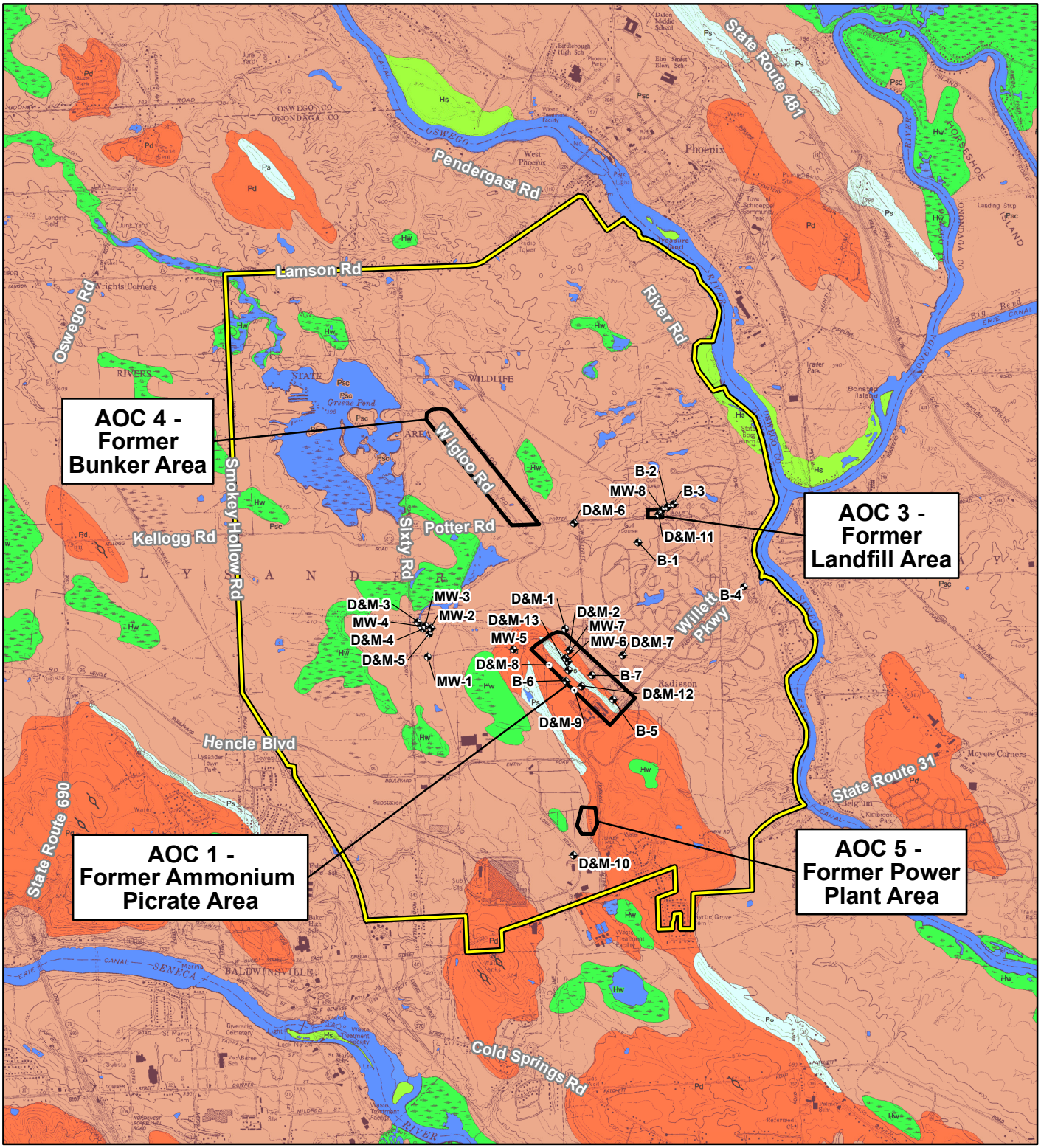
Figure 10-8: Site Glacial Geology Map (Muller and Cadwell, 1986) and Site Features

N W E S

0 0.4 0.8 1.6 Miles

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; Muller & Cadwell, 1986.
*Note: Site feature locations are approximate.



Wetland Deposit (Hw) - Peat, muck, marl, silt, clay, or sand deposited in association with wetland environments. Various sediments can be present as transitional from one facies to another.
Silt and Clay (Psc) – Stratified fine sediment consisting of silt and clay. Inferred to be deposited in deepwater settings or glacial lakes. May include rhymites and varves.
Stratified silt, sand, and gravel (Ps) – Well sorted and stratified sand, and gravel, deposited by rivers and streams. May include cobbles. Inferred as delta or fan deposits deposited in glacial lakes.
Diamicton (Pd) – An admixture of unsorted sediment ranging from clay to boulders.

Legend*
Surficial Geology (Pair, 2014)
Water
Holocene
Hw Wetland Deposit
Pleistocene
Psc Silt and clay
Ps Stratified silt, sand, and gravel
Pd Diamicton

Monitoring Well (D&M, 1981, M&E, 1990)
Soil Boring (D&M, 1981)
Area of Concern (approx.)
Former NYOW

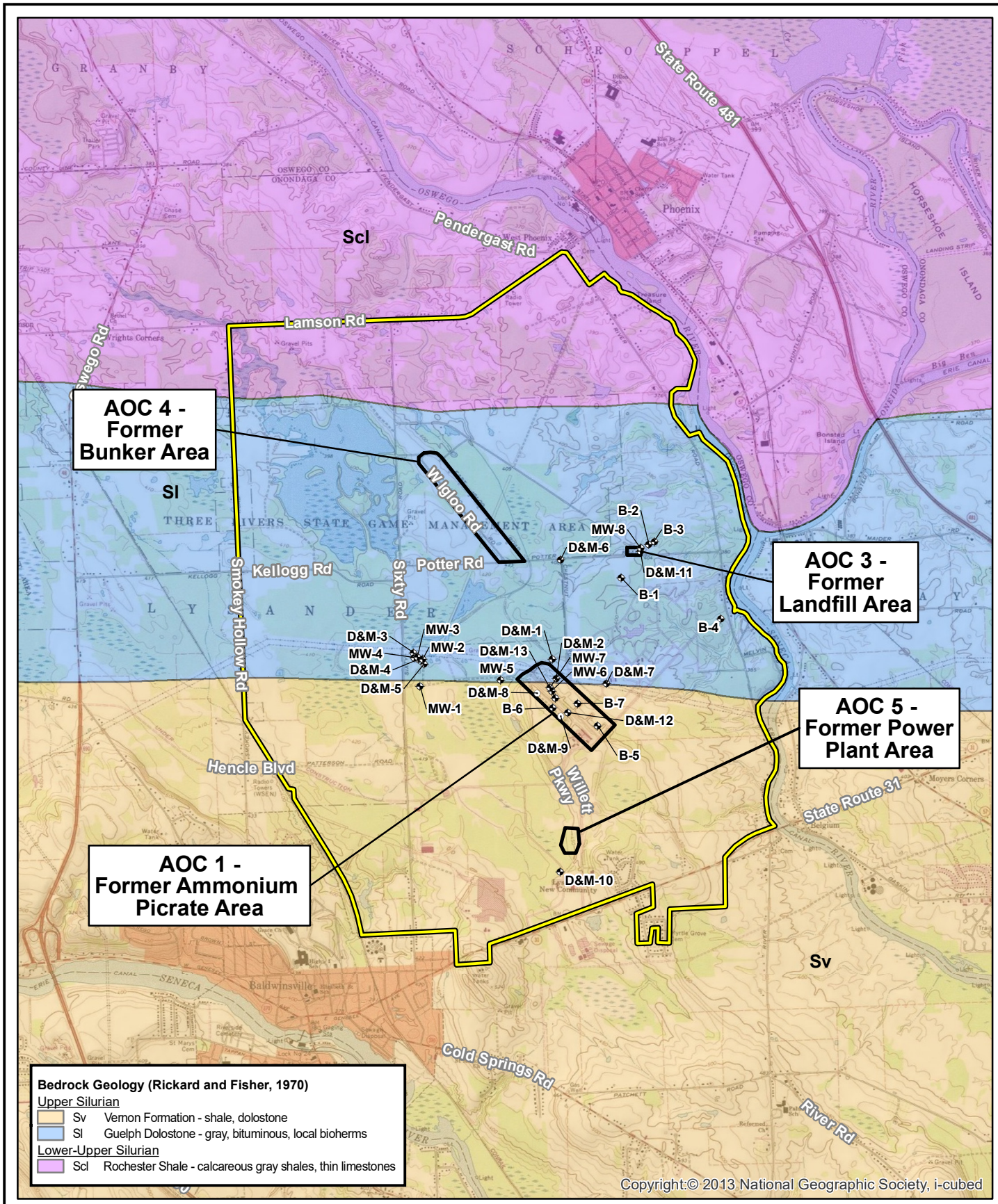
Figure 10-9: Site Surficial Geology Map (Pair, 2014) and Site Features

N
W E
S

0 0.4 0.8 1.6 *Miles*

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; Pair, 2014.
*Note: Site feature locations are approximate.



Legend*

- Monitoring Well (D&M, 1981, M&E, 1990)
- Soil Boring (D&M, 1981)
- Area of Concern (approx.)
- Former NYOW

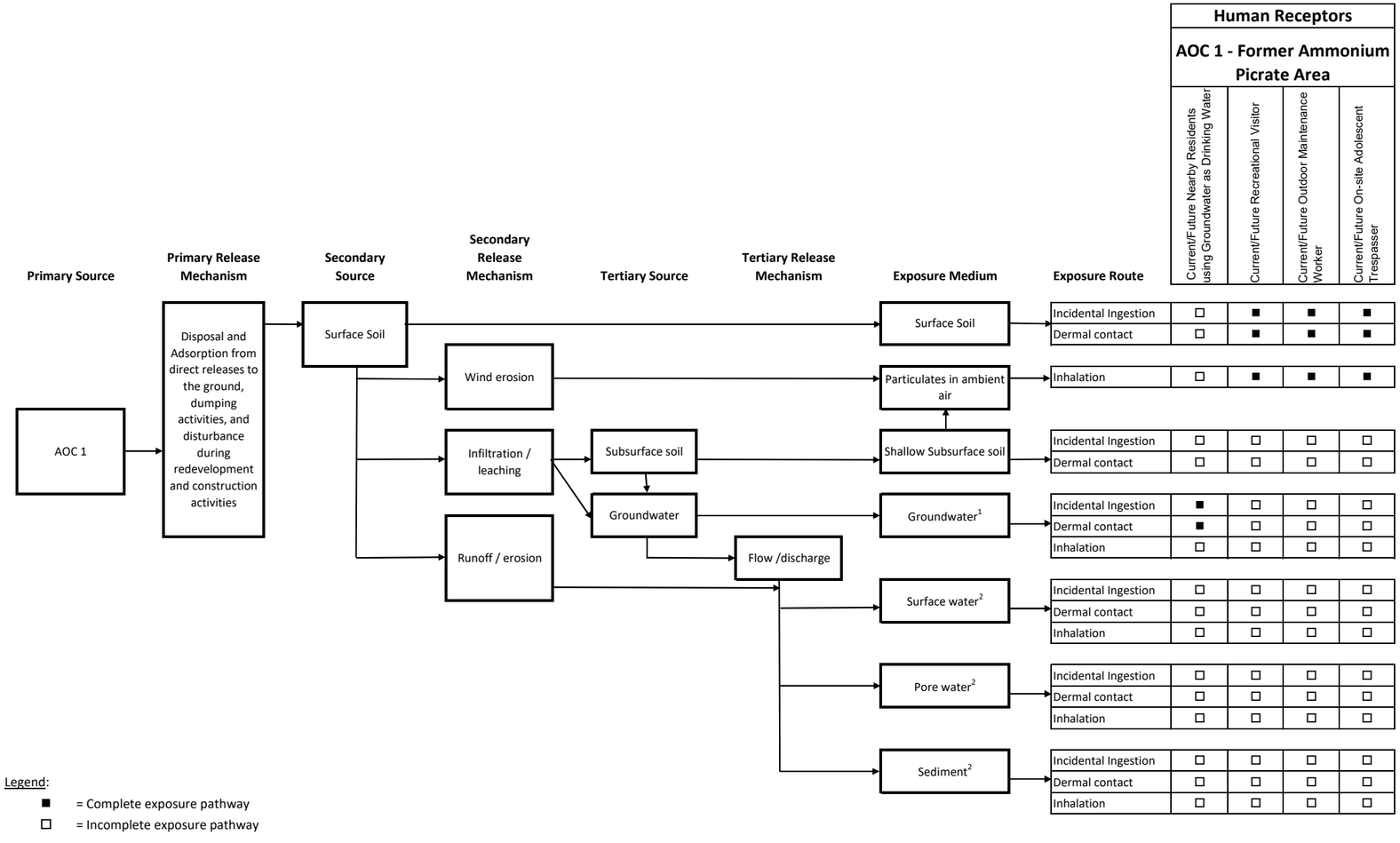
Figure 10-10: Site Bedrock Geology Map (Rickard and Fisher, 1970) and Site Features

N
W E
S

0 0.4 0.8 1.6 Miles

Scale is approximate - for illustrative purposes only.
Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; NYS GIS, 2019; Rickard and Fisher, 1970; USACE, 2019.
*Note: Site feature locations are approximate.

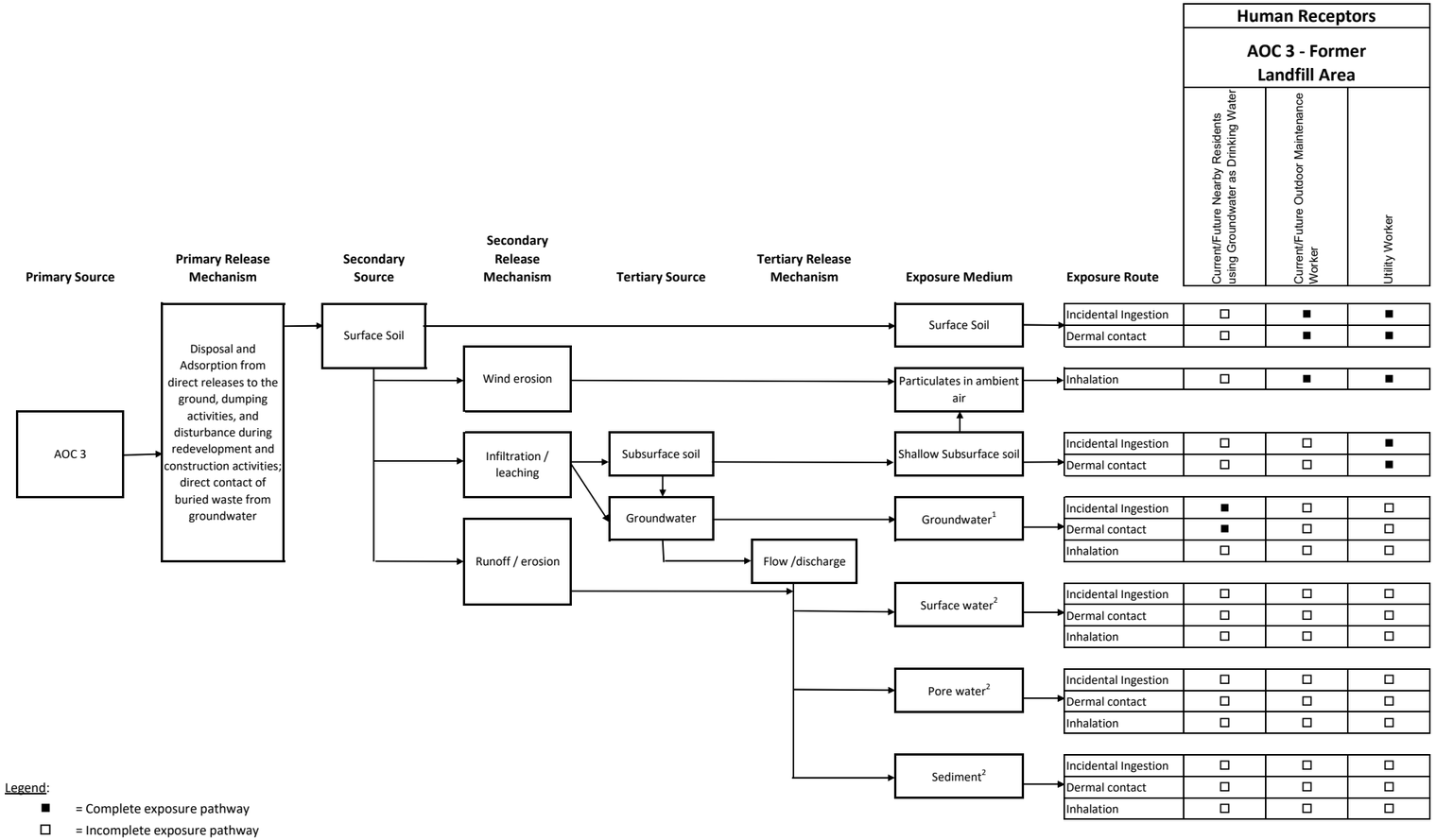
Figure 10-11a. New York Ordnance Works
AOC 1 - Former Ammonium Picrate Area
Preliminary Conceptual Site Model - Human Receptors



Legend:
■ = Complete exposure pathway
□ = Incomplete exposure pathway

Notes:
1 Groundwater on-site is not currently used as a drinking water source; therefore, groundwater exposure to on-site residents is currently considered an incomplete exposure pathway. However, some residences in the surrounding area may have private groundwater wells and no restrictions are in place preventing groundwater use in the area as drinking water. Therefore, ingestion and dermal exposures to groundwater used as a drinking water resource at each of the AOCs are considered potentially complete pathways for current/future nearby residents. There are no VOCs among contaminants of concern; therefore, groundwater exposure via vapor intrusion and/or inhalation of volatiles from groundwater used as drinking water by current or future on-site residents or nearby residents are considered incomplete pathways.
2 Surface water, pore water, and sediment may be sampled as part of a phased assessment of groundwater contamination and potential migration. No complete human exposure pathways.
3 The following analyses will be performed per medium in AOC 1 during Phase 1:
• Surface soil: TAL metals, DNCB and 2,4-DNP, picric acid; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
• Subsurface soil: TAL metals, DNCB and 2,4-DNP, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
• Groundwater (grab samples): TAL metals (field-filtered), DNCB and 2,4-DNP, and picric acid; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Figure 10-11b. New York Ordnance Works
AOC 3 - Former Landfill Area
Preliminary Conceptual Site Model - Human Receptors



Notes:

1 Groundwater on-site is not currently used as a drinking water source; therefore, groundwater exposure to on-site residents is currently considered an incomplete exposure pathway. However, some residences in the surrounding area may have private groundwater wells and no restrictions are in place preventing groundwater use in the area as drinking water. Therefore, ingestion and dermal exposures to groundwater used as a drinking water resource at each of the AOCs are considered potentially complete pathways for current/future nearby residents. There are no VOCs among contaminants of concern; therefore, groundwater exposure via vapor intrusion and/or inhalation of volatiles from groundwater used as drinking water by current or future on-site residents or nearby residents are considered incomplete pathways.

2 Surface water, pore water, and sediment may be sampled during Phase 2. No complete human exposure pathways.

3 The following analyses will be performed in AOC 3 during Phase 1:

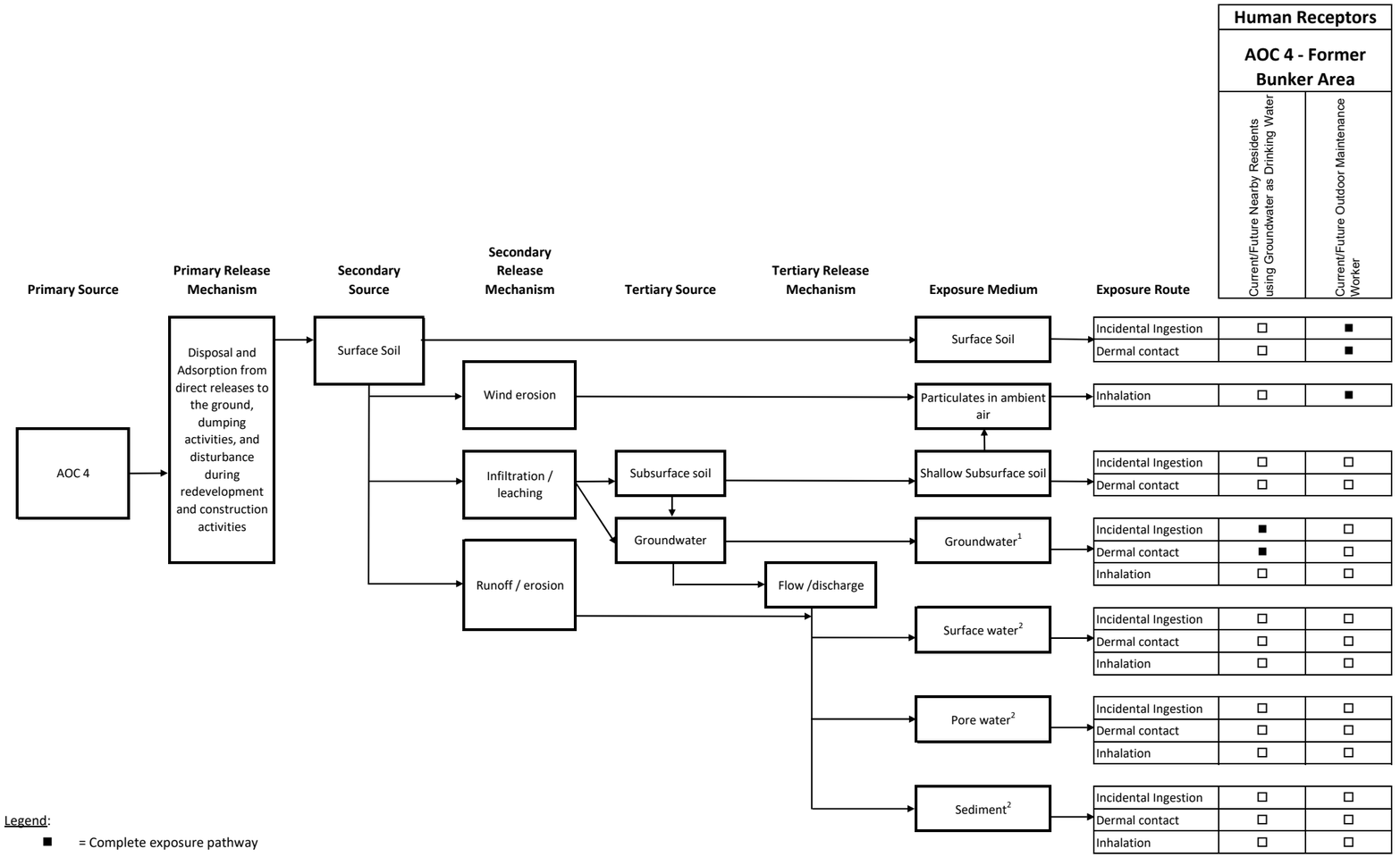
Landfill Area

- Surface soil – TAL metals, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, PCBs, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil – TAL metals, VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, PCBs, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples): TAL metals (field-filtered), VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, and PCBs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).
- VOCs in subsurface soil adjacent to the golf club house.

Open Storage Area

- Surface soil – TAL metals, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil – TAL metals, VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples) – TAL metals (field-filtered), VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, and picric acid; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Figure 10-11c. New York Ordnance Works
AOC 4 - Former Bunker Area
Preliminary Conceptual Site Model - Human Receptors



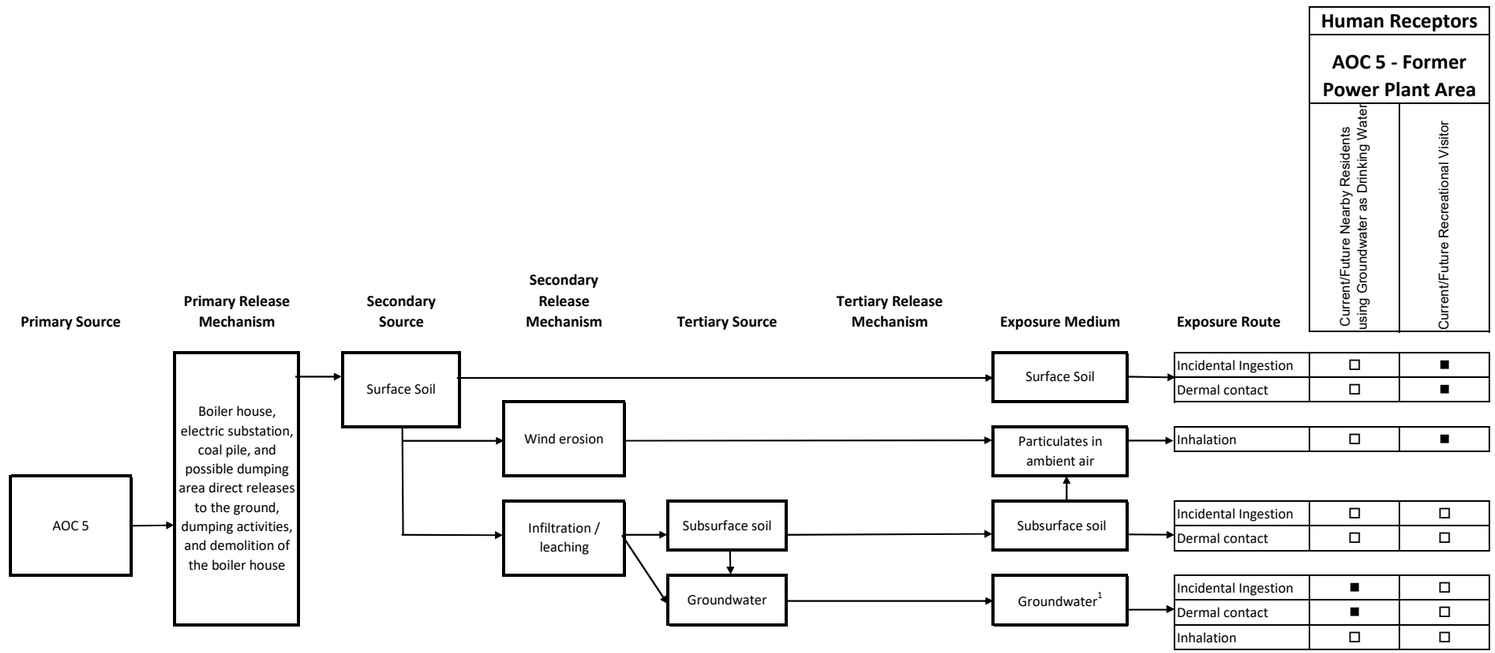
Legend:
- ■ = Complete exposure pathway
- ☐ = Incomplete exposure pathway

Notes:
1 Groundwater on-site is not currently used as a drinking water source; therefore, groundwater exposure to on-site residents is currently considered an incomplete exposure pathway. However, some residences in the surrounding area may have private groundwater wells and no restrictions are in place preventing groundwater use in the area as drinking water. Therefore, ingestion and dermal exposures to groundwater used as a drinking water resource at each of the AOCs are considered potentially complete pathways for current/future nearby residents. There are no VOCs among contaminants of concern; therefore, groundwater exposure via vapor intrusion and/or inhalation of volatiles from groundwater used as drinking water by current or future on-site residents or nearby residents are considered incomplete pathways.

2 Surface water, pore water, and sediment may be sampled as part of a phased assessment of groundwater contamination and potential migration. No complete human exposure pathways.

3 The following analyses will be performed in AOC 4 during Phase 1:
- Surface soil: DNCB and 2,4-DNP, picric acid.
- Subsurface soil: DNCB and 2,4-DNP, picric acid.
- Groundwater (grab samples): DNCB and 2,4-DNP, and picric acid; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Figure 10-11d. New York Ordnance Works
AOC 5 - Former Power Plant Area
Preliminary Conceptual Site Model - Human Receptors



Legend:

- ■ = Complete exposure pathway
- □ = Incomplete exposure pathway

Notes:

1 Groundwater on-site is not currently used as a drinking water source; therefore, groundwater exposure to on-site residents is currently considered an incomplete exposure pathway. However, some residences in the surrounding area may have private groundwater wells and no restrictions are in place preventing groundwater use in the area as drinking water. Therefore, ingestion and dermal exposures to groundwater used as a drinking water resource at each of the AOCs are considered potentially complete pathways for current/future nearby residents. There are no VOCs among contaminants of concern; therefore, groundwater exposure via vapor intrusion and/or inhalation of volatiles from groundwater used as drinking water by current or future on-site residents or nearby residents are considered incomplete pathways.

2 The following analyses will be performed at AOC 5 during Phase 1:

Boiler House
- Surface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

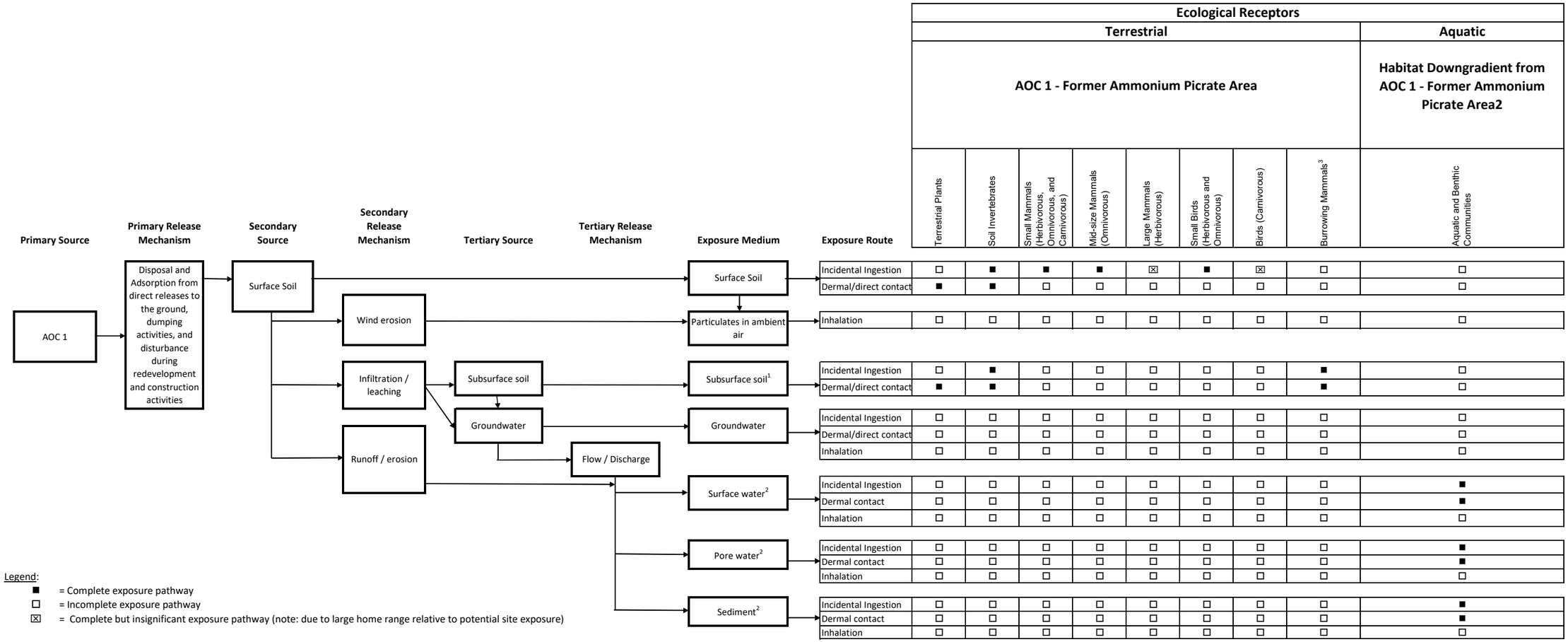
Coal Storage Area
- Surface soil: Coal Ash, TAL metals, PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Sub-Station
- Surface soil: TAL metals, SVOCs, PAHs, and PCBs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Possible Dump
- Surface soil: TAL metals, SVOCs, PAHs, and PCBs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, VOCs, SVOCs, PAHs, and PCBs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples): TAL metals (field-filtered), VOCs, SVOCs, PAHs, and PCBs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Coal Aggregate Bins Area
- Surface soil: Coal Ash, TAL metals, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Groundwater (grab samples): TAL metals (field-filtered), SVOCs, and PAHs; and water quality field parameters (i.e., temperature, pH, conductivity, DO, ORP, and turbidity).

Figure 10-12a. New York Ordnance Works
AOC 1 - Former Ammonium Picrate Area
Preliminary Conceptual Site Model - Ecological Receptors

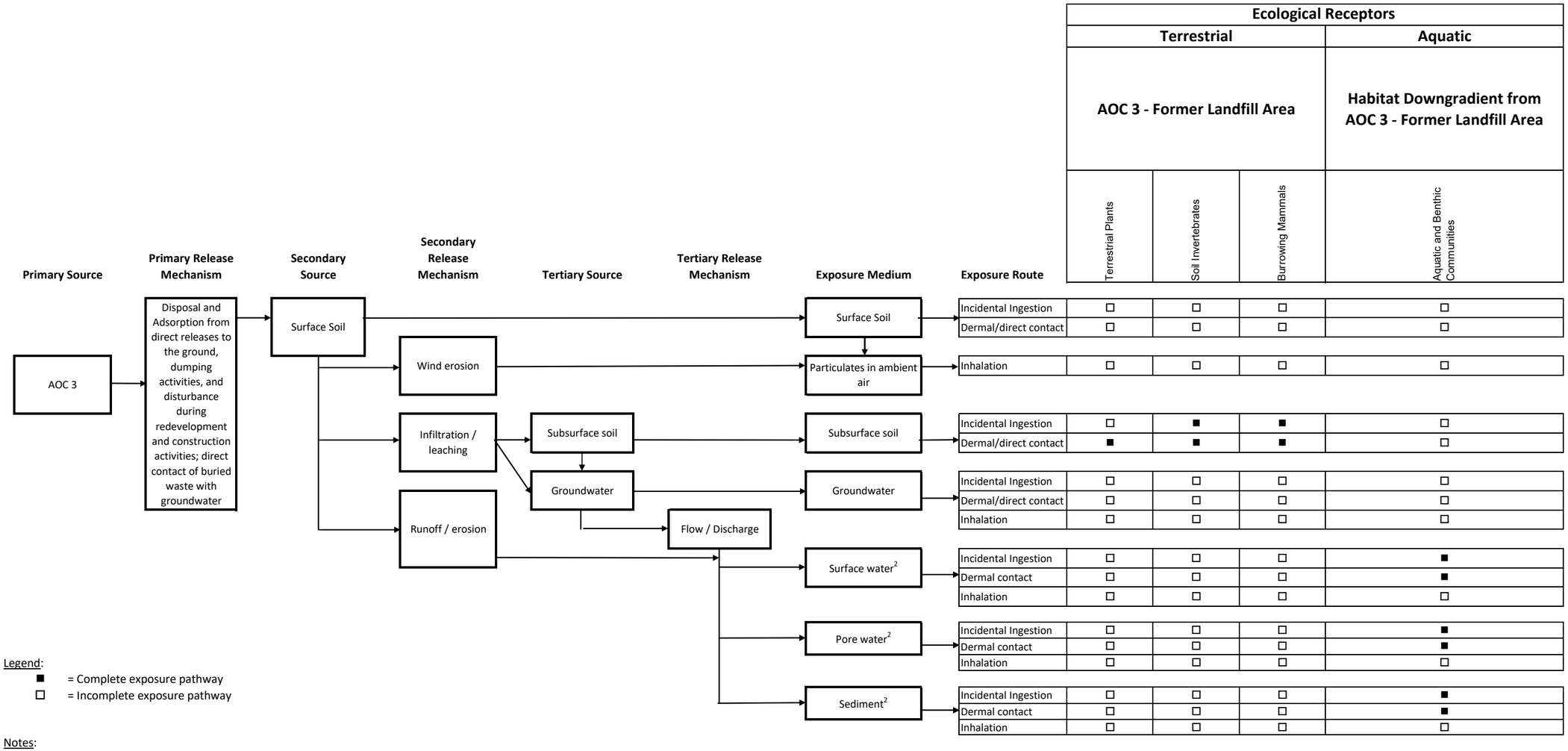


Exposure Route	Terrestrial Plants	Soil Invertebrates	Small Mammals (Herbivorous, Omnivorous, and Carnivorous)	Mid-size Mammals (Omnivorous)	Large Mammals (Herbivorous)	Small Birds (Herbivorous and Omnivorous)	Birds (Carnivorous)	Burrowing Mammals[3]	Aquatic and Benthic Communities
	Ecological Receptors								
	Terrestrial								**Aquatic**
	AOC 1 - Former Ammonium Picrate Area								**Habitat Downgradient from AOC 1 - Former Ammonium Picrate Area2**
Surface Soil: Incidental Ingestion	☐	■	■	■	☒	■	☒	☐	☐
Surface Soil: Dermal/direct contact	■	■	☐	☐	☐	☐	☐	☐	☐
Particulates in ambient air: Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Subsurface soil[1]: Incidental Ingestion	☐	■	☐	☐	☐	☐	☐	■	☐
Subsurface soil[1]: Dermal/direct contact	■	■	☐	☐	☐	☐	☐	■	☐
Groundwater: Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	☐
Groundwater: Dermal/direct contact	☐	☐	☐	☐	☐	☐	☐	☐	☐
Groundwater: Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Surface water[2]: Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
Surface water[2]: Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
Surface water[2]: Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Pore water[2]: Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
Pore water[2]: Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
Pore water[2]: Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Sediment[2]: Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
Sediment[2]: Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
Sediment[2]: Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐

Legend:
- ■ = Complete exposure pathway
- ☐ = Incomplete exposure pathway
- ☒ = Complete but insignificant exposure pathway (note: due to large home range relative to potential site exposure)

Notes:
1. Chemical concentrations in discrete subsurface soil samples will be compared to appropriate ecological benchmarks as part of the site characterization process and will be used to determine the need for additional sampling and subsequent risk evaluation.
2. Surface water, pore water, and sediment may be sampled as part of a phased assessment of groundwater contamination and will only be considered if DOD-related COPECs identified in groundwater are potentially discharging to nearby wetland areas.
3. Burrowing mammals are included in the "small mammal" receptor category when considering surface soil exposure.
4. The following analyses will be performed per medium in AOC 1 during Phase 1:
   - Surface soil: TAL metals, DNCB and 2,4-DNP, picric acid; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
   - Subsurface soil: TAL metals, DNCB and 2,4-DNP, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

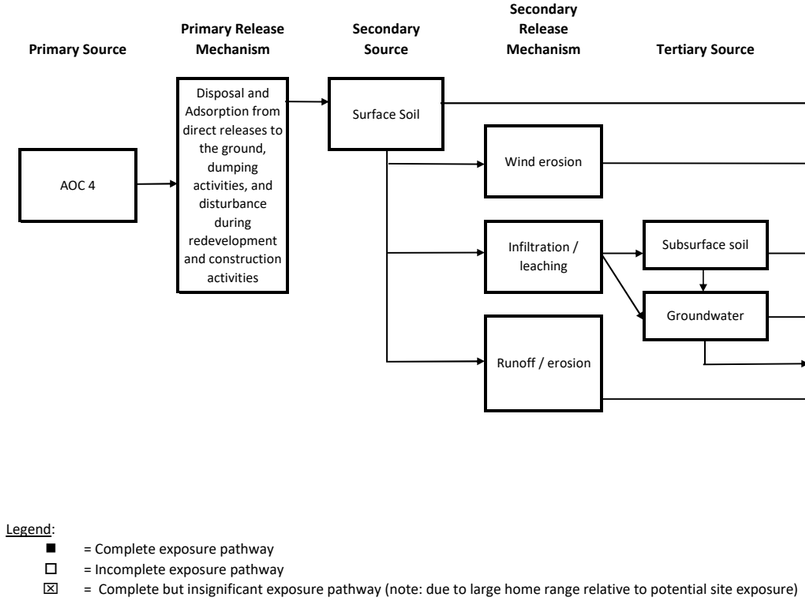
Figure 10-12b. New York Ordnance Works
AOC 3 - Former Landfill Area
Preliminary Conceptual Site Model - Ecological Receptors



Legend:
- ■ = Complete exposure pathway
- □ = Incomplete exposure pathway

Notes:
1 Chemical concentrations in discrete subsurface soil samples will be compared to appropriate ecological benchmarks as part of the site characterization process and will be used to determine the need for additional sampling and subsequent risk evaluation.
2 Surface water, pore water, and sediment may be sampled as part of a phased assessment of soil and groundwater contamination and will only be considered if DOD-related COPECs identified in soil and groundwater are potentially affecting the unnamed stream north of the landfill.
3 The following analyses will be performed in AOC 3 during Phase 1:
Landfill Area
• Surface soil – TAL metals, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, PCBs, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
• Subsurface soil – TAL metals, VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, PCBs, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
Open Storage Area
• Surface soil – TAL metals, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
• Subsurface soil – TAL metals, VOCs, SVOCs (including DNCB and 2,4-DNP), PAHs, picric acid, and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

Figure 10-12c. New York Ordnance Works
AOC 4 - Former Bunker Area
Preliminary Conceptual Site Model - Ecological Receptors



		Ecological Receptors								
		Terrestrial								Aquatic
		AOC 4 - Former Bunker Area								Habitat Downgradient from AOC 4 - Former Bunker Area2
Exposure Medium	Exposure Route	Terrestrial Plants	Soil Invertebrates	Small Mammals (Herbivorous, Omnivorous, and Carnivorous)	Mid-size Mammals (Omnivorous)	Large Mammals (Herbivorous)	Small Birds (Herbivorous and Omnivorous)	Birds (Carnivorous)	Burrowing Mammals[3]	Aquatic and Benthic Communities
Surface Soil	Incidental Ingestion	☐	■	■	■	☒	■	☒	☐	☐
	Dermal/direct contact	■	■	☐	☐	☐	☐	☐	☐	☐
Particulates in ambient air	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Subsurface soil[1]	Incidental Ingestion	☐	■	☐	☐	☐	☐	☐	■	☐
	Dermal/direct contact	■	■	☐	☐	☐	☐	☐	■	☐
Groundwater	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	☐
	Dermal/direct contact	☐	☐	☐	☐	☐	☐	☐	☐	☐
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Surface water[2]	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
	Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Pore water[2]	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
	Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Sediment[2]	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
	Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐

Legend:
- ■ = Complete exposure pathway
- ☐ = Incomplete exposure pathway
- ☒ = Complete but insignificant exposure pathway (note: due to large home range relative to potential site exposure)

Notes:
1 Chemical concentrations in discrete subsurface soil samples will be compared to appropriate ecological benchmarks as part of the site characterization process and will be used to determine the need for additional sampling and subsequent risk evaluation.
2 Surface water, pore water, and sediment may be sampled as part of a phased assessment of groundwater contamination and will only be considered if DOD-related COPECs identified in groundwater are potentially discharging to nearby wetland areas.
3 Burrowing mammals are included in the "small mammal" receptor category when considering surface soil exposure.
4 The following analyses will be performed in AOC 4 during Phase 1:
- Surface soil: DNCB and 2,4-DNP, picric acid.
- Subsurface soil: DNCB and 2,4-DNP, picric acid.

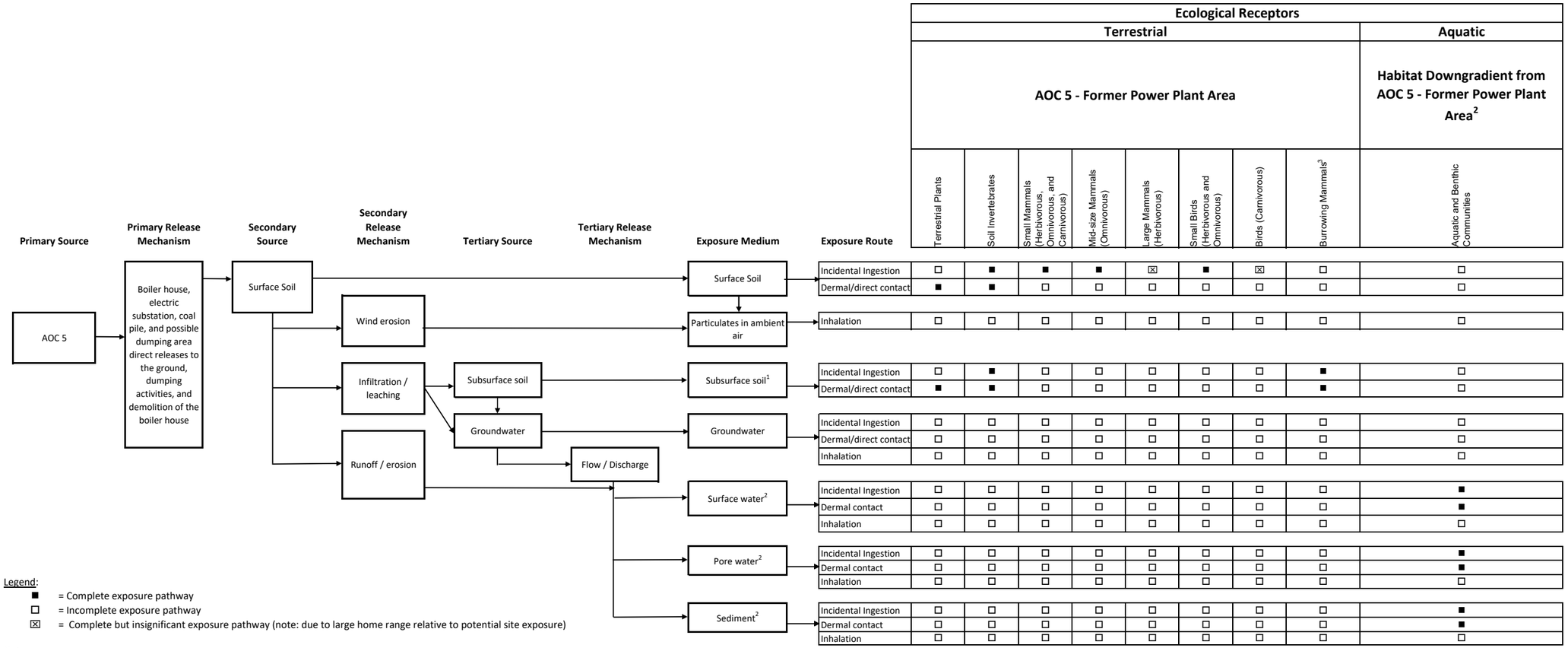


Figure 10-12d. New York Ordnance Works
AOC 5 - Former Power Plant Area
Preliminary Conceptual Site Model - Ecological Receptors

		Ecological Receptors								
		Terrestrial								Aquatic
		AOC 5 - Former Power Plant Area								Habitat Downgradient from AOC 5 - Former Power Plant Area^2
Exposure Medium	Exposure Route	Terrestrial Plants	Soil Invertebrates	Small Mammals (Herbivorous, Omnivorous, and Carnivorous)	Mid-size Mammals (Omnivorous)	Large Mammals (Herbivorous)	Small Birds (Herbivorous and Omnivorous)	Birds (Carnivorous)	Burrowing Mammals^3	Aquatic and Benthic Communities
Surface Soil	Incidental Ingestion	☐	■	■	■	☒	■	☒	☐	☐
	Dermal/direct contact	■	■	☐	☐	☐	☐	☐	☐	☐
Particulates in ambient air	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Subsurface soil^1	Incidental Ingestion	☐	■	☐	☐	☐	☐	☐	■	☐
	Dermal/direct contact	■	■	☐	☐	☐	☐	☐	■	☐
Groundwater	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	☐
	Dermal/direct contact	☐	☐	☐	☐	☐	☐	☐	☐	☐
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Surface water^2	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
	Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Pore water^2	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
	Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐
Sediment^2	Incidental Ingestion	☐	☐	☐	☐	☐	☐	☐	☐	■
	Dermal contact	☐	☐	☐	☐	☐	☐	☐	☐	■
	Inhalation	☐	☐	☐	☐	☐	☐	☐	☐	☐

Legend:

■ = Complete exposure pathway
☐ = Incomplete exposure pathway
☒ = Complete but insignificant exposure pathway (note: due to large home range relative to potential site exposure)

Notes:

1 Chemical concentrations in discrete subsurface soil samples will be compared to appropriate ecological benchmarks as part of the site characterization process and will be used to determine the need for additional sampling and subsequent risk evaluation.
2 Surface water, pore water, and sediment may be sampled as part of a phased assessment of groundwater contamination and will only be considered if DOD-related COPECs identified in groundwater are potentially discharging to nearby wetland areas.
3 Burrowing mammals are included in the "small mammal" receptor category when considering surface soil exposure.
4 The following analyses will be performed at AOC 5 during Phase 1:

Boiler House

- Surface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

Coal Storage Area

- Surface soil: Coal Ash, TAL metals and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

Sub-Station

- Surface soil: TAL metals, SVOCs, PAHs, and PCBs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

Possible Dump

- Surface soil: TAL metals, SVOCs, PAHs, and PCBs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, VOCs, SVOCs, PAHs, and PCBs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

Coal Aggregate Bins Area

- Surface soil: Coal Ash, TAL metals, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.
- Subsurface soil: TAL metals, SVOCs, and PAHs; and a subset for TOC, grain size distribution (including hydrometer analysis), and CEC.

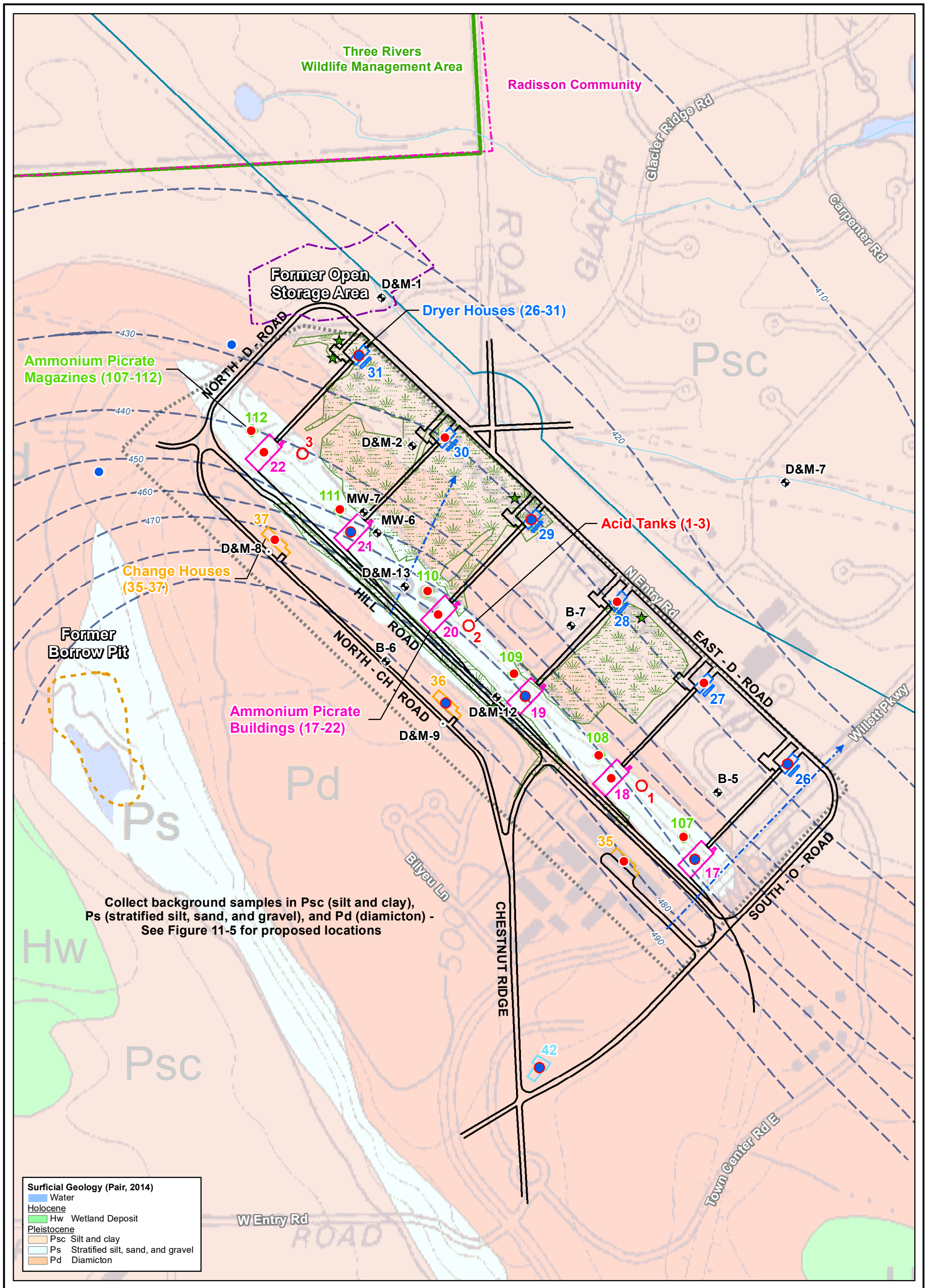


Figure 11-1: Phase 1 Former NYOW - AOC 1 - Former Ammonium Picrate Area Proposed Discrete Sampling Locations with Surficial Geology (Pair, 2014)

Legend*

- Proposed Grab Groundwater Sample and co-located Surface/Subsurface Soil Sample
- Proposed Discrete Surface/ Subsurface Soil Sample
- Proposed Grab Groundwater Sample
- Groundwater Contour (D&M, 1981)
- Inferred Groundwater Flow Direction
- Pre-Existing Monitoring Well (D&M, 1981, M&E, 1990)
- Historic Soil Boring (D&M, 1981)
- Area of Concern (approx.)
- Radisson Community
- Three Rivers Wildlife Management Area
- Stream
- Former Wastewater Ditch
- Candidate Vernal Pool (May 2020)[1]
- Wetland (delineated May 2020)[1]
- Former Borrow Pit
- Former Open Storage Area
- Historical Site Feature (approx.)
- Former Acid Tank (Not FUDS-Eligible)
- Ammonium Picrate Building
- Ammonium Picrate Magazine
- Change House
- Dryer House
- Laboratory Building

N W E S

0 100 200 300 400 800 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; Pair, 2014; RFC, undated; USGS; WRS, 2020.

*Note:
- Ground surface contours are shown on Figure 10-3.
- Site feature locations are approximate.
- Existing road names are shown in white while historic road names are shown in black.

[1] Wetlands and Vernal Pools within the AOC were delineated in May 2020.

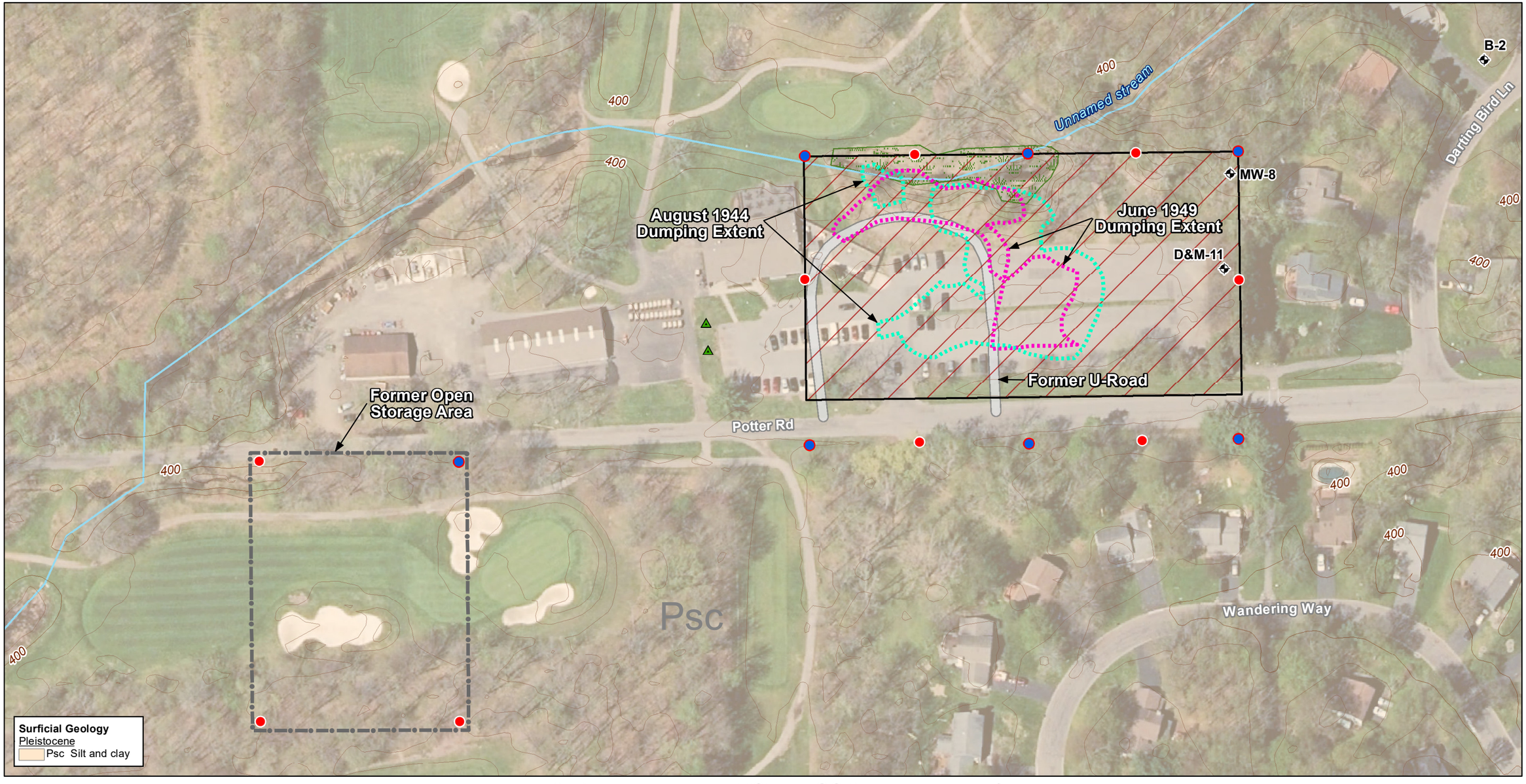


Figure 11-2: Phase 1 Former NYOW - AOC 3 - Former Landfill Area
Proposed Discrete Sampling Locations with Surficial Geology (Pair, 2014)

Legend*

- Proposed Grab Groundwater Sample and co-located Surface/Subsurface Soil Sample
- Proposed Discrete Surface/Subsurface Soil Sample
- Proposed Vapor Intrusion Point
- Pre-Existing Monitoring Well (D&M, 1981, M&E, 1990)
- Former Landfill & Area of Concern (approx.)
- Proposed Geophysical Survey Area
- Stream
- Wetland (delineated May 2020)[1]
- 10' Contour
- 2' Contour

Historical Site Features (USACE)

- Former U-Road
- August 1944 Dumping Extent
- June 1949 Dumping Extent
- Former Open Storage Area

N W E S

0 50 100 150 200 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Metcalf & Eddy, 1990; NYS GIS, 2012 and 2019; Pair, 2014; USACE, 2019; USGS, 2019; WRS, 2020.

*Note: Site feature locations are approximate.
[1] Wetlands within the AOC were delineated in May 2020.

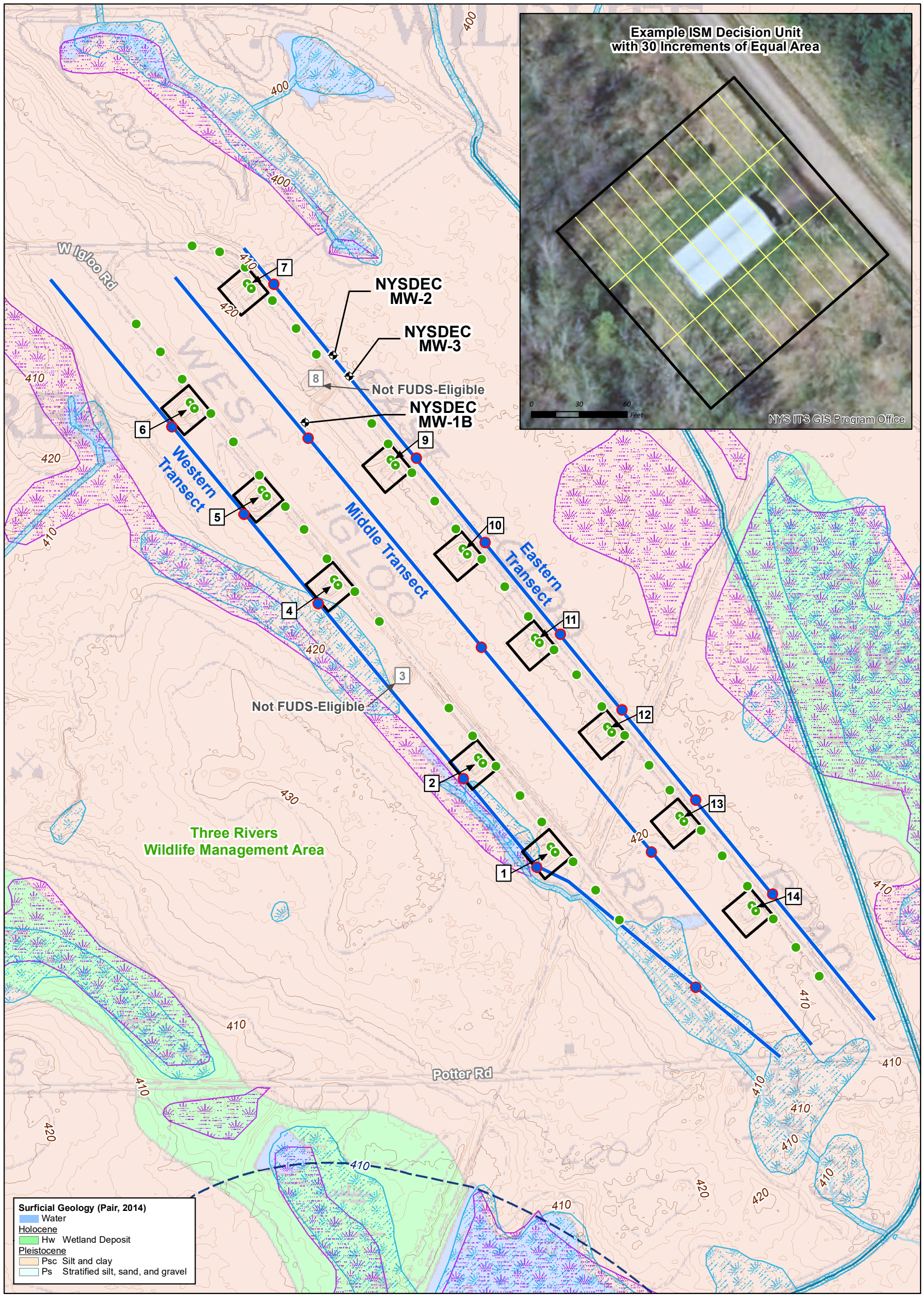


Figure 11-3: Phase 1 Former NYOW - AOC 4 - Former Bunker Area
Proposed Discrete and ISM Sampling Locations with Surficial Geology (Pair, 2014)

Legend*

- Proposed Discrete Subsurface Soil Sample at Outlet
- Proposed Discrete Subsurface Soil Sample in Swale
- Proposed Grab Groundwater Sample and co-located Surface/Subsurface Soil Sample
- Proposed DPT Transect
- Proposed ISM Decision Unit (0.5 acre)
- Pre-Existing Monitoring Well (NYSDEC, 1990)
- 5 Existing Bunker
- 3 Former Bunker
- Former Wastewater Ditch
- Stream
- Groundwater Contour (D&M, 1981)
- NYS Freshwater Wetland[1]
- USFWS NWI Wetland
- 10' Contour
- 2' Contour

N
W E
S

0 200 400 600 800 Feet

Scale is apporximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; Pair, 2014; NYSDEC, 1990 and 2010; NYS GIS, 2012 and 2019; RFC, undated; USFWS, 1981; USGS, 2019.
*Note: Site feature locations are approximate.
[1] The "check zone" is an area around mapped NYS freshwater wetlands in which the actual wetland may occur. Check zones (not shown) are 200 ft wide for linear wetlands and 500 ft wide for wetland polygons.

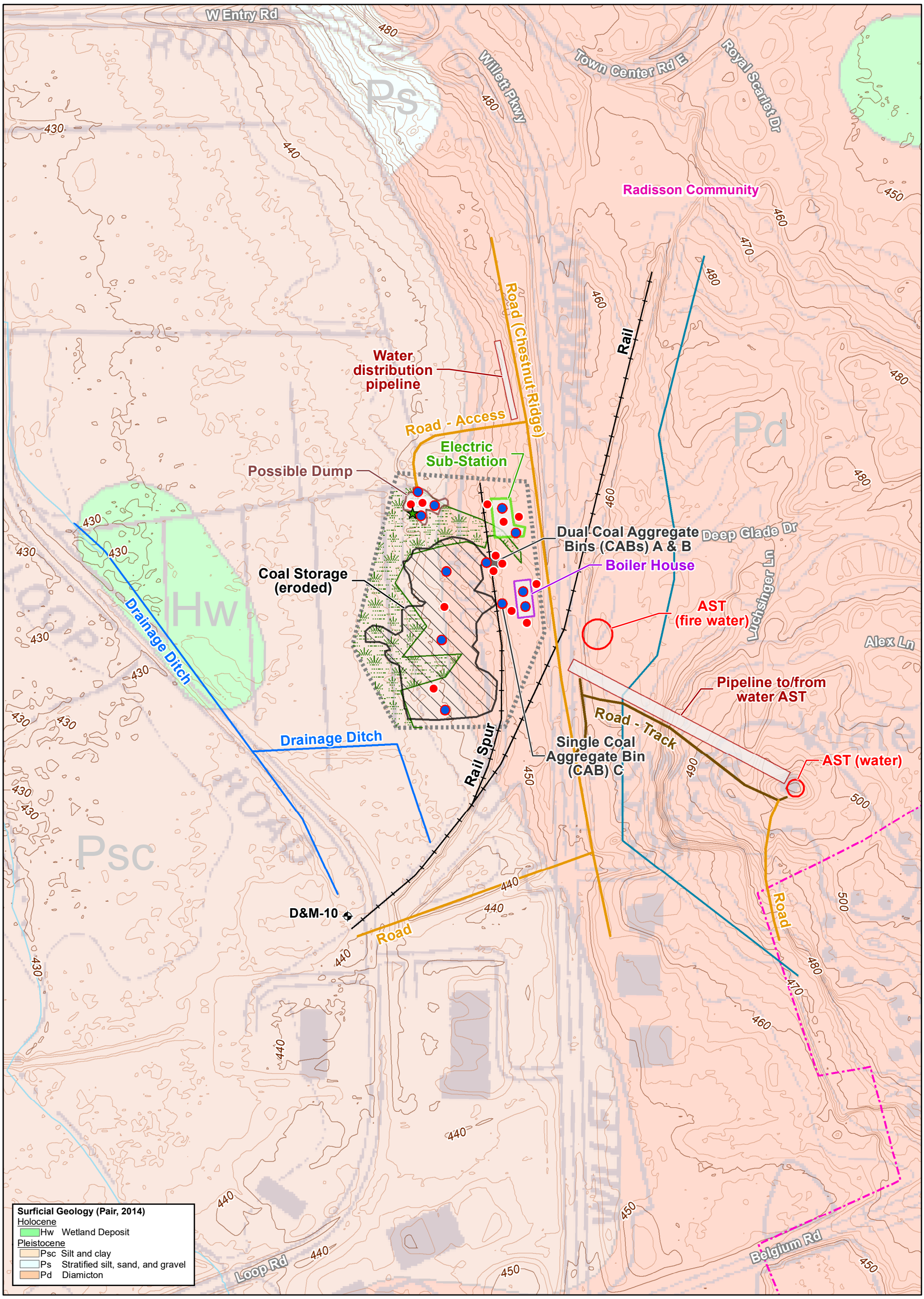


Figure 11-4: Phase 1 Former NYOW - AOC 5 - Former Power Plant Area Proposed Discrete Sampling Locations with Surficial Geology (Pair, 2014)

Legend*

- Proposed Discrete Surface/Subsurface Soil Sample
- Proposed Grab Groundwater Sample and co-located Surface/Subsurface Soil Sample
- Pre-Existing Monitoring Well (D&M, 1981, M&E, 1990)
- Former Wastewater Ditch (D&M, 1981)
- Stream
- Area of Concern (approx.)
- Radisson Community
- 10' Contour
- 2' Contour
- Candidate Vernal Pool (May 2020)[1]
- Wetland (delineated May 2020)[1]

Historic Site Features (USACE)

- AST
- Coal Storage (Eroded)
- Boiler House
- Electric Substation
- Possible Dump
- Pipeline
- Coal Aggregate Bins (CABs) (Approx.)
- Railroad
- Road
- Road - Track
- Drainage Ditch

N W E S

0 75 150 225 300 600 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: Dames & Moore, 1981; NYS GIS, 2012 and 2019; Pair, 2014; RFC, undated; USACE, 2019; USGS, 2019; WRS, 2020.

*Note:
- Site feature locations are approximate.
- Existing road names are shown in white.
[1] Wetlands and Vernal Pools within the AOC were delineated in May 2020.

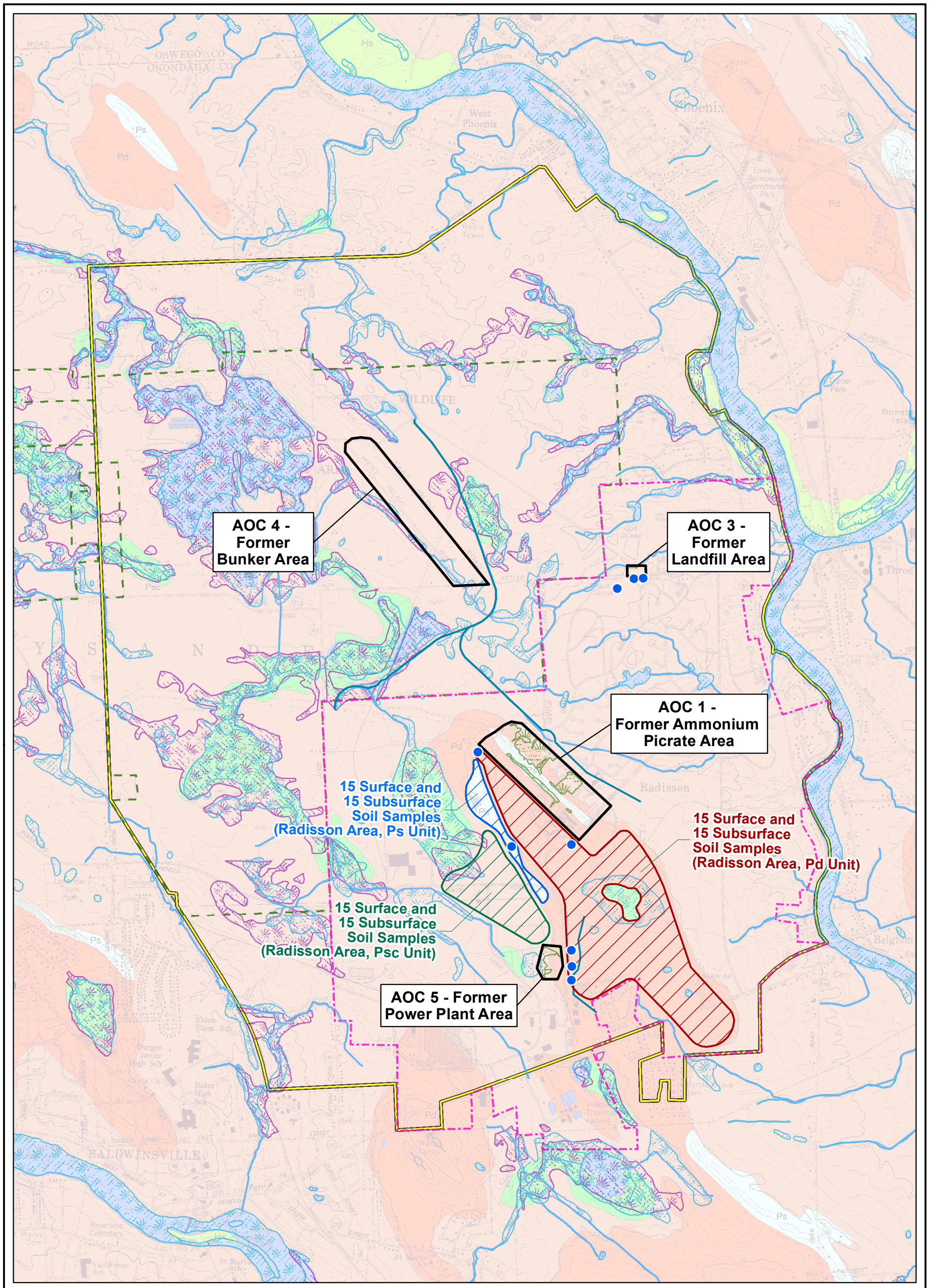


Figure 11-5: Phase 1 Former NYOW - Background - Proposed Discrete Sampling Locations with Surficial Geology (Pair, 2014)

Legend*

- Area of Concern (approx.)
- Former Wastewater Ditch
- Stream
- Former NYOW
- Wetland (delineated May 2020)[1]
- NYS Freshwater Wetland[2]
- USFWS NWI Wetland
- Radisson Community
- Three Rivers Wildlife Management Area (WMA)

Proposed Background

- Proposed Upgradient Grab Groundwater Sample
- Radisson Area, Pd
- Radisson Area, Ps
- Radisson Area, Psc

Surficial Geology (Pair, 2014)

- Water
- Holocene
  - Hw Wetland Deposit
- Pleistocene
  - Psc Silt and clay
  - Ps Stratified silt, sand, and gravel
  - Pd Diamicton

Note: Exact sampling locations will be based on actual field conditions, AOC groundwater flow direction investigations, and the professional judgment of the Field Team Leader.

N W E S

0 550 1,100 2,200 3,300 4,400 Feet

Scale is approximate - for illustrative purposes only.

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Additional Sources: NYSDEC, 2010; RFC, undated; Pair, 2014; USFWS, 1981; USGS, 2019; WRS, 2020.
*Note: Site feature locations are approximate.
[1] Wetlands within AOCs 1, 3, and 5 were delineated in May 2020.
[2] The "check zone" is an area around mapped NYS freshwater wetlands in which the actual wetland may occur. Check zones (not shown) are 200 ft wide for linear wetlands and 500 ft wide for wetland polygons.

TABLES

Table 10-1. D&M Sampling Results - Groundwater

Sample Location:	Current USEPA Tapwater	AOC 1 - Ammonium Picrate Area							
		B-5	B-6	B-7	D&M-1		D&M-2		
Sample Date:	RSLs(1)	2/16/1981	2/17/1981	2/16/1981	2/18/1981	4/8/1981	2/18/1981	3/31/1981	4/8/1981
Groundwater Chemistry (mg/L)									
Ammonia	N/A	--	--	--	--	--	--	--	--
Total Acid	N/A	--	--	--	--	--	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	--	--	--	0.0098	ND	ND	ND	ND
2,4- and 2,6-Dinitrophenol (DNP)	0.039	--	--	--	ND	ND	ND	ND	ND
2,4,6-Trinitrophenol (TNP, picric acid)	0.040	ND	ND	ND	ND	ND	ND	ND	ND

NOTES:

-- = Not Analyzed

N/A = Not Available

ND = Not Detected (detection limits for groundwater parameters were not provided in D&M Report)

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. For non-carcinogens, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

Screening Versus Current USEPA Criteria (1):

Orange shaded values represent exceedance of Tapwater RSLs.

Table 10-1. D&M Sampling Results - Groundwater (Continued)

Sample Location:	Current USEPA Tapwater	AOC 1 - Ammonium Picrate Area						
		D&M-7			D&M-8	D&M-9	D&M-12	
Sample Date:	RSLs(1)	2/18/1981	3/31/1981	4/8/1981	Dry	Dry	3/26/1981	4/14/1981
Groundwater Chemistry (mg/L)								
Ammonia	N/A	--	--	--	--	--	--	--
Total Acid	N/A	--	--	--	--	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	0.0061	ND	ND	NA	NA	ND	ND
2,4- and 2,6-Dinitrophenol (DNP)	0.039	ND	ND	ND	NA	NA	ND	ND
2,4,6-Trinitrophenol (TNP, picric acid)	0.040	ND	ND	ND	NA	NA	0.0076	ND

NOTES:

-- = Not Analyzed

N/A = Not Available

ND = Not Detected (detection limits for groundwater parameters were not provided in D&M Report)

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. For non-carcinogens, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

Screening Versus Current USEPA Criteria (1):

Orange shaded values represent exceedance of Tapwater RSLs.

Table 10-1. D&M Sampling Results - Groundwater (Continued)

Sample Location:	Current USEPA Tapwater RSLs(1)	AOC 1 - Ammonium Picrate Area			
		D&M-13			AMS-1
Sample Date:		3/26/1981	3/31/1981	4/14/1981	3/26/1981
Groundwater Chemistry (mg/L)					
Ammonia	N/A	--	--	--	--
Total Acid	N/A	--	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	ND	ND	ND	ND
2,4- and 2,6-Dinitrophenol (DNP)	0.039	ND	ND	ND	ND
2,4,6-Trinitrophenol (TNP, picric acid)	0.040	0.0204	ND	ND	0.0567

NOTES:

-- = Not Analyzed

N/A = Not Available

ND = Not Detected (detection limits for groundwater parameters were not provided in D&M Report)

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. For non-carcinogens, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

Screening Versus Current USEPA Criteria (1):

Orange shaded values represent exceedance of Tapwater RSLs.

Table 10-1. D&M Sampling Results - Groundwater (Continued)

Sample Location:	Current USEPA Tapwater	AOC 3 - Landfill Area					South of Landfill
		D&M-11			B-2	B-3	B-1
Sample Date:	RSLs(1)	3/26/1981	3/31/1981	4/8/1981	2/7/1981	Dry	2/7/1981
Groundwater Chemistry (mg/L)							
Ammonia	N/A	--	--	--	0.50	--	0.90
Total Acid	N/A	--	--	--	36.0	--	130
Dinitrochlorobenzene (DNCB)	N/A	0.0029	ND	ND	--	--	--
2,4- and 2,6-Dinitrophenol (DNP)	0.039	ND	ND	ND	--	--	--
2,4,6-Trinitrophenol (TNP, picric acid)	0.040	ND	ND	ND	ND	--	ND

NOTES:

-- = Not Analyzed

N/A = Not Available

ND = Not Detected (detection limits for groundwater parameters were not provided in D&M Report)

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. For non-carcinogens, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

Screening Versus Current USEPA Criteria (1):

Orange shaded values represent exceedance of Tapwater RSLs.

Table 10-1. D&M Sampling Results - Groundwater (Continued)

Sample Location:	Current USEPA Tapwater	West of Landfill			Near River	Administration Area		
		D&M-6			B-4	D&M-10		
Sample Date:	RSLs(1)	2/18/1981	3/31/1981	4/8/1981	2/17/1981	3/26/1981	3/31/1981	4/14/1981
Groundwater Chemistry (mg/L)								
Ammonia	N/A	--	--	--	--	--	--	--
Total Acid	N/A	--	--	--	--	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	ND	ND	ND	--	ND	ND	ND
2,4- and 2,6-Dinitrophenol (DNP)	0.039	ND	ND	ND	--	ND	ND	ND
2,4,6-Trinitrophenol (TNP, picric acid)	0.040	ND	ND	ND	ND	0.197	ND	ND

NOTES:

-- = Not Analyzed

N/A = Not Available

ND = Not Detected (detection limits for groundwater parameters were not provided in D&M Report)

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. For non-carcinogens, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

Screening Versus Current USEPA Criteria (1):

Orange shaded values represent exceedance of Tapwater RSLs.

Table 10-2. D&M Sampling Results - Soil

Sample Location:	Current USEPA Residential	USEPA	AOC 1 - Ammonium Picrate Area					
			B-5	B-6		B-7	A-3	D&M-1
Sample Depth (ft bgs):	Soil RSLs[1]	ECO-SSL[2]	6.5	6.5	25.5	6.5	Grab[3]	6.5
Soil Chemistry (mg/kg)								
Ammonium	N/A	N/A	--	--	--	--	--	--
Total Acid	N/A	N/A	--	--	--	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	N/A	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2,4-& 2,6-Dinitrophenol (DNP)	130	N/A	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2,4,6-Trinitrophenol (TNP, picric acid)	130	N/A	0.0122	0.0188	0.0182	0.0086	0.0589	0.0024

NOTES:

Sample dates were not specified in the D&M Report, for the soil samples.

-- = Not Analyzed

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Table, USEPA, November 2020. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

(2) USEPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008. (a) = Avian; (i) = Soil Invertebrates; (m) = Mammalian; (p) = Plants

(3) Grab samples were collected by D&M at the ground surface within the drainage ditches.

Screening Versus Current USEPA Criteria (1,2):

Orange shaded and bolded values represent exceedance of residential RSLs, bolded and italicized values represent exceedance of residential RSLs and ECO-SSLs.

Table 10-2. D&M Sampling Results - Soil (Continued)

Sample Location:	Current USEPA Residential Soil RSLs[1]	USEPA ECO-SSL[2]	AOC 1 - Ammonium Picrate Area		
			D&M-2	D&M-7	D&M-13
Sample Depth (ft bgs):			2.5		12.0
Soil Chemistry (mg/kg)					
Ammonium	N/A	N/A	--	--	--
Total Acid	N/A	N/A	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	N/A	< 0.005	< 0.005	< 0.005
2,4-& 2,6-Dinitrophenol (DNP)	130	N/A	< 0.005	< 0.005	< 0.005
2,4,6-Trinitrophenol (TNP, picric acid)	130	N/A	< 0.005	0.0622	< 0.005

NOTES:

Sample dates were not specified in the D&M Report, for the soil samples.

-- = Not Analyzed

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Table, USEPA, November 2020. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

(2) USEPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008. (a) = Avian; (i) = Soil Invertebrates; (m) = Mammalian; (p) = Plants

(3) Grab samples were collected by D&M at the ground surface within the drainage ditches.

Screening Versus Current USEPA Criteria (1,2):

Orange shaded and bolded values represent exceedance of residential RSLs, bolded and italicized values represent exceedance of residential RSLs and ECO-SSLs.

Table 10-2. D&M Sampling Results - Soil (Continued)

Sample Location:	Current USEPA Residential	USEPA	AOC 3 - Landfill Area				
			B-2			B-3	
Sample Depth (ft bgs):	Soil RSLs[(1)]	ECO-SSL[(2)]	6.2	5.7	11.0	6.5	10.3
Soil Chemistry (mg/kg)							
Ammonium	N/A	N/A	--	<1	100	200	200
Total Acid	N/A	N/A	--	2.0	<1.0	6.0	1.0
Dinitrochlorobenzene (DNCB)	N/A	N/A	< 0.005	--	--	--	--
2,4-& 2,6-Dinitrophenol (DNP)	130	N/A	< 0.005	--	--	--	--
2,4,6-Trinitrophenol (TNP, picric acid)	130	N/A	0.016	--	--	--	--

NOTES:

Sample dates were not specified in the D&M Report, for the soil samples.

-- = Not Analyzed

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Table, USEPA, November 2020. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

(2) USEPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008. (a) = Avian; (i) = Soil Invertebrates; (m) = Mammalian; (p) = Plants

Screening Versus Current USEPA Criteria (1,2):

Orange shaded and bolded values represent exceedance of residential RSLs, bolded and italicized values represent exceedance of residential RSLs and ECO-SSLs.

Table 10-2. D&M Sampling Results - Soil (Continued)

Sample Location:	Current USEPA Residential Soil RSLs[1]	USEPA ECO-SSL[2]	South of Landfill B-1			West of Landfill D&M-6	Near River B-4	Administration Area D&M-10
Sample Depth (ft bgs):			2.5	13.5	15.5	6.5	6.5	7.0
Soil Chemistry (mg/kg)								
Ammonium	N/A	N/A	--	400	200	--	--	--
Total Acid	N/A	N/A	--	<1.0	<1.0	--	--	--
Dinitrochlorobenzene (DNCB)	N/A	N/A	< 0.005	--	--	< 0.005	< 0.005	< 0.005
2,4-& 2,6-Dinitrophenol (DNP)	130	N/A	< 0.005	--	--	< 0.005	< 0.005	< 0.005
2,4,6-Trinitrophenol (TNP, picric acid)	130	N/A	0.013	--	--	0.0316	0.0193	< 0.005

NOTES:

Sample dates were not specified in the D&M Report, for the soil samples.

-- = Not Analyzed

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Table, USEPA, November 2020. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6.

(2) USEPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008. (a) = Avian; (i) = Soil Invertebrates; (m) = Mammalian; (p) = Plants

Screening Versus Current USEPA Criteria (1,2):

Orange shaded and bolded values represent exceedance of residential RSLs, bolded and italicized values represent exceedance of residential RSLs and ECO-SSLs.

Table 10-3. M&E Sampling Results - Groundwater

Sample Name:	Current USEPA Tapwater RSLs(1)	AOC 1 - Ammonium Picrate Area			AOC 3 - Landfill Area
		MW-5	MW-6	MW-7	MW-8
Inorganics (mg/L)					
Nitrite/Nitrate	N/A	<0.050	<0.050	0.560	0.340
Total Metals (mg/L)					
Arsenic	0.000052	<0.010	0.011	<0.010	<0.010
Barium	3.8	0.459	<0.200	<0.200	<0.200
Cadmium	0.0092	<0.005	<0.005	<0.005	<0.005
Chromium	0.000035 (CrVI)	0.034	<0.01	<0.01	0.018
Lead	0.015 (al)	0.037	0.0082	<0.005	0.196

NOTES:

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. Tapwater RSLs. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6. [al = lead action level]

Screening Versus Current USEPA Criteria (1):

Orange shaded values represent exceedance of Tapwater RSLs.

No VOCs or SVOCs detected above detection limits.

Table 10-4. M&E Sampling Results - Soil

				AOC 1 - Ammonium Picrate Area					AOC 3 - Landfill Area
Sample Name:	Current USEPA Residential	USEPA	NYS Maximum Background for Statewide Rural Surface	Soil #7	Soil #8	Soil #9	Soil #10	Soil #11	Soil #12
Sample Depth:	Soil RSLs[1]	ECO-SSL[2]	Soil [3]	6 in bgs	6 in bgs	6 in bgs	6 in bgs	6 in bgs	6 in bgs
Semivolatiles (mg/kg)									
Phenanthrene	N/A	29.0 (i)	N/A	1.500	<0.420	<0.460	<0.530	0.150	0.150
Fluoranthene	2,400	1.10 (m)	3.200	3.400	<0.420	0.140	<0.530	0.540	0.220
Pyrene	1,800	1.10 (m)	4.600	3.400	<0.420	0.190	<0.530	0.640	0.340
Benzo(a)anthracene	1.1	1.10 (m)	N/A	***1.600***	<0.420	<0.460	<0.530	0.240	0.140
Chrysene	110	1.10 (m)	N/A	1.900	<0.420	<0.460	<0.530	0.320	0.200
bis(2-Ethylhexyl)phthalate	39	N/A	N/A	<9.600	0.440 B	0.330 B	<0.530	0.260 B	0.280 B
Benzo(b)fluoranthene	1.1	1.10 (m)	N/A	***1.500***	<0.420	<0.460	<0.530	0.290	0.140
Benzo(k)fluoranthene	11	1.10 (m)	N/A	1.700	<0.420	<0.460	<0.530	0.270	0.160
Benzo(a)pyrene	0.11	1.10 (m)	N/A	***1.800***	<0.420	<0.460	<0.530	**0.230**	**0.140**
Indeno(1,2,3-cd)pyrene	1.1	1.10 (m)	N/A	***2.600***	<0.420	<0.460	<0.530	<0.400	<0.390
Benzo(g,h,i)perylene	N/A	1.10 (m)	N/A	1.200	<0.420	<0.460	<0.530	<0.400	<0.390
Inorganics (mg/kg)									
Nitrite/Nitrate	N/A	N/A	N/A	1.900	<0.280	0.520	0.950	2.300	1.300
Metals (mg/kg)									
Arsenic	0.68	18 (p)	28.1	<4.200	10.600	7.600	5.800	3.900	2.800
Barium	15,000	330 (i)	278	<43.80	<38.30	92.10	59.30	50.20	<41.30
Chromium	0.3 (CrVI)	26 (a)	24.4 (total)	5.400	4.000	8.400	8.500	10.000	6.300
Lead	400	11 (a)	112	16.00	<0.970	12.20	10.70	15.40	17.30

NOTES:

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Table, USEPA, November 2020. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x1

(2) USEPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008. (a) = Avian; (i) = Soil Invertebrates; (m) = Mammalian; (p) = Plants

(3) Concentrations of Selected Analytes in Rural New York State Surface Soils: A Summary Report on the Statewide Rural Surface Soil Survey, NYSDEC, August 2005.

Data Qualifiers (Organics):

B Compound detected in one or more QA/QC blank sample(s).

Screening Versus Current USEPA Criteria (1,4):

Orange shaded and bolded values represent exceedance of residential RSLs, bolded and italicized values represent exceedance of residential RSLs and ECO-SSLs.

Green shaded values represent exceedance of ECO-SSLs.

Table 10-5. M&E Sampling Results - Tank Water

Sample Name:	Current USEPA Tap Water RSLs[(1)]	BTAG Freshwater Screening Benchmarks[(2)]	AOC 1 - Ammonium Picrate Area			AOC 2 - Acid Area	
			Tank #1	Tank #2	Tank #3	Tank #4	Tank #5
Semivolatiles (mg/L)							
Phenol	5.8	0.004	<0.010	0.013	<0.010	<0.010	<0.010
Inorganics (mg/L)							
Nitrite/Nitrate	N/A	0.020 (nitrite)	0.073	<0.050	0.140	0.790	0.490
Total Metals (mg/L)							
Arsenic	0.000052	0.005	<0.020	**0.074**	<0.020	<0.020	<0.020
Barium	3.8	0.004	<0.200	0.254	<0.200	<0.200	<0.200
Cadmium	0.0092	0.00025	<0.005	**0.021**	<0.005	<0.005	<0.005
Chromium (total)	0.000035 (CrVI)	0.085	0.027	**0.192**	0.025	<0.010	<0.010
Lead	0.015 (al)	0.0025	0.012	**1.140**	**0.023**	**0.021**	**0.025**
Mercury	0.0057	0.000026	<0.0002	0.001	<0.0002	<0.0002	<0.0002

NOTES:

N/A = Not Available

(1) USEPA Regional Screening Levels (RSLs) Summary Table, USEPA, November 2020. Tap Water RSLs. For non-carcinogens except lead, value shown is equal to HI=1.0. Carcinogenic values equal to 1x10-6. [al = lead action level]

(2) USEPA Region III BTAG Freshwater Screening Benchmarks, July 2006.

Screening Versus Current USEPA Criteria (1,2):

Orange shaded values represent exceedance of Tap Water RSLs; Bolded indicates exceedance of all criteria.

Green shaded values represent exceedance of BTAG Fresh Water criteria.

No VOCs were detected above detection limits.

APPENDICES

APPENDIX A
Project Action Limit Back-up

This appendix provides back-up for the project action limits presented in Worksheet #15. Please see the up-front text of Worksheet #15 for the hierarchy of screening levels and full citations.

THIS PAGE LEFT BLANK INTENTIONALLY

APPENDIX A.1

SURROGATE COMPOUND SELECTION

This Page Left Blank Intentionally

APPENDIX A.1

Surrogate Compound Selection

Ecological screening benchmarks are not available for two of the main COPECs at the Site: picric acid (2,4,6-trinitrophenol) and 2,4-dinitrochlorobenene (DNCB). In order to have a project action limit (PAL) with which to determine whether or not the selected analytical methods have limits of quantitation (LOQs) sufficiently low to meet data quality objectives, it was decided to use benchmarks from surrogate compounds. Appropriate surrogates would have similar chemical properties and available toxicity benchmarks. The selection of surrogates for picric acid and DNCB are presented below.

Picric acid is an aromatic compound with three electron-withdrawing (nitro) substituents at the para and at both ortho positions relative to the hydroxyl group; as a result, the phenolic proton is quite acidic (pKa < 1). As a starting point, the minimum requirements for finding an acceptable surrogate for picric acid included finding another phenolic compound with similar substituents in terms of steric hindrance, location on the aromatic ring and electron-withdrawing capacity (see Table A.1-1 for candidate structural analogs). Although chlorine and bromine are electron-withdrawing substituents, they are not the best choices as nitro group replacements, as they are single atoms and would not help delocalize the charge of a phenoxide ion in the same manner as the nitro groups. Therefore, a reasonable compromise would be a nitro-substituted phenol with substitution in the ortho- and/or para- positions if possible. The compounds 2,4-dinitrophenol (pKa ≈ 4) and 2,5-dinitrophenol (pKa ≈ 5) are good candidates. The substitution pattern of 2,4-dinitrophenol (ortho-, para-) is more favorable than the substitution pattern of 2,5-dinitrophenol (ortho, meta-) for stabilization of charge, as both of its nitro groups are appropriately positioned to delocalize charge. In addition, 2,4-dinitrophenol has available EPA (Region 4) ecological screening benchmarks; whereas, 2,5-dinitrophenol does not. As a result of these considerations, 2,4-dinitrophenol was selected as the best surrogate for picric acid. A safety factor of ten has been applied to the 2,4-dinitrophenol benchmarks to account for potential differences between picric acid and 2,4-dinitrophenol toxicity.

Medium	2,4-Dinitrophenol Value[1]	Value used for Picric Acid PAL in Worksheet #15[2]
Soil	0.061 mg/kg	0.0061 mg/kg

[1]EPA, 2018.

[2]2,4-Dinitrophenol value divided by 10.

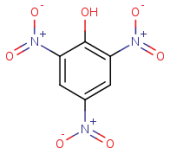
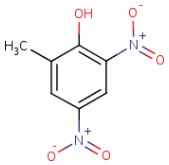
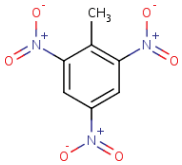
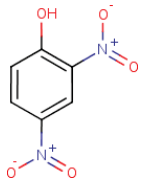
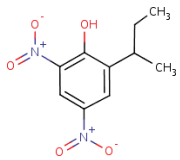
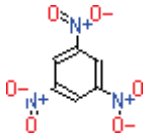
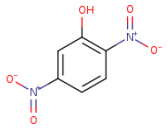
DNCB is an aromatic compound with two nitro substituents at the para- and ortho- positions relative to the chloride group. As a starting point, the minimum requirements for finding an acceptable substitute for DNCB included finding another polynitrobenzene with a similar substituent pattern on the aromatic ring (see Table A.1-2 for candidate structural analogs). Although an alternate 2,4-dinitrohalobenzene would be most similar, there were no compounds of that type with available toxicity benchmarks. Therefore, a reasonable compromise is to look for a dinitro-substituted benzene with the nitro groups oriented in the meta position relative to each other if possible. The compounds 2,4-dinitrotoluene (DNT) and 1,3-dinitrobenzene are good candidates; both have toxicity benchmarks available from acceptable regulatory guidance sources. 2,4-dinitrotoluene has water solubility and log K_{ow} values closer to those of 2,4-dinitrochlorobenzene than does 1,3-dinitrobenzene. In addition, 2,4-dinitrotoluene has a methyl group at the same position on the aromatic ring as the chloride in 2,4-dinitrochlorobenzene, giving a similar steric hindrance around the adjacent nitro group. As a result of these considerations, 2,4-dinitrotoluene was selected as the best surrogate for 2,4-dinitrochlorobenzene. A safety factor of ten has been applied to the surrogate benchmark to account for the difference between 2,4-dinitrochlorobenzene and 2,4-dinitrotoluene.

Medium	2,4-Dinitrotoluene Value[1]	Value used for DNCB PAL in Worksheet #15[2]
Soil	6 mg/kg	0.6 mg/kg

[1]EPA, 2018.

[2]2,4-Dinitrotoluene value divided by 10.

Table A.1-1. Physicochemical Properties of Picric Acid (CASRN 88-89-1) and Candidate Structural Analogs

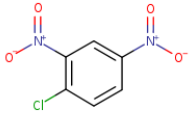
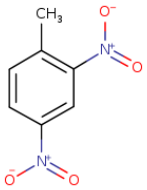
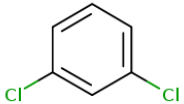
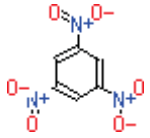
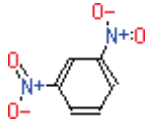
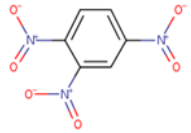
Chemical	2,4,6-Trinitrophenol (picric acid)	2-Methyl-4,6-dinitrophenol (DNOC)	2,4,6-Trinitrotoluene	2,4-Dinitrophenol (2,4 DNP)	2-(1-Methylpropyl)-4,6-dinitrophenol (Dinoseb)	1,3,5-Trinitrobenzene	2,5-Dinitrophenol
Structure							
CASRN	88-89-1	534-52-1	118-96-7	51-28-5	88-85-7	99-35-4	329-71-5
Molecular weight[a]	229.10	198.133	227.132	184.11	240.214	213.105	184.11
DSSTox similarity score (%)	100	78	58.3	99	60.6	73.6	95
ChemID Plus similarity score (%)[a]	100	83.86	83.51	80.26	80.17	75.03	74.82
Melting point (°C)[a]	122.5	86.6	80.1	115.5	40	121.5	108
Boiling point (°C)[a]	300[b]	378	NV	NV	332	315	NV
Vapor pressure (mmHg [at °C])[a]	7.50×10^{-7} (at 25°C)	1.06×10^{-4} (at 25°C)	8.02×10^{-6} (at 25°C)	3.90×10^{-4} (at 20°C)	NV	NV	1.22×10^{-4} (at 25°C)
Henry's law constant (atm-m^3/mole [at °C])[a]	1.70×10^{-11} (at 25°C)	1.4×10^{-6} (at 25°C)	2.08×10^{-8} (at 25°C)	8.60×10^{-8} (at 20°C)	4.56×10^{-7} (at 25°C)	3.31×10^{-10}	7.68×10^{-8} (at 25°C)
Water solubility (mg/L [at C])[a]	1.27×10^{4} (at 25°C)	198 (at 20°C)	130 (at 25°C)	2,790 (at 25°C)	52 (at 25°C)	278 (at 15°C)	385 (at 25°C)
Log K_{ow} [a]	1.33	2.12	1.6	1.67	3.56	1.18	1.75
pKa[a]	0.38 (at 25°C)	4.31 (at 21°C)	NV	4.09 (at 25°C)	4.62	NV	5.21 (at 25°C)

[a] ChemIDplus (2015).
[b] ChemicalBook (2015)

NV = not available.

Source: EPA 2015. Provisional Peer-Reviewed Toxicity Values for Picric Acid (2,4,6-Trinitrophenol) (CASRN 88-89-1). Superfund Health Risk Technical Support Center. National Center for Environmental Assessment. Office of Research and Development. Cincinnati, OH. EPA/690/R-15/010F.

Table A.1-2. Physicochemical Properties of DNCB (CASRN 97-00-7) and Candidate Structural Analogs

Chemical	2,4-Dinitrochlorobenzene (DNCB)	2,4-Dinitrotoluene	1,3-Dichlorobenzene	1,3,5-Trinitrobenzene	1,3-Dinitrobenzene	1,2,4-Trinitrobenzene*
Structure[a]						
CASRN	97-00-7	121-14-2	541-73-1	99-35-4	99-65-0	610-31-1
Molecular weight[a]	202.553	182.134	147.004	213.105	168.108	213.105
ChemID Plus similarity score (%)[a]	100	80.63	NV	67.41	70.89	68.39
Melting point (°C)[a]	53	71	-24.8	121.5	90	218.55 (EPISuite)
Boiling point (°C)[a]	315	300	173	315	291	512.74 (EPISuite)
Vapor pressure (mmHg [at °C])[a]	8.49×10^{-5} (at 25°C)[b]	1.47×10^{-4} (at 25°C)	2.15 (at 25°C)	NV	NV	0.9 (at 25°C) (ACD/Labs) 6.54×10^{-13} (EPISuite)
Henry's law constant (atm-m^3/mole [at °C])[a]	2.82 × 10−6 (at 25°C)	5.4×10^{-8} (at 25°C)	2.63×10^{-3} (at 25°C)	3.31×10^{-10}	4.90×10^{-8}	2.776×10^{-18} (EPISuite)
Water solubility (mg/L [at C])[a]	8 (at 15°C)	270 (at 22°C)	125 (at 25°C)	278 (at 15°C)	533 (at 25°C)	6.638×10^{4} (at 25°C) (EPISuite)
Log K_{ow} [a]	2.17	1.98	3.53	1.18	1.49	-0.75 (EPISuite)
pKa[a]	NV	13.53[b]	NV	NV	NV	NV

[a] ChemIDplus (2019).
[b] NCBI (2019)

* = Chemicals properties predicted by ACD/Labs and EPI Suite.
NV = not available.

References

ChemicalBook (ChemicalBook Inc.) (2015). Available online at https://www.chemicalbook.com/

ChemicalBook (ChemicalBook Inc.) (2019). Available online at https://www.chemicalbook.com/

ChemIDplus (2015). ChemIDplus advanced. Bethesda, MD. U.S. National Library of Medicine. Retrieved from: http://www.chem.sis.nlm.nih.gov/chemidplus/

ChemIDplus (2019). ChemIDplus advanced. Bethesda, MD. U.S. National Library of Medicine. Retrieved from: http://www.chem.sis.nlm.nih.gov/chemidplus/

EPA Region 4 (2018) *Ecological Risk Assessment Supplemental Guidance* https://www.epa.gov/sites/production/files/2018-03/documents/era_regional_supplemental_guidance_report-march-2018_update.pdf

National Center for Biotechnology Information. PubChem Database. 1-Chloro-2,4-dinitrobenzene, CID=6, https://pubchem.ncbi.nlm.nih.gov/compound/1-Chloro-2_4-dinitrobenzene (accessed on Dec. 20, 2019)

This Page Left Blank Intentionally

APPENDIX A.2
Ecological PAL Hierarchy Values

Note: See Worksheet #15 text for additional details regarding the hierarchies.

Table A.2-1
Soil Ecological PALs Hierarchy
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	1) EPA Eco SSLs (mg/kg)	2) EPA Region 4 (mg/kg)	3) EPA Region 5 RCRA ESLs (mg/kg)	4) LANL ECORISK (mg/kg)	5) Derived - See Appendix A.1 (mg/kg)	Ecological Project Action Limit	
								Value (mg/kg)	Reference
BNASIM	1-Methylnaphthalene	90-12-0	-	-	-	-	-	See LMW PAHs	NA
BNASIM	2-Methylnaphthalene	91-57-6	-	-	3.24	16	-	See LMW PAHs	NA
BNASIM	Acenaphthene	83-32-9	-	-	682	0.25	-	See LMW PAHs	NA
BNASIM	Acenaphthylene	208-96-8	-	-	682	120	-	See LMW PAHs	NA
BNASIM	Anthracene	120-12-7	-	-	1480	6.8	-	See LMW PAHs	NA
BNASIM	Benzo(a)anthracene	56-55-3	-	-	5.21	0.73	-	See HMW PAHs	NA
BNASIM	Benzo(a)pyrene	50-32-8	-	-	1.52	62	-	See HMW PAHs	NA
BNASIM	Benzo(b)fluoranthene	205-99-2	-	-	59.8	18	-	See HMW PAHs	NA
BNASIM	Benzo(g,h,i)perylene	191-24-2	-	-	119	25	-	See HMW PAHs	NA
BNASIM	Benzo(k)fluoranthene	207-08-9	-	-	148	71	-	See HMW PAHs	NA
BNASIM	Chrysene	218-01-9	-	-	4.73	3.1	-	See HMW PAHs	NA
BNASIM	Dibenz(a,h)anthracene	53-70-3	-	-	18.4	14	-	See HMW PAHs	NA
BNASIM	Fluoranthene	206-44-0	-	-	122	10	-	See LMW PAHs	NA
BNASIM	Fluorene	86-73-7	-	-	122	3.7	-	See LMW PAHs	NA
BNASIM	Indeno(1,2,3-c,d)pyrene	193-39-5	-	-	109	71	-	See HMW PAHs	NA
BNASIM	Naphthalene	91-20-3	-	-	0.0994	1	-	See LMW PAHs	NA
BNASIM	Phenanthrene	85-01-8	-	-	45.7	5.5	-	See LMW PAHs	NA
BNASIM	Pyrene	129-00-0	-	-	78.5	10	-	See HMW PAHs	NA
Calculated	Low Molecular Weight PAHs		29	29	-	-	-	29	1) EPA Eco SSLs
Calculated	High Molecular Weight PAHs		1.1	1.1	-	-	-	1.1	1) EPA Eco SSLs
SW6010C	Aluminum	7429-90-5	Narrative	Narrative	-	-	-	Narrative	1) EPA Eco SSLs
SW6010C	Barium	7440-39-3	330	330	1.04	110	-	330	1) EPA Eco SSLs
SW6010C	Beryllium	7440-41-7	21	2.5	1.06	2.5	-	21	1) EPA Eco SSLs
SW6010C	Calcium	7440-70-2	-	-	-	-	-	NA	NA
SW6010C	Chromium	7440-47-3	26	23	0.4	23	-	26	1) EPA Eco SSLs
SW6010C	Cobalt	7440-48-4	13	13	0.14	13	-	13	1) EPA Eco SSLs
SW6010C	Copper	7440-50-8	28	28	5.4	14	-	28	1) EPA Eco SSLs
SW6010C	Iron	7439-89-6	Narrative	Narrative	-	-	-	Narrative	1) EPA Eco SSLs
SW6010C	Lead	7439-92-1	11	11	0.0537	11	-	11	1) EPA Eco SSLs
SW6010C	Magnesium	7439-95-4	-	-	-	-	-	NA	NA
SW6010C	Manganese	7439-96-5	220	220	-	220	-	220	1) EPA Eco SSLs
SW6010C	Nickel	7440-02-0	38	38	13.6	-	-	38	1) EPA Eco SSLs
SW6010C	Potassium	7440-09-7	-	-	-	-	-	NA	NA
SW6010C	Silver	7440-22-4	4.2	4.2	4.04	2.6	-	4.2	1) EPA Eco SSLs
SW6010C	Sodium	7440-23-5	-	-	-	-	-	NA	NA
SW6010C	Vanadium	7440-62-2	7.8	7.8	1.59	4.7	-	7.8	1) EPA Eco SSLs
SW6010C	Zinc	7440-66-6	46	46	6.62	47	-	46	1) EPA Eco SSLs
SW6020A	Antimony	7440-36-0	0.27	0.27	0.142	2.3	-	0.27	1) EPA Eco SSLs

Table A.2-1
Soil Ecological PALs Hierarchy
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	1) EPA Eco SSLs (mg/kg)	2) EPA Region 4 (mg/kg)	3) EPA Region 5 RCRA ESLs (mg/kg)	4) LANL ECORISK (mg/kg)	5) Derived - See Appendix A.1 (mg/kg)	Ecological Project Action Limit	
								Value (mg/kg)	Reference
SW6020A	Arsenic	7440-38-2	18	18	5.7	6.8	-	18	1) EPA Eco SSLs
SW6020A	Cadmium	7440-43-9	0.36	0.36	0.00222	0.27	-	0.36	1) EPA Eco SSLs
SW6020A	Selenium	7782-49-2	0.52	0.52	0.0276	0.52	-	0.52	1) EPA Eco SSLs
SW6020A	Thallium	7440-28-0	-	0.05	0.0569	0.05	-	0.05	2) EPA Region 4 Soil
SW7471B	Mercury	7439-97-6	-	0.013	0.1	0.013	-	0.013	2) EPA Region 4 Soil
SW8082A	PCB-1016	12674-11-2	-	-	-	1.1	-	See Total PCBs	NA
SW8082A	PCB-1221	11104-28-2	-	-	-	-	-	See Total PCBs	NA
SW8082A	PCB-1232	11141-16-5	-	-	-	-	-	See Total PCBs	NA
SW8082A	PCB-1242	53469-21-9	-	-	-	0.041	-	See Total PCBs	NA
SW8082A	PCB-1248	12672-29-6	-	-	-	0.0073	-	See Total PCBs	NA
SW8082A	PCB-1254	11097-69-1	-	-	-	0.041	-	See Total PCBs	NA
SW8082A	PCB-1260	11096-82-5	-	-	-	0.88	-	See Total PCBs	NA
SW8082A	PCB-1262	37324-23-5	-	-	-	-	-	See Total PCBs	NA
SW8082A	PCB-1268	11100-14-4	-	-	-	-	-	See Total PCBs	NA
Calculated	Total PCBs		-	0.041	0.000332	-	-	0.041	2) EPA Region 4 Soil
SW8260	1,1,1-Trichloroethane	71-55-6	-	0.04	29.8	260	-	0.04	2) EPA Region 4 Soil
SW8260	1,1,2,2-Tetrachloroethane	79-34-5	-	0.127	0.127	-	-	0.127	2) EPA Region 4 Soil
SW8260	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	-	-	-	-	-	NA	NA
SW8260	1,1,2-Trichloroethane	79-00-5	-	0.32	28.6	-	-	0.32	2) EPA Region 4 Soil
SW8260	1,1-Dichloroethane	75-34-3	-	0.14	20.1	210	-	0.14	2) EPA Region 4 Soil
SW8260	1,1-Dichloroethene	75-35-4	-	0.04	8.28	11	-	0.04	2) EPA Region 4 Soil
SW8260	1,2,3-Trichlorobenzene	87-61-6	-	20	-	-	-	20	2) EPA Region 4 Soil
SW8260	1,2,4-Trichlorobenzene	120-82-1	-	0.27	11.1	0.27	-	0.27	2) EPA Region 4 Soil
SW8260	1,2-Dibromo-3-chloropropane	96-12-8	-	-	0.0352	-	-	0.0352	3) RCRA ESLs
SW8260	1,2-Dibromoethane (EDB)	106-93-4	-	-	1.23	-	-	1.23	3) RCRA ESLs
SW8260	1,2-Dichlorobenzene	95-50-1	-	0.09	2.96	0.92	-	0.09	2) EPA Region 4 Soil
SW8260	1,2-Dichloroethane	107-06-2	-	0.4	21.2	0.85	-	0.4	2) EPA Region 4 Soil
SW8260	1,2-Dichloropropane	78-87-5	-	0.28	32.7	-	-	0.28	2) EPA Region 4 Soil
SW8260	1,3-Dichlorobenzene	541-73-1	-	0.08	37.7	0.74	-	0.08	2) EPA Region 4 Soil
SW8260	1,3-Dichloropropane	142-28-9	-	-	-	-	-	NA	NA
SW8260	1,4-Dichlorobenzene	106-46-7	-	0.88	0.546	0.89	-	0.88	2) EPA Region 4 Soil
SW8260	2-Butanone (MEK)	78-93-3	-	1	89.6	350	-	1	2) EPA Region 4 Soil
SW8260	2-Hexanone	591-78-6	-	0.36	12.6	0.36	-	0.36	2) EPA Region 4 Soil
SW8260	4-Methyl-2-pentanone (MIBK)	108-10-1	-	-	443	9.7	-	443	3) RCRA ESLs
SW8260	Acetone	67-64-1	-	1.2	2.5	1.2	-	1.2	2) EPA Region 4 Soil
SW8260	Benzene	71-43-2	-	0.12	0.255	24	-	0.12	2) EPA Region 4 Soil
SW8260	Bromochloromethane	74-97-5	-	-	-	-	-	NA	NA
SW8260	Bromodichloromethane	75-27-4	-	-	0.54	-	-	0.54	3) RCRA ESLs

Table A.2-1
Soil Ecological PALs Hierarchy
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	1) EPA Eco SSLs (mg/kg)	2) EPA Region 4 (mg/kg)	3) EPA Region 5 RCRA ESLs (mg/kg)	4) LANL ECORISK (mg/kg)	5) Derived - See Appendix A.1 (mg/kg)	Ecological Project Action Limit	
								Value (mg/kg)	Reference
SW8260	Bromoform	75-25-2	-	0.07	15.9	-	-	0.07	2) EPA Region 4 Soil
SW8260	Bromomethane	74-83-9	-	0.002	0.235	-	-	0.002	2) EPA Region 4 Soil
SW8260	Carbon disulfide	75-15-0	-	0.005	0.0941	0.81	-	0.005	2) EPA Region 4 Soil
SW8260	Carbon tetrachloride	56-23-5	-	0.05	2.98	-	-	0.05	2) EPA Region 4 Soil
SW8260	Chlorobenzene	108-90-7	-	2.4	13.1	2.4	-	2.4	2) EPA Region 4 Soil
SW8260	Chloroethane	75-00-3	-	-	-	-	-	NA	NA
SW8260	Chloroform	67-66-3	-	0.05	1.19	8	-	0.05	2) EPA Region 4 Soil
SW8260	Chloromethane	74-87-3	-	-	10.4	-	-	10.4	3) RCRA ESLs
SW8260	cis-1,2-Dichloroethene	156-59-2	-	0.04	-	24	-	0.04	2) EPA Region 4 Soil
SW8260	cis-1,3-Dichloropropene	10061-01-5	-	0.001	0.398	-	-	0.001	2) EPA Region 4 Soil
SW8260	Cyclohexane	110-82-7	-	-	-	-	-	NA	NA
SW8260	Dibromochloromethane	124-48-1	-	-	2.05	-	-	2.05	3) RCRA ESLs
SW8260	Dichlorodifluoromethane	75-71-8	-	-	39.5	-	-	39.5	3) RCRA ESLs
SW8260	Ethylbenzene	100-41-4	-	0.27	5.16	-	-	0.27	2) EPA Region 4 Soil
SW8260	Isopropylbenzene (Cumene)	98-82-8	-	0.04	-	-	-	0.04	2) EPA Region 4 Soil
SW8260	m,p-Xylene	179601-23-1	-	0.1	10	1.4	-	0.1	2) EPA Region 4 Soil
SW8260	Methyl acetate	79-20-9	-	-	-	-	-	NA	NA
SW8260	Methyl tert-butyl ether (MTBE)	1634-04-4	-	-	-	-	-	NA	NA
SW8260	Methylcyclohexane	108-87-2	-	-	-	-	-	NA	NA
SW8260	Methylene chloride	75-09-2	-	0.21	4.05	2.6	-	0.21	2) EPA Region 4 Soil
SW8260	o-Xylene	95-47-6	-	0.1	10	1.4	-	0.1	2) EPA Region 4 Soil
SW8260	Styrene	100-42-5	-	1.2	4.69	1.2	-	1.2	2) EPA Region 4 Soil
SW8260	Tetrachloroethene (PCE)	127-18-4	-	0.06	9.92	0.18	-	0.06	2) EPA Region 4 Soil
SW8260	Toluene	108-88-3	-	0.15	5.45	23	-	0.15	2) EPA Region 4 Soil
SW8260	trans-1,2-Dichloroethene	156-60-5	-	0.04	0.784	24	-	0.04	2) EPA Region 4 Soil
SW8260	trans-1,3-Dichloropropene	10061-02-6	-	0.001	0.398	-	-	0.001	2) EPA Region 4 Soil
SW8260	Trichlorofluoromethane	75-69-4	-	16.4	16.4	52	-	16.4	2) EPA Region 4 Soil
SW8260	Vinyl chloride	75-01-4	-	0.03	0.646	0.12	-	0.03	2) EPA Region 4 Soil
SW8270	1,2,4,5-Tetrachlorobenzene	95-94-3	-	0.18	2.02	-	-	0.18	2) EPA Region 4 Soil
SW8270	1,4-Dioxane (p-Dioxane)	123-91-1	-	-	2.05	-	-	2.05	3) RCRA ESLs
SW8270	2,2'-Oxybis(1-chloro)propane	108-60-1	-	-	19.9	-	-	19.9	3) RCRA ESLs
SW8270	2,3,4,6-Tetrachlorophenol	58-90-2	-	0.04	0.199	-	-	0.04	2) EPA Region 4 Soil
SW8270	2,4,5-Trichlorophenol	95-95-4	-	4	14.1	-	-	4	2) EPA Region 4 Soil
SW8270	2,4,6-Trichlorophenol	88-06-2	-	9.94	9.94	-	-	9.94	2) EPA Region 4 Soil
SW8270	2,4-Dichlorophenol	120-83-2	-	0.05	87.5	-	-	0.05	2) EPA Region 4 Soil
SW8270	2,4-Dimethylphenol	105-67-9	-	0.04	0.01	-	-	0.04	2) EPA Region 4 Soil
SW8270	2,4-Dinitrochlorobenzene	97-00-7	-	-	-	-	0.6	0.6	5) Derived - See Appendix A.1
SW8270	2,4-Dinitrophenol	51-28-5	-	0.061	0.0609	-	-	0.061	2) EPA Region 4 Soil

Table A.2-1
Soil Ecological PALs Hierarchy
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	1) EPA Eco SSLs (mg/kg)	2) EPA Region 4 (mg/kg)	3) EPA Region 5 RCRA ESLs (mg/kg)	4) LANL ECORISK (mg/kg)	5) Derived - See Appendix A.1 (mg/kg)	Ecological Project Action Limit	
								Value (mg/kg)	Reference
SW8270	2,4-Dinitrotoluene	121-14-2	-	6	1.28	6	-	6	2) EPA Region 4 Soil
SW8270	2,6-Dinitrotoluene	606-20-2	-	4	0.0328	4	-	4	2) EPA Region 4 Soil
SW8270	2-Chloronaphthalene	91-58-7	-	-	0.0122	-	-	0.0122	3) RCRA ESLs
SW8270	2-Chlorophenol	95-57-8	-	0.06	0.243	0.39	-	0.06	2) EPA Region 4 Soil
SW8270	2-Methylphenol (o-Cresol)	95-48-7	-	0.1	40.4	0.67	-	0.1	2) EPA Region 4 Soil
SW8270	2-Nitroaniline	88-74-4	-	0.02	74.1	5.3	-	0.02	2) EPA Region 4 Soil
SW8270	2-Nitrophenol	88-75-5	-	-	1.6	-	-	1.6	3) RCRA ESLs
SW8270	3,3'-Dichlorobenzidine	91-94-1	-	0.03	0.646	-	-	0.03	2) EPA Region 4 Soil
SW8270	3-Nitroaniline	99-09-2	-	-	-	-	-	NA	NA
SW8270	4,6-Dinitro-2-methylphenol	534-52-1	-	-	0.144	-	-	0.144	3) RCRA ESLs
SW8270	4-Bromophenyl phenyl ether	101-55-3	-	-	-	-	-	NA	NA
SW8270	4-Chloro-3-methylphenol	59-50-7	-	-	7.95	-	-	7.95	3) RCRA ESLs
SW8270	4-Chloroaniline	106-47-8	-	1	1.1	1	-	1	2) EPA Region 4 Soil
SW8270	4-Chlorophenyl phenyl ether	7005-72-3	-	-	-	-	-	NA	NA
SW8270	4-Nitroaniline	100-01-6	-	-	21.9	-	-	21.9	3) RCRA ESLs
SW8270	4-Nitrophenol	100-02-7	-	5.12	5.12	-	-	5.12	2) EPA Region 4 Soil
SW8270	Acetophenone	98-86-2	-	-	300	-	-	300	3) RCRA ESLs
SW8270	Atrazine	1912-24-9	-	0.00005	-	-	-	0.00005	2) EPA Region 4 Soil
SW8270	Benzaldehyde	100-52-7	-	-	-	-	-	NA	NA
SW8270	Benzyl butyl phthalate	85-68-7	-	0.59	0.239	90	-	0.59	2) EPA Region 4 Soil
SW8270	Biphenyl (Diphenyl)	92-52-4	-	0.2	-	-	-	0.2	2) EPA Region 4 Soil
SW8270	bis(2-Chloroethoxy) methane	111-91-1	-	-	0.302	-	-	0.302	3) RCRA ESLs
SW8270	bis(2-Chloroethyl) ether (2-Chloroethyl ether)	111-44-4	-	-	23.7	-	-	23.7	3) RCRA ESLs
SW8270	bis(2-Ethylhexyl) phthalate	117-81-7	-	0.02	0.925	0.02	-	0.02	2) EPA Region 4 Soil
SW8270	Caprolactam	105-60-2	-	-	-	-	-	NA	NA
SW8270	Carbazole	86-74-8	-	0.07	-	79	-	0.07	2) EPA Region 4 Soil
SW8270	Cresols, m- & p-	MEPH1314	-	0.08	3.49	0.69	-	0.08	2) EPA Region 4 Soil
SW8270	Dibenzofuran	132-64-9	-	0.15	-	6.1	-	0.15	2) EPA Region 4 Soil
SW8270	Diethyl phthalate	84-66-2	-	0.25	24.8	100	-	0.25	2) EPA Region 4 Soil
SW8270	Dimethyl phthalate	131-11-3	-	0.35	734	10	-	0.35	2) EPA Region 4 Soil
SW8270	Di-n-butyl phthalate	84-74-2	-	0.011	0.15	0.011	-	0.011	2) EPA Region 4 Soil
SW8270	Di-n-octyl phthalate	117-84-0	-	0.91	709	0.91	-	0.91	2) EPA Region 4 Soil
SW8270	Hexachlorobenzene	118-74-1	-	0.079	0.199	0.079	-	0.079	2) EPA Region 4 Soil
SW8270	Hexachlorobutadiene	87-68-3	-	0.009	0.0398	-	-	0.009	2) EPA Region 4 Soil
SW8270	Hexachlorocyclopentadiene	77-47-4	-	0.001	0.755	-	-	0.001	2) EPA Region 4 Soil
SW8270	Hexachloroethane	67-72-1	-	0.024	0.596	-	-	0.024	2) EPA Region 4 Soil
SW8270	Isophorone	78-59-1	-	-	139	-	-	139	3) RCRA ESLs
SW8270	Nitrobenzene	98-95-3	-	2.2	1.31	2.2	-	2.2	2) EPA Region 4 Soil

Table A.2-1
Soil Ecological PALs Hierarchy
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	1) EPA Eco SSLs (mg/kg)	2) EPA Region 4 (mg/kg)	3) EPA Region 5 RCRA ESLs (mg/kg)	4) LANL ECORISK (mg/kg)	5) Derived - See Appendix A.1 (mg/kg)	Ecological Project Action Limit	
								Value (mg/kg)	Reference
SW8270	n-Nitrosodi-n-propylamine	621-64-7	-	-	0.544	-	-	0.544	3) RCRA ESLs
SW8270	n-Nitrosodiphenylamine	86-30-6	-	0.545	0.545	-	-	0.545	2) EPA Region 4 Soil
SW8270	Pentachlorophenol	87-86-5	2.1	2.1	0.119	0.36	-	2.1	1) EPA Eco SSLs
SW8270	Phenol	108-95-2	-	0.79	120	0.79	-	0.79	2) EPA Region 4 Soil
SW8330A/B	Picric acid	88-89-1	-	-	-	-	0.0061	0.0061	5) Derived - See Appendix A.1

Notes:
Highlighting = Value selected for ecological PAL.
Chromium by method SW6020 is total chromium. The Eco-SSL is for trivalent chromium because the value is more conservative than that for hexavalent chromium. The Region 4, Region 5, and LANL values are for total chromium.
Cresols, m & p values: Region 4 value presented is the lower of the two isomers (p-cresol); LANL value presented is m-cresol value.
Aluminum is identified as a COPC only for those soils with a pH less than 5.5. The technical basis for this procedure is that the soluble and toxic forms of aluminum are only present in soil under soil pH values of less than 5.5. Site-specific considerations could, however warrant inclusion of aluminum as a COPC.
1,3-Dichloropropene (cis and trans) value used for each isomer from Region 4 and LANL.

Iron narrative statement (EPA Eco SSL, 2003): The main concern from an ecological risk perspective for iron is not direct chemical toxicity per se, but the effect of iron as a mediator in the geochemistry of other (potentially toxic) metals and the potential physical hazard of depositing flocculent. Soil pH and Eh should be included as standard soil chemical field parameters when conducting any field investigation. Although some EPA analytical methods require the collection of soil pH prior to analysis, this information is not always made available to the ultimate data users. In addition, the pH measured in the lab may no longer be representative of field conditions since the samples can be exposed to oxygen during storage prior to analysis. A determination of the geochemical conditions (i.e., pH and Eh at a minimum) of the environmental setting, as well as the presence of iron floc and the toxic metals, is critical to the determination of the relative importance of iron at a site.
Xylenes (total) value used for each isomer.
EPA Eco SSL - Environmental Protection Agency Ecological Soil Screening Values.
HMW PAHs = High molecular weight polycyclic aromatic hydrocarbons.
LANL = Los Alamos National Laboratory.
LMW PAHs = Low molecular weight polycyclic aromatic hydrocarbons.
mg/kg = Milligrams per kilogram.
NA = Not available.
PAL = Project Action Limit.
RCRA = Resource Conservation and Recovery Act.

Sources:
1) EPA Ecological Soil Screening Levels (EcoSSLs; various dates) (https://www.epa.gov/chemical-research/interim-ecological-soil-screening-level-documents)
2) EPA Region 4 Ecological Risk Assessment Supplemental Guidance (2018) – Soil Screening Values for Hazardous Waste Sites (https://www.epa.gov/sites/production/files/2018-03/documents/era_regional_supplemental_guidance_report-march-20
3) EPA Region 5 (2003) RCRA Ecological Screening Levels (ESLs) (https://archive.epa.gov/region5/waste/cars/web/pdf/ecological-screening-levels-200308.pdf)
4) Los Alamos National Laboratory (LANL) ECORISK Database Ecological Screening Levels (2017) (https://www.lanl.gov/environment/protection/eco-risk-assessment.php)
5) Derived - See Appendix A.1

THIS PAGE LEFT BLANK INTENTIONALLY

APPENDIX A.3

Final Soil PALs

Note: See Worksheet #15 text for additional details regarding the hierarchies.

Table A.3
Final Soil PALs
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	Human Health Project Action Limit			Ecological Project Action Limit			Final Project Action Limit		
			Value	Units	Reference	Value	Units	Reference	Value	Units	Reference
BNASIM	1-Methylnaphthalene	90-12-0	18	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	18	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	2-Methylnaphthalene	91-57-6	24	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	24	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Acenaphthene	83-32-9	360	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	360	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Acenaphthylene	208-96-8	NA	---	NA	See LMW PAHs	---	NA	NA	---	NA
BNASIM	Anthracene	120-12-7	1800	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	1800	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Benzo(a)anthracene	56-55-3	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Benzo(a)pyrene	50-32-8	0.11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	0.11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Benzo(g,h,i)perylene	191-24-2	NA	---	NA	See HMW PAHs	---	NA	NA	---	NA
BNASIM	Benzo(k)fluoranthene	207-08-9	11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Chrysene	218-01-9	110	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	110	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Dibenz(a,h)anthracene	53-70-3	0.11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	0.11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Fluoranthene	206-44-0	240	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	240	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Fluorene	86-73-7	240	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	240	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Naphthalene	91-20-3	2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See LMW PAHs	---	NA	2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
BNASIM	Phenanthrene	85-01-8	NA	---	NA	See LMW PAHs	---	NA	NA	---	NA
BNASIM	Pyrene	129-00-0	180	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See HMW PAHs	---	NA	180	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
Calculated	Low Molecular Weight PAHs		NA	---	NA	29	mg/kg	1) EPA Eco SSLs	29	mg/kg	1) EPA Eco SSLs
Calculated	High Molecular Weight PAHs		NA	---	NA	1.1	mg/kg	1) EPA Eco SSLs	1.1	mg/kg	1) EPA Eco SSLs
SW6010C	Aluminum	7429-90-5	7700	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	Narrative	mg/kg	1) EPA Eco SSLs	7700	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW6010C	Barium	7440-39-3	1500	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	330	mg/kg	1) EPA Eco SSLs	330	mg/kg	1) EPA Eco SSLs
SW6010C	Beryllium	7440-41-7	16	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	21	mg/kg	1) EPA Eco SSLs	16	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW6010C	Calcium	7440-70-2	NA	---	NA	NA	---	NA	NA	---	NA
SW6010C	Chromium	7440-47-3	12000	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	26	mg/kg	1) EPA Eco SSLs	26	mg/kg	1) EPA Eco SSLs
SW6010C	Cobalt	7440-48-4	2.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	13	mg/kg	1) EPA Eco SSLs	2.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW6010C	Copper	7440-50-8	310	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	28	mg/kg	1) EPA Eco SSLs	28	mg/kg	1) EPA Eco SSLs
SW6010C	Iron	7439-89-6	5500	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	Narrative	mg/kg	1) EPA Eco SSLs	5500	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW6010C	Lead	7439-92-1	400	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	11	mg/kg	1) EPA Eco SSLs	11	mg/kg	1) EPA Eco SSLs
SW6010C	Magnesium	7439-95-4	NA	---	NA	NA	---	NA	NA	---	NA
SW6010C	Manganese	7439-96-5	180	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	220	mg/kg	1) EPA Eco SSLs	180	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW6010C	Nickel	7440-02-0	150	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	38	mg/kg	1) EPA Eco SSLs	38	mg/kg	1) EPA Eco SSLs
SW6010C	Potassium	7440-09-7	NA	---	NA	NA	---	NA	NA	---	NA
SW6010C	Silver	7440-22-4	39	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	4.2	mg/kg	1) EPA Eco SSLs	4.2	mg/kg	1) EPA Eco SSLs
SW6010C	Sodium	7440-23-5	NA	---	NA	NA	---	NA	NA	---	NA
SW6010C	Vanadium	7440-62-2	39	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	7.8	mg/kg	1) EPA Eco SSLs	7.8	mg/kg	1) EPA Eco SSLs
SW6010C	Zinc	7440-66-6	2300	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	46	mg/kg	1) EPA Eco SSLs	46	mg/kg	1) EPA Eco SSLs
SW6020A	Antimony	7440-36-0	3.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.27	mg/kg	1) EPA Eco SSLs	0.27	mg/kg	1) EPA Eco SSLs
SW6020A	Arsenic	7440-38-2	0.68	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	18	mg/kg	1) EPA Eco SSLs	0.68	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW6020A	Cadmium	7440-43-9	7.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.36	mg/kg	1) EPA Eco SSLs	0.36	mg/kg	1) EPA Eco SSLs
SW6020A	Selenium	7782-49-2	39	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.52	mg/kg	1) EPA Eco SSLs	0.52	mg/kg	1) EPA Eco SSLs

Table A.3
Final Soil PALs
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	Human Health Project Action Limit			Ecological Project Action Limit			Final Project Action Limit		
			Value	Units	Reference	Value	Units	Reference	Value	Units	Reference
SW6020A	Thallium	7440-28-0	0.078	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.05	mg/kg	2) EPA Region 4 Soil	0.05	mg/kg	2) EPA Region 4 Soil
SW7471B	Mercury	7439-97-6	1.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.013	mg/kg	2) EPA Region 4 Soil	0.013	mg/kg	2) EPA Region 4 Soil
SW8082A	PCB-1016	12674-11-2	0.41	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.41	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1221	11104-28-2	0.2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1232	11141-16-5	0.17	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.17	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1242	53469-21-9	0.23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1248	12672-29-6	0.23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1254	11097-69-1	0.12	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.12	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1260	11096-82-5	0.24	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	See Total PCBs	---	NA	0.24	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8082A	PCB-1262	37324-23-5	NA	---	NA	See Total PCBs	---	NA	NA	---	NA
SW8082A	PCB-1268	11100-14-4	NA	---	NA	See Total PCBs	---	NA	NA	---	NA
Calculated	Total PCBs		NA	---	NA	0.041	mg/kg	2) EPA Region 4 Soil	0.041	mg/kg	2) EPA Region 4 Soil
SW8260	1,1,1-Trichloroethane	71-55-6	810	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8260	1,1,2,2-Tetrachloroethane	79-34-5	0.6	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.127	mg/kg	2) EPA Region 4 Soil	0.127	mg/kg	2) EPA Region 4 Soil
SW8260	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	670	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	670	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	1,1,2-Trichloroethane	79-00-5	0.15	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.32	mg/kg	2) EPA Region 4 Soil	0.15	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	1,1-Dichloroethane	75-34-3	3.6	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.14	mg/kg	2) EPA Region 4 Soil	0.14	mg/kg	2) EPA Region 4 Soil
SW8260	1,1-Dichloroethene	75-35-4	23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8260	1,2,3-Trichlorobenzene	87-61-6	6.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	20	mg/kg	2) EPA Region 4 Soil	6.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	1,2,4-Trichlorobenzene	120-82-1	5.8	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.27	mg/kg	2) EPA Region 4 Soil	0.27	mg/kg	2) EPA Region 4 Soil
SW8260	1,2-Dibromo-3-chloropropane	96-12-8	0.0053	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.0352	mg/kg	3) RCRA ESLs	0.0053	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	1,2-Dibromoethane (EDB)	106-93-4	0.036	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	1.23	mg/kg	3) RCRA ESLs	0.036	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	1,2-Dichlorobenzene	95-50-1	180	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.09	mg/kg	2) EPA Region 4 Soil	0.09	mg/kg	2) EPA Region 4 Soil
SW8260	1,2-Dichloroethane	107-06-2	0.46	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.4	mg/kg	2) EPA Region 4 Soil	0.4	mg/kg	2) EPA Region 4 Soil
SW8260	1,2-Dichloropropane	78-87-5	1.6	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.28	mg/kg	2) EPA Region 4 Soil	0.28	mg/kg	2) EPA Region 4 Soil
SW8260	1,3-Dichlorobenzene	541-73-1	NA	---	NA	0.08	mg/kg	2) EPA Region 4 Soil	0.08	mg/kg	2) EPA Region 4 Soil
SW8260	1,3-Dichloropropane	142-28-9	160	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	160	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	1,4-Dichlorobenzene	106-46-7	2.6	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.88	mg/kg	2) EPA Region 4 Soil	0.88	mg/kg	2) EPA Region 4 Soil
SW8260	2-Butanone (MEK)	78-93-3	2700	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	1	mg/kg	2) EPA Region 4 Soil	1	mg/kg	2) EPA Region 4 Soil
SW8260	2-Hexanone	591-78-6	20	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.36	mg/kg	2) EPA Region 4 Soil	0.36	mg/kg	2) EPA Region 4 Soil
SW8260	4-Methyl-2-pentanone (MIBK)	108-10-1	3300	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	443	mg/kg	3) RCRA ESLs	443	mg/kg	3) RCRA ESLs
SW8260	Acetone	67-64-1	6100	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	1.2	mg/kg	2) EPA Region 4 Soil	1.2	mg/kg	2) EPA Region 4 Soil
SW8260	Benzene	71-43-2	1.2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.12	mg/kg	2) EPA Region 4 Soil	0.12	mg/kg	2) EPA Region 4 Soil
SW8260	Bromochloromethane	74-97-5	15	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	15	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	Bromodichloromethane	75-27-4	0.29	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.54	mg/kg	3) RCRA ESLs	0.29	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	Bromoform	75-25-2	19	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.07	mg/kg	2) EPA Region 4 Soil	0.07	mg/kg	2) EPA Region 4 Soil
SW8260	Bromomethane	74-83-9	0.68	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.002	mg/kg	2) EPA Region 4 Soil	0.002	mg/kg	2) EPA Region 4 Soil
SW8260	Carbon disulfide	75-15-0	77	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.005	mg/kg	2) EPA Region 4 Soil	0.005	mg/kg	2) EPA Region 4 Soil
SW8260	Carbon tetrachloride	56-23-5	0.65	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.05	mg/kg	2) EPA Region 4 Soil	0.05	mg/kg	2) EPA Region 4 Soil
SW8260	Chlorobenzene	108-90-7	28	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	2.4	mg/kg	2) EPA Region 4 Soil	2.4	mg/kg	2) EPA Region 4 Soil
SW8260	Chloroethane	75-00-3	1400	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	1400	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)

Table A.3
Final Soil PALs
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	Human Health Project Action Limit			Ecological Project Action Limit			Final Project Action Limit		
			Value	Units	Reference	Value	Units	Reference	Value	Units	Reference
SW8260	Chloroform	67-66-3	0.32	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.05	mg/kg	2) EPA Region 4 Soil	0.05	mg/kg	2) EPA Region 4 Soil
SW8260	Chloromethane	74-87-3	11	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	10.4	mg/kg	3) RCRA ESLs	10.4	mg/kg	3) RCRA ESLs
SW8260	cis-1,2-Dichloroethene	156-59-2	16	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8260	cis-1,3-Dichloropropene	10061-01-5	1.8	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.001	mg/kg	2) EPA Region 4 Soil	0.001	mg/kg	2) EPA Region 4 Soil
SW8260	Cyclohexane	110-82-7	650	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	650	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	Dibromochloromethane	124-48-1	8.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	2.05	mg/kg	3) RCRA ESLs	2.05	mg/kg	3) RCRA ESLs
SW8260	Dichlorodifluoromethane	75-71-8	8.7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	39.5	mg/kg	3) RCRA ESLs	8.7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	Ethylbenzene	100-41-4	5.8	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.27	mg/kg	2) EPA Region 4 Soil	0.27	mg/kg	2) EPA Region 4 Soil
SW8260	Isopropylbenzene (Cumene)	98-82-8	190	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8260	m,p-Xylene	179601-23-1	55	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.1	mg/kg	2) EPA Region 4 Soil	0.1	mg/kg	2) EPA Region 4 Soil
SW8260	Methyl acetate	79-20-9	7800	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	7800	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	Methyl tert-butyl ether (MTBE)	1634-04-4	47	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	47	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8260	Methylcyclohexane	108-87-2	NA	---	NA	NA	---	NA	NA	---	NA
SW8260	Methylene chloride	75-09-2	35	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.21	mg/kg	2) EPA Region 4 Soil	0.21	mg/kg	2) EPA Region 4 Soil
SW8260	o-Xylene	95-47-6	65	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.1	mg/kg	2) EPA Region 4 Soil	0.1	mg/kg	2) EPA Region 4 Soil
SW8260	Styrene	100-42-5	600	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	1.2	mg/kg	2) EPA Region 4 Soil	1.2	mg/kg	2) EPA Region 4 Soil
SW8260	Tetrachloroethene (PCE)	127-18-4	8.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.06	mg/kg	2) EPA Region 4 Soil	0.06	mg/kg	2) EPA Region 4 Soil
SW8260	Toluene	108-88-3	490	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.15	mg/kg	2) EPA Region 4 Soil	0.15	mg/kg	2) EPA Region 4 Soil
SW8260	trans-1,2-Dichloroethene	156-60-5	7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8260	trans-1,3-Dichloropropene	10061-02-6	1.8	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.001	mg/kg	2) EPA Region 4 Soil	0.001	mg/kg	2) EPA Region 4 Soil
SW8260	Trichlorofluoromethane	75-69-4	2300	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	16.4	mg/kg	2) EPA Region 4 Soil	16.4	mg/kg	2) EPA Region 4 Soil
SW8260	Vinyl chloride	75-01-4	0.059	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.03	mg/kg	2) EPA Region 4 Soil	0.03	mg/kg	2) EPA Region 4 Soil
SW8270	1,2,4,5-Tetrachlorobenzene	95-94-3	2.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.18	mg/kg	2) EPA Region 4 Soil	0.18	mg/kg	2) EPA Region 4 Soil
SW8270	1,4-Dioxane	123-91-1	5.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	2.05	mg/kg	3) RCRA ESLs	2.05	mg/kg	3) RCRA ESLs
SW8270	2,2'-Oxybis(1-chloro)propane	108-60-1	310	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	19.9	mg/kg	3) RCRA ESLs	19.9	mg/kg	3) RCRA ESLs
SW8270	2,3,4,6-Tetrachlorophenol	58-90-2	190	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8270	2,4,5-Trichlorophenol	95-95-4	630	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	4	mg/kg	2) EPA Region 4 Soil	4	mg/kg	2) EPA Region 4 Soil
SW8270	2,4,6-Trichlorophenol	88-06-2	6.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	9.94	mg/kg	2) EPA Region 4 Soil	6.3	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	2,4-Dichlorophenol	120-83-2	19	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.05	mg/kg	2) EPA Region 4 Soil	0.05	mg/kg	2) EPA Region 4 Soil
SW8270	2,4-Dimethylphenol	105-67-9	130	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.04	mg/kg	2) EPA Region 4 Soil	0.04	mg/kg	2) EPA Region 4 Soil
SW8270	2,4-Dinitrochlorobenzene	97-00-7	NA	---	NA	0.6	mg/kg	5) Derived - See Appendix A.1	0.6	mg/kg	5) Derived - See Appendix A.1
SW8270	2,4-Dinitrophenol	51-28-5	13	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.061	mg/kg	2) EPA Region 4 Soil	0.061	mg/kg	2) EPA Region 4 Soil
SW8270	2,4-Dinitrotoluene	121-14-2	1.7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	6	mg/kg	2) EPA Region 4 Soil	1.7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	2,6-Dinitrotoluene	606-20-2	0.36	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	4	mg/kg	2) EPA Region 4 Soil	0.36	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	2-Chloronaphthalene	91-58-7	480	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.0122	mg/kg	3) RCRA ESLs	0.0122	mg/kg	3) RCRA ESLs
SW8270	2-Chlorophenol	95-57-8	39	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.06	mg/kg	2) EPA Region 4 Soil	0.06	mg/kg	2) EPA Region 4 Soil
SW8270	2-Methylphenol (o-Cresol)	95-48-7	320	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.1	mg/kg	2) EPA Region 4 Soil	0.1	mg/kg	2) EPA Region 4 Soil
SW8270	2-Nitroaniline	88-74-4	63	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.02	mg/kg	2) EPA Region 4 Soil	0.02	mg/kg	2) EPA Region 4 Soil
SW8270	2-Nitrophenol	88-75-5	NA	---	NA	1.6	mg/kg	3) RCRA ESLs	1.6	mg/kg	3) RCRA ESLs
SW8270	3,3'-Dichlorobenzidine	91-94-1	1.2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.03	mg/kg	2) EPA Region 4 Soil	0.03	mg/kg	2) EPA Region 4 Soil
SW8270	3-Nitroaniline	99-09-2	NA	---	NA	NA	---	NA	NA	---	NA

Table A.3
Final Soil PALs
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	Human Health Project Action Limit			Ecological Project Action Limit			Final Project Action Limit		
			Value	Units	Reference	Value	Units	Reference	Value	Units	Reference
SW8270	4,6-Dinitro-2-methylphenol	534-52-1	0.51	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.144	mg/kg	3) RCRA ESLs	0.144	mg/kg	3) RCRA ESLs
SW8270	4-Bromophenyl phenyl ether	101-55-3	NA	---	NA	NA	---	NA	NA	---	NA
SW8270	4-Chloro-3-methylphenol	59-50-7	630	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	7.95	mg/kg	3) RCRA ESLs	7.95	mg/kg	3) RCRA ESLs
SW8270	4-Chloroaniline	106-47-8	2.7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	1	mg/kg	2) EPA Region 4 Soil	1	mg/kg	2) EPA Region 4 Soil
SW8270	4-Chlorophenyl phenyl ether	7005-72-3	NA	---	NA	NA	---	NA	NA	---	NA
SW8270	4-Nitroaniline	100-01-6	25	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	21.9	mg/kg	3) RCRA ESLs	21.9	mg/kg	3) RCRA ESLs
SW8270	4-Nitrophenol	100-02-7	NA	---	NA	5.12	mg/kg	2) EPA Region 4 Soil	5.12	mg/kg	2) EPA Region 4 Soil
SW8270	Acetophenone	98-86-2	780	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	300	mg/kg	3) RCRA ESLs	300	mg/kg	3) RCRA ESLs
SW8270	Atrazine	1912-24-9	2.4	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.00005	mg/kg	2) EPA Region 4 Soil	0.00005	mg/kg	2) EPA Region 4 Soil
SW8270	Benzaldehyde	100-52-7	170	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	170	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	Benzyl butyl phthalate	85-68-7	290	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.59	mg/kg	2) EPA Region 4 Soil	0.59	mg/kg	2) EPA Region 4 Soil
SW8270	Biphenyl (Diphenyl)	92-52-4	4.7	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.2	mg/kg	2) EPA Region 4 Soil	0.2	mg/kg	2) EPA Region 4 Soil
SW8270	bis(2-Chloroethoxy) methane	111-91-1	19	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.302	mg/kg	3) RCRA ESLs	0.302	mg/kg	3) RCRA ESLs
SW8270	bis(2-Chloroethyl) ether (2-Chloroethyl ether)	111-44-4	0.23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	23.7	mg/kg	3) RCRA ESLs	0.23	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	bis(2-Ethylhexyl) phthalate	117-81-7	39	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.02	mg/kg	2) EPA Region 4 Soil	0.02	mg/kg	2) EPA Region 4 Soil
SW8270	Caprolactam	105-60-2	3100	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	NA	---	NA	3100	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	Carbazole	86-74-8	NA	---	NA	0.07	mg/kg	2) EPA Region 4 Soil	0.07	mg/kg	2) EPA Region 4 Soil
SW8270	Cresols, m & p	MEPH1314	320	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.08	mg/kg	2) EPA Region 4 Soil	0.08	mg/kg	2) EPA Region 4 Soil
SW8270	Dibenzofuran	132-64-9	7.8	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.15	mg/kg	2) EPA Region 4 Soil	0.15	mg/kg	2) EPA Region 4 Soil
SW8270	Diethyl phthalate	84-66-2	5100	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.25	mg/kg	2) EPA Region 4 Soil	0.25	mg/kg	2) EPA Region 4 Soil
SW8270	Dimethyl phthalate	131-11-3	NA	---	NA	0.35	mg/kg	2) EPA Region 4 Soil	0.35	mg/kg	2) EPA Region 4 Soil
SW8270	Di-n-butyl phthalate	84-74-2	630	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.011	mg/kg	2) EPA Region 4 Soil	0.011	mg/kg	2) EPA Region 4 Soil
SW8270	Di-n-octyl phthalate	117-84-0	63	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.91	mg/kg	2) EPA Region 4 Soil	0.91	mg/kg	2) EPA Region 4 Soil
SW8270	Hexachlorobenzene	118-74-1	0.21	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.079	mg/kg	2) EPA Region 4 Soil	0.079	mg/kg	2) EPA Region 4 Soil
SW8270	Hexachlorobutadiene	87-68-3	1.2	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.009	mg/kg	2) EPA Region 4 Soil	0.009	mg/kg	2) EPA Region 4 Soil
SW8270	Hexachlorocyclopentadiene	77-47-4	0.18	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.001	mg/kg	2) EPA Region 4 Soil	0.001	mg/kg	2) EPA Region 4 Soil
SW8270	Hexachloroethane	67-72-1	1.8	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.024	mg/kg	2) EPA Region 4 Soil	0.024	mg/kg	2) EPA Region 4 Soil
SW8270	Isophorone	78-59-1	570	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	139	mg/kg	3) RCRA ESLs	139	mg/kg	3) RCRA ESLs
SW8270	Nitrobenzene	98-95-3	5.1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	2.2	mg/kg	2) EPA Region 4 Soil	2.2	mg/kg	2) EPA Region 4 Soil
SW8270	n-Nitrosodi-n-propylamine	621-64-7	0.078	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.544	mg/kg	3) RCRA ESLs	0.078	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	n-Nitrosodiphenylamine	86-30-6	110	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.545	mg/kg	2) EPA Region 4 Soil	0.545	mg/kg	2) EPA Region 4 Soil
SW8270	Pentachlorophenol	87-86-5	1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	2.1	mg/kg	1) EPA Eco SSLs	1	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)
SW8270	Phenol	108-95-2	1900	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.79	mg/kg	2) EPA Region 4 Soil	0.79	mg/kg	2) EPA Region 4 Soil
SW8330A	Picric acid	88-89-1	13	mg/kg	6) EPA Nov 2020 Resident Soil RSL (THQ = 0.1)	0.0061	mg/kg	5) Derived - See Appendix A.1	0.0061	mg/kg	5) Derived - See Appendix A.1

Notes:

Highlighting = Value selected for Final PAL.

Chromium by method SW6020 is total chromium. Trivalent chromium values presented.

Cresols, m & p values: The more conservative value from the two isomers is presented. Human health = m-cresol and ecological = p-cresol.

Aluminum is identified as an ecological COPC only for those soils with a pH less than 5.5. The technical basis for this procedure is that the soluble and toxic forms of aluminum are only present in soil under soil pH values of less than 5.5. Site-specific considerations could, however warrant inclusion of aluminum as a COPC.

Table A.3
Final Soil PALs
New York Ordnance Works
Onondaga, NY

Method	Analyte	CAS Registry Number	Human Health Project Action Limit			Ecological Project Action Limit			Final Project Action Limit		
			Value	Units	Reference	Value	Units	Reference	Value	Units	Reference

1,3-Dichloropropene (total) values presented.

Iron narrative statement (EPA Eco SSL, 2003): The main concern from an ecological risk perspective for iron is not direct chemical toxicity per se, but the effect of iron as a mediator in the geochemistry of other (potentially toxic) metals and the potential physical hazard of depositing flocculent. Soil pH and Eh should be included as standard soil chemical field parameters when conducting any field investigation. Although some EPA analytical methods require the collection of soil pH prior to analysis, this information is not always made available to the ultimate data users. In addition, the pH measured in the lab may no longer be representative of field conditions since the samples can be exposed to oxygen during storage prior to analysis. A determination of the geochemical conditions (i.e., pH and Eh at a minimum) of the environmental setting, as well as the presence of iron floc and the toxic metals, is critical to the determination of the relative importance of iron at a site.
m-Xylene used for human health m,p-xylene value.
Xylenes (total) value used for each isomer for the ecological PALs.
EPA Eco SSL - Environmental Protection Agency Ecological Soil Screening Values.
HMW PAHs = High molecular weight polycyclic aromatic hydrocarbons.
LANL = Los Alamos National Laboratory.
LMW PAHs = Low molecular weight polycyclic aromatic hydrocarbons.
mg/kg = Milligrams per kilogram.
NA = Not available.
PAL = Project Action Limit.
RCRA = Resource Conservation and Recovery Act.

Sources:

1) EPA Ecological Soil Screening Levels (EcoSSLs; various dates) (https://www.epa.gov/chemical-research/interim-ecological-soil-screening-level-documents)
2) EPA Region 4 Ecological Risk Assessment Supplemental Guidance (2018) – Soil Screening Values for Hazardous Waste Sites (https://www.epa.gov/sites/production/files/2018-03/documents/era_regional_supplemental_guidance_report-march-2018_update.pdf).
3) EPA Region 5 (2003) RCRA Ecological Screening Levels (ESLs) (https://archive.epa.gov/region5/waste/cars/web/pdf/ecological-screening-levels-200308.pdf)
4) Los Alamos National Laboratory (LANL) ECORISK Database Ecological Screening Levels (2017) (https://www.lanl.gov/environment/protection/eco-risk-assessment.php)
5) Derived - See Appendix A.1
6) EPA Regional Screening Levels (RSL) Resident Soil (Total Hazard Quotient [THQ] = 0.1, Target Risk [TR] = 1E-06) (November 2020) (https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables)

THIS PAGE LEFT BLANK INTENTIONALLY

APPENDIX B

Field SOPs

STANDARD OPERATING PROCEDURE NO. 01
ABANDONMENT OF MONITORING WELLS AND PIEZOMETERS

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology to properly abandon (remove) a monitoring well or piezometer using a standard drill rig. The associated rationale and project objectives (i.e., locations and depths of wells to be abandoned, drilling methods, and final site restoration) should be detailed in the Work Plan and/or Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP). Bluestone does not self-perform well abandonment activities; therefore, the requirements described in this SOP apply to the oversight and QC of work performed by subcontractors.

The abandonment of monitoring wells and piezometers is regulated in most states. It is the responsibility of both the Project Manager and site personnel to ensure that work is performed in accordance with applicable State and Federal regulations, that the driller is properly licensed for work in that state, and that required paperwork is completed by the responsible party (typically the drilling contractor) and submitted to the proper regulatory agency.

Monitoring wells and piezometers are similar in construction but are intended for different uses: monitoring wells are designed for the long-term monitoring of groundwater quality while piezometers are designed for monitoring groundwater elevation. Throughout this SOP, the term ‘monitoring well’ is used interchangeably with ‘piezometer’.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident

Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards created by drill rigs and associated drilling tasks include working around heavy machinery, excessive noise, overhead rotating parts, pinch points, heavy lifting, projectiles, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with drilling tasks, and includes a hard hat, safety glasses, steel-toed boots, leather and/or nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 WELL ABANDONMENT

Monitoring wells and piezometers are typically installed at project sites to evaluate hydrogeologic conditions and determine the presence of environmental contaminants in groundwater. When a well or piezometer is no longer needed, or has been damaged or compromised, it should be properly removed and/or sealed (i.e. abandoned). If not properly abandoned, monitoring wells and piezometers may provide pathways for contaminant migration in the subsurface. In addition, deserted monitoring wells and piezometers have the potential to become a physical hazard to both humans and animals.

Monitoring well abandonment consists of either removing the components of a monitoring well and backfilling the borehole with a relatively impermeable material; or simply backfilling the screen and casing in place with relatively impermeable fill material. For either case, the surface expression of the well (i.e., flush-mount cover or stick-up casing and protective bollards) are removed. The selected method of abandonment must consider the size of the monitoring well, construction materials and current condition; total depth; hydrogeologic setting; known impacts; and regulatory requirements, as applicable. Specifications for methods used and the corresponding rational should be detailed in the Work Plan.

5.0 CAUTIONS

Well abandonment is a fairly standard process but has the potential to become complex during the progression of tasks. This may be due to a variety of circumstances, including unknown well conditions, data gaps in the surrounding lithology, etc. Site personnel should be prepared for

issues that may arise and remain in communication with the Project Manager and/or regulator as needed during the progression of work.

6.0 DEFINITIONS

For this SOP the following definitions apply:

- **Annulus** - The cylindrical space between the well casing and surrounding borehole.
- **Bentonite** - Any type of commercial sodium bentonite clay used in the construction or sealing of groundwater wells.
- **Bentonite Cement Grout** - A cement grout slurry, generally consisting of one 94-pound bag of Portland cement, mixed with approximately seven gallons of potable water and two pounds of bentonite.
- **Borehole** - Any hole drilled into the subsurface for the purpose of identifying lithology, collecting soil samples, and/or installing groundwater wells.
- **Casing/Riser** - An impervious durable pipe placed in a borehole, which extends from the top of the well screen to ground surface. The casing/riser pipe protects the well from collapse of the surrounding formation and provides a seal from upper level water-bearing zones and/or surface contaminants. Well casing/riser pipe is typically composed of polyvinyl chloride (PVC), or stainless steel.
- **Filter Pack** - Granular sediment (i.e., sand or fine gravel) placed in the well annulus around the screened portion of casing to increase the effective diameter of the well and prevent fine-grained sediment from entering the well.
- **Monitoring Well** - A groundwater well installed for the purpose of collecting representative groundwater quality samples and groundwater elevation data. In addition, monitoring wells may be used to determine the presence (or absence) of light or dense non-aqueous phase liquids (LNAPL and DNAPL, respectively), and for measurement of free product levels in a contaminant plume.
- **Piezometer** - A shallow, small-diameter groundwater well installed for the purpose of obtaining groundwater elevation data and evaluating hydrogeologic properties pertaining to the interaction between shallow water-bearing zones and local surface water features.
- **Project-Specific Work Plan** - A plan that details the scope of work, rationale and techniques to be employed at the Site to achieve the project objectives. Work Plans may include field sampling plans, UFP-QAPP, technical memorandums, and other documentation of

proposed work.

- **Tremie pipe -** A section of 1 to 2-inch PVC pipe used during well or piezometer construction or abandonment. The Tremie pipe extends to the bottom of the borehole, and is used to place grout, bentonite, and/or the filter pack in the annulus continuously from the bottom of the borehole to the surface. This method prevents any bridging or gaps in the well anulus or borehole during the backfilling process.
- **Well Screen** – The prefabricated component of a well that is designed to maximize the entry of water from the producing zone, while minimizing the entrance of sand from the filter pack. Well screens may be constructed of stainless-steel mesh (i.e., wire-wound or continuous-wrap), or slotted PVC casing, and vary in length depending on hydrogeologic conditions.

7.0 EQUIPMENT AND SUPPLIES

Equipment used during the oversight and direction of monitoring well abandonment may include the following:

- Water level meter (or interface probe)
- Fiberglass or steel measuring tape with weight
- Field logbook and appropriate field forms
- PPE and safety equipment per the Project-Specific Safety and Health Plans.

8.0 WELL ABANDONMENT PROCEDURES

Monitoring well abandonment will be performed by a subcontractor to Bluestone in accordance with applicable Federal, State, and local regulatory requirements. For sites overseen by the U.S. Army Corps of Engineers, well abandonment procedures must follow the guidance document EM 1110-1-4000, *Engineering and Design – Monitor Well Design, Installation, and Documentation at Hazardous, Toxic, and Radioactive Waste Sites* (USACE, 1994). To the extent practicable, Bluestone will adhere to any Client requests regarding safety and aesthetics at the well site. All wells will be abandoned in accordance with applicable state regulations.

8.1 Inspection and Preparation for Well Abandonment

Prior to the start of well abandonment activities, Bluestone will inspect the well head and surrounding area for any visible damage or disturbance. Once the well is accessed, Bluestone will record any observations made with respect to the condition of the well, and record depth-to-

water and total depth measurements. Any dedicated equipment inside the well casing, such as *in-situ* pumps or sampling tethers must be removed.

8.2 Well Abandonment with Casing Removal

To achieve a competent seal in the subsurface, and subsequently reduce the risk of contaminant migration through the well site, it is recommended that both the casing and well screen are removed from the subsurface. This applies to any sites with known environmental impacts, any damaged well that is suspected of having a compromised seal, and for any well cased through multiple water-bearing zones. Well casings can be removed in a variety of methods, as described below.

8.2.1 Removal of Casing by Pulling

Shallow monitoring wells may be deconstructed by pulling up or by gently bumping the casing with the drill rig. The condition of the screen and casing must be known, and structurally sound prior to applying force. Crews should be prepared with a grout slurry and Tremie pipe in the event the borehole begins to collapse as the screen and riser pipe are removed. Casing removal and backfilling should be conducted in sequence to ensure a proper subsurface seal.

8.2.2 Removal of Casing by Over-drilling

Over-drilling is a technique ideal for the removal of small-diameter wells. The process involves the advancement of large-diameter hollow-stem augers around the well casing to a depth slightly greater than the total depth of the well to ensure that all components are recovered. Once the target depth is reached, the casing and screen are removed, and the borehole is Tremie-grouted to grade.

8.2.3 Removal of the Casing by Drilling through the Well

Well abandonment by drilling through a well can be done only on wells constructed with plastic or Teflon® materials. For this method, a solid-stem auger or rotary bit is advanced though the casing, which destroys the materials and brings the cuttings to the surface. The bit must be larger than the diameter of the original borehole and should be advanced to a depth slightly greater than the total well depth. Extra care and caution must be used to ensure that the bit is centered during the advancement through casing. Cuttings can be difficult to removed using this method.

8.2.4 Backfilling of a Borehole

The backfilling of boreholes may be performed using a variety of materials, include bentonite pellets, bentonite chips, high solids bentonite grout, cement slurry, etc. Any materials intended for backfilling must be approved for environmental use. In general, a borehole should be backfilled to a minimum of three feet below ground surface (ft bgs). This depth is based on the expanding properties of bentonite/cement grout slurry as the materials sets inside the borehole. Bluestone field personnel should verify the quantities of the mixture/slurry and document the amount of materials used.

Boreholes should be backfilled by lowering a Tremie pipe to the bottom of the borehole. The pipe is slowly raised as the grout slurry fills the borehole from the bottom to the surface, while keeping the end of the Tremie pipe submerged below the surface of the slurry. Bentonite pellets or chips may be poured through a Tremie pipe; however, the pellets/chips should be hydrated every foot to ensure a proper seal. Bentonite pellets/chips should not be poured from the surface into a borehole with a large water column, as the material may hydrate too quickly and create voids or bridged sections in the borehole

After a period of 24 hours, the abandoned borehole should be inspected for grout settlement. If the top of grout is greater than three feet bgs, an additional volume of grout should be pumped into the borehole. No Tremie pipe is required for this step if the distance between ground surface and the top of slurry is less than 15 feet, at which point the additional grout can be poured into the borehole from the surface.

8.3 Well Abandonment by Sealing in Place

Monitoring wells may be sealed without casing removal when the construction details are known, the annular seal is intact, and the filter pack does not penetrate more than one water-bearing zone. Some environmental sites or wells may be too hazardous to safely remove casing; therefore, well abandonment by sealing the well in place is appropriate.

Situations that warrant sealing in place over casing removal must be evaluated in advance and may require a formal request for variances from regulatory agencies. Depending on State regulations and site conditions, it may be appropriate to perforate the casing prior to sealing the

well. If applicable, the procedures for the perforation of well casing will be addressed in the Work Plan.

The backfill requires the use of grout consisting of a bentonite cement, bentonite slurry, or bentonite chips/pellets installed through a Tremie pipe. The on-site person will check and document the materials used including brand name and amount, and, if using a slurry, that the grout was mixed per the manufacturer's directions. Grout should be approved for environmental use.

8.4 Site Restoration

To the extent practicable, the site should be restored to its original condition at the completion of well abandonment activities. The borehole should be finished to grade with like materials (i.e., topsoil, asphalt patching, or concrete). In addition, any voids created by the removal of stick-up casing and/or the concrete pad should be filled accordingly. Areas surrounding the well and any pathways that were disturbed by accessing the well may require regrading, placement of sod, or re-seeding, as preferred by the Client. The disposal of well construction materials and drill cuttings will be handled as investigation-derived wastes (IDW). The characterization and disposal of IDW is discussed in Bluestone's SOP No. 10 – *Storage and Sampling of IDW*.

9.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

10.0 DATA MANAGEMENT

Data collected during well abandonment activities (i.e., depth to groundwater, total well depth, volume of materials used for production of grout, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

11.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 02
EQUIPMENT CALIBRATION AND MAINTENANCE

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for performing routine calibrations of equipment used in environmental investigations, in addition to the necessary long-term care and maintenance of equipment.

[NOTE: Bluestone SOP No. 2 incorporates a project-specific equipment list.]

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated calibration and equipment maintenance include splashing from calibration fluids and operating compressed gas canisters, and gas fumes, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for equipment calibration and maintenance, and includes safety glasses, safety shoes and nitrile gloves. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 PROJECT-SPECIFIC EQUIPMENT

4.1 Water Level Indicator

Procedures for the operation and maintenance (O&M) of a water level indicator is described in the following sections. The operation manual supplied by the manufacturer should be reviewed for instructions concerning the variations pertinent to specific equipment brands and models.

4.1.1 Calibration

Factory calibration procedures should be performed on a quarterly basis by the rental agency to determine if the measuring scale on the water level indicator is accurate. Calibration procedures consist of verifying water level measuring increments against a calibrated device.

4.1.2 Operation

The following steps should be completed to operate the water level indicator:

1. Unlock well protective casing and remove well cap.
2. Lower decontaminated water-level probe into well.
3. Continue lowering probe until indicator sounds and light is illuminated.
4. Confirm that the water surface has been contacted by repeatedly raising and lowering the probe a few hundredths of an inch until a consistent sounding level has been achieved.
5. Mark the line of indicator (where water surface has been contacted) by pinching the line between the thumb and forefinger while holding the line level with the measuring point at the well head.
6. Measure and record sounding depth (nearest 0.01 foot) in the field book or gauging sheet.
7. Lower the indicator to tag the well bottom and record total depth of well. Note in the field book or gauging sheet whether the bottom feels hard or soft (degree of silting).
8. Retrieve the water level probe and decontaminate the meter in accordance with procedures outlined in SOP 05 – *Field Equipment Decontamination Procedures* prior to the next use. Note any observations in the field book or gauging sheet, including the presence of silt or residual product and odors on the probe.

4.1.3 Preventative Maintenance

The water level indicator should be rinsed with soapy water, followed by DI water after each use to avoid cross contamination between groundwater wells. Solvents may be used sparingly to aid in the removal of contamination. The probe should be kept free of silt and product coatings. Dye-free paper towels can be used to wipe the probe, and any solvents used should not be sprayed directly onto the probe.

4.2 Water Quality Meter – YSI Pro Plus (or similar model)

The procedures for calibration and the O&M of the YSI Pro Plus multiparameter meter is described in the following sections. This meter may be used to measure pH, specific conductance, temperature, turbidity, oxidation-reduction potential (ORP), and dissolved oxygen (DO). The operation manual supplied by the manufacturer should be consulted for instructions concerning the operation of other water quality parameters.

4.2.1 Calibration

Calibration should be performed each day prior to sample collection activities or when excess variability is noted. The YSI Pro Plus requires a one-point calibration for specific conductance and ORP, a two-point calibration for turbidity and a three-point calibration for pH. A separate calibration solution is required for most of the parameters, although one solution may be used to calibrate multiple parameters (that is, a 7 pH and 0 NTU turbidity solution). Each parameter will be calibrated on an individual basis as outlined in the instruction manual.

4.2.2 Operation

The following steps should be completed to operate the YSI Pro Plus multiparameter meter:

1. Activate instrument by pressing the [POWER] key.
2. Based on the intended use, attach the flow-through cell or the threaded protective cage around the probes.
3. Gently place the probe into the water sample or connect the tubing from the well to the input port of the flow-through cell.
4. Record measurements in the field logbook or purge log.

Additional operational features are available with the YSI Pro Plus meter. The instruction manual can be reference as needed for O&M procedures.

4.2.3 Preventative Maintenance

The YSI Pro Plus meter should be cleaned and inspected daily before and after use. The batteries should be replaced frequently, and the electrodes should be replaced when cracked or when the instrument cannot maintain a calibration to the manufacturer specifications. The pH sensor must be kept moist by filling the small rubber cap with water and storing the electrode with the cap attached.

4.3 Photo-Ionization Detector (MiniRAE® 3000, or similar model) & Multi-Gas Meter (MultiRAE®, or similar model)

Procedures for the calibration and O&M of a MiniRAE® 3000 photo-ionization detection (PID) and a MultiRAE® multi-gas meter are described in the following sections. At a minimum, these meters should be calibrated daily; however, more frequent calibrations may be necessary if the instrument begins to drift from background (0 parts per million [ppm]). Justification and rationale for performing additional calibrations should be noted in the field book. Directions for calibrating are listed below, as available in the operation manual supplied by the manufacturer.

4.3.1 Calibration

To calibrate the MiniRAE® PID meter:

1. Activate the MiniRAE® unit by pressing the [MODE] key.
2. The unit will perform a self-diagnostic routine.
3. After the MiniRAE® has gone through its self-diagnostic routine, simultaneously press [N/-] and [MODE] keys for three seconds. This will put the meter in Programming Mode.
4. The meter will prompt “Calibrate /Select Gas”. Press [Y/+] key.
5. The meter will prompt “Fresh air cal?” Press [Y/+] key. The display shows “zero in progress” followed by “wait...” and a countdown timer.
6. After about 15 seconds, the display will show the message “update data…zeroed…reading = X.X ppm…”. Record this reading in the field book or calibration log sheet. Note: Make sure the Fresh Air Cal is done outside because this is the value the meter will use as zero.
7. The meter will prompt “Span cal?’ Press [Y/+] key.
8. The meter will prompt if the cal gas is Isobutylene. Press [Y/+] key.
9. The meter will then ask the user to “Apply gas now!” Note: Make sure the meter is connected to the Isobutylene canister and open the pressure valve to release the gas.
10. The display will show “wait…30” with a countdown timer showing the number of remaining seconds. When the countdown timer reaches 0, the display shows the calibrated value. Record this value in the field book or calibration log sheet.
11. The reading should be close to the actual concentration of the gas. If not, wait a few seconds and press the [Mode] key. This process may need to be repeated until the calibration gas as stabilized to 1 to 2 parts per million (ppm) with the calibration gas range.

12. Press the [MODE] key to exit the standard gas calibration mode, turn off the gas, and disconnect the calibration gas from the MiniRAE®. The calibration gas used for calibration is isobutylene at a concentration of 100 ppm. The use of this calibration gas will result in a reading of 100 ppm during the calibration mode.

4.3.2 Operation

The following steps should be completed to operate the MiniRAE® PID meter:

1. Activate the MiniRAE® unit by pressing the [MODE] key.
2. The unit will perform a self-diagnostic routine and ". dIAG" will be displayed during this operation.
3. After 30 seconds, the display will change to real-time readings of the gas concentrations.
4. Observe concentrations and record in the field book.
5. After the necessary measurements have been observed and recorded, turn the MiniRAE® off by pressing and holding [MODE] key for 5 seconds.

4.3.3 Preventative Maintenance

The MiniRAE® unit should be inspected at the end of each workday. In addition, the battery must be charged for each day of use.

[NOTE: Due to instrument complexity, the use of a hand-held Global Positioning Survey (GPS) unit is discussed separately in Bluestone's SOP No. 04 – *GPS Survey*.]

5.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

6.0 DATA MANAGEMENT

Equipment calibration values will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 03
DESIGN AND INSTALLATION OF MONITORING WELLS AND WELL DEVELOPMENT

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for the installation of monitoring wells and well development. Information obtained during the installation of monitoring wells, include soil type, lithology, hydrogeologic data (i.e., water-bearing zones, groundwater elevation and gradient), fracture zones, and visual evidence of contamination must be compiled and interpreted by the site geologist. Copies of geologic field logs and formal Well Construction Logs will be prepared and compiled for reporting purposes. In accordance with local or State regulatory agencies, the drilling contractor must register the monitoring well or piezometer in the appropriate drilling database.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards created by drill rigs and associated drilling tasks include working around heavy machinery, excessive noise, overhead rotating parts, pinch points, heavy lifting, projectiles, biological hazards (i.e., poison ivy, bees/wasps, spiders, ticks), etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with drilling tasks, and includes a hard hat, safety glasses, steel-toed boots, leather and/or nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the

APP and/or HASP.

4.0 MONITORING WELL INSTALLATION PROCEDURES

Monitoring wells and piezometers installed during field investigations must be constructed in accordance with the Work Plan and/or a Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP), in addition to and any local or State regulations. Additional guidance may be provided by the Client. Drilling locations will be determined in advance and will be cleared of subsurface utilities prior to mobilization (refer to Bluestone's SOP No. 18 – *Utility Clearance*). The site geologist should carry a copy of the applicable guidance documents for reference during work activities. The following installation procedures and construction requirements will be implemented:

1. Monitoring wells/piezometers will be constructed with National Sanitation Foundation (NSF) potable water grade, flush-threaded PVC riser and screen that conforms to American Society of Testing and Materials (ASTM)-D 1785 standards. Riser pipe and screen will be packaged in containers that bear the manufacturer's seal.

2. PVC monitoring well/piezometer screen and riser pipe will be flush-threaded. The joints will be tightened to form a watertight seal. Screen bottoms will be sealed with a flush-threaded PVC cap or slip-on cap secured with three, stainless-steel, self-tapping screws. No glues or solvents shall be used in the construction of PVC monitoring wells or piezometers.

3. PVC well screen shall be factory slotted and sized to be compatible with the respective filter pack and surrounding formation media. Field slotted or cut screens will not be used.

4. The well annulus must be a minimum of 2 inches wide to provide space for tools and well construction materials.

5. Boreholes will be checked for plumbness and alignment prior to monitoring well construction. A 10-foot section of pipe, ½ to ¾-inch smaller in diameter than the inner diameter of the riser pipe and screen will be extended through the entire length of the well to check the alignment.

6. Centralizers will be used to stabilize the casing inside the borehole. It should be noted that no centralizers will be installed in the filter pack or bentonite seal, and that centralizers will not be used on wells installed using hollow stem auger (HSA) methods, or in wells less than 30 feet deep.

7. The filter pack will consist of clean, inert, silica-based, homogenous sand. The filter pack will be installed from the bottom of the borehole to at least 2 feet above the top of the well screen to allow for settlement. The site geologist will confirm that the filter pack is properly placed, and that no bridging or gaps are present. This task will be accomplished by frequently recording borehole depth measurements during installation using a weighted tape measure.

8. Approximately two feet of bentonite pellets or chips will be placed in the annulus above the sand pack, to create a proper well seal. The bentonite will not be Tremie-piped into place within the borehole but will be poured from the ground surface into the annulus and continually checked with a weighted tape to verify that no bridging has occurred. When the interval of bentonite is above the water table, the pellets or chips will be hydrated every foot using non-chlorinated potable water. The bentonite seal will be a minimum of 2 feet thick.

9. The bentonite pellets and chips will be allowed to hydrate for at least a 4-hour period.

10. A high-solids bentonite grout slurry mixed to manufacturer's specifications will be used to fill the annulus above the bentonite seal to within 3 feet of the ground surface. The grout slurry will be emplaced using a Tremie pipe with a side discharge port. The grout may be poured into the borehole from the surface if the depth to the bentonite seal is less than 15 feet and the top of the bentonite seal is above the water table. The grout will be allowed to cure for a minimum of 24 hours before well development begins. If the top of the primary seal is above the water table, bentonite chips or pellets may be used to backfill the remainder of the borehole, rather than grout.

11. When the grout has cured, a concrete pad will be installed around the monitoring well/piezometer. The surface of the concrete pad will slope gently away from the protective cover to prevent ponding of rainwater at the well head. A permanent well label will be placed in the concrete pad, with the well number stamped on the label.

12. Depending on the location of the well (and Client preference), the well head may be finished as either a flush mount well or with stick-up casing. In either case, the well will be equipped with an expandable plug in the rise pipe and a protective outer cap and lock.

13. If necessary, three or four 3 inch-diameter steel or concrete-filled bollard posts may be placed around the concrete pad as an extra measure of protection for the stick-up well head. The bollards will be positioned at least 2 to 3 feet outside the well pad and equally spaced. The bollards must be driven a minimum of 2-ft bgs and extend at least 3 feet above ground surface. Stick-up casings and bollards should be painted with highly visible colors.

14. At locations where a monitoring well/piezometer is targeted but groundwater is not apparent in the unconsolidated overburden during drilling, the borehole may be left open

for a period of 24 hours to monitor for seepage. The borehole will be bermed and covered during this period to reduce the potential for entry of surface water runoff or contamination. After the 24 hour-period (unless specified otherwise in the Work Plan or UFP-QAPP), the site geologist will consult with the Project Manager to determine if the borehole should be advanced further.

15. Prior to the collection of groundwater samples, a monitoring well must be developed.

4.1 Screen Size and Filter Pack

Screen size and length and filter pack size will be specified in the Work Plan or UFP-QAPP.

5.0 TYPES OF MONITORING WELLS

5.1 Overburden Monitoring Wells

An overburden monitoring well refers to a well constructed in unconsolidated material. Depending on depth, overburden wells may penetrate multiple water-bearing zones. The following sections describe various types of overburden wells and the rationale for installation based on local lithology. Site-specific details for construction of overburden wells should be included in the Work Plan or UFP-QAPP.

5.2 Water Table Monitoring Wells

Water table wells are ideal for the evaluation of groundwater quality in the shallow subsurface, with known impacts of volatile and/or semi-volatile organic compounds (VOCs and SVOCs, respectively) with a specific gravity less than 1.0. These 'light' compounds are referred to as light non-aqueous phase liquids (LNAPLs) and tend to accumulate on the surface of the water table. Wells designed to monitor the presence of LNAPL must be screened across the top of the water table and have sufficient screen length to accommodate the seasonal fluctuation of local groundwater. To install water table monitoring wells, the borehole is advanced to the target depth below the water table, to drilling refusal, or to bedrock, whichever is encountered first, and the screened interval is centered across the water table surface.

5.3 Monitoring Wells with Submerged Screens

Submerged screen wells are ideal for monitoring groundwater impacted with dense non-aqueous phase liquid (DNAPL) constituents, which have a specific gravity greater than 1.0, and therefore may accumulate at the bottom of the well. The total depth of the well should be estimated prior

to drilling, by evaluating available information (i.e., geophysical survey interpretations, adjacent borehole data, published literature, etc.).

To construct a submerged screen well, the borehole is advanced to the target depth, or refusal, whichever is encountered first, and the bottom of the screen is set at or directly below the overburden/bedrock interface. In some cases, the well boring may be terminated at a predetermined depth for the purpose of intersecting a specific water-bearing zone. The monitoring well is constructed in the same manner as described previously.

5.4 Bedrock Monitoring Wells

Bedrock monitoring wells are ideal for monitoring particular fracture zones or deep water-bearing zones. Bedrock monitoring well construction requirements include the following, in addition to the items listed above:

1. Prior to the start of drilling, the depth of the overburden/bedrock interface should be estimated using available information (i.e., geophysical survey interpretations, adjacent borehole data, and published literature, etc.).
2. The borehole must be advanced at least five feet into competent bedrock.
3. A 6-inch stainless-steel surface casing is installed through the unconsolidated overburden and set at a minimum of five feet into competent bedrock. The casing maintains the integrity of the borehole and isolates the bedrock zone from groundwater in the overburden while drilling into underlying bedrock.
4. If the scope of work includes bedrock coring, the rock core will be obtained as detailed in the Work Plan and/or UFP-QAPP. Once the core is extracted, the borehole is reamed to a nominal 6-inch diameter to the target depth.
5. If no bedrock core is needed, a nominal 6-inch diameter roller bit, or similar is advanced to the target depth.
6. Screen will be installed at the bottom of the borehole, as detailed in the Work Plan and/or UFP-QAPP. Depending on the lithology, some bedrock wells may be finished as open rock wells, and do not require a screen.
7. For screened bedrock wells, the filter pack should extend to 3 feet above the top of the screen.

6.0 DEVELOPMENT OF MONITORING WELLS AND PIEZOMETERS

Newly installed monitoring wells and piezometers, or wells that have been damaged must be developed to remove excessive fine particles and sediment from the well screen and filter pack. Well development methods vary; however, this SOP describes the procedures for swabbing with a surge block or similar apparatus, followed by pumping and/or bailing the well. Swabbing consists of raising and lowering a surge block inside the casing, while targeting the screened interval. Caution should be exercised when swabbing within the screened interval so as not to damage the screen. The sediment load and volume of water removed from the well should be monitored and recorded regularly until the well is fully developed. The development of a monitoring well should be initiated between 48 hours and seven days after well installation is complete. The initial static water level and total well depth will be measured and recorded on the field logbook and/or field data sheets (refer to Bluestone's SOP No. 06 – *Field Documentation*). In addition, the length of the water column should be recorded.

The pH, specific conductance, temperature, and turbidity of the water will be recorded before the start of well development. After several volumes of water have been removed, a second set of water quality parameters will be recorded. The data will be recorded in the field logbook and/or field data sheets (refer to Bluestone's SOP No. 06 – *Field Documentation*). Well development activities will continue until the well or piezometer stabilizes, based on the attainment of specified standards for turbidity units, pH, specific conductance, and temperature. The required standard criteria for water quality parameters are detailed in the Work Plan and/or UFP-QAPP.

The well development sequence is as follows:

1. Collect water sample and record pH, specific conductance, temperature, and turbidity.
2. Record the depth-to-water and total well depth.
3. Swab the well with a surge block for a 10 to 15 minute-period.
4. Re-measure and record the total well depth.

5. Bail and/or pump the well to remove any suspended sediment in the water column.

6. Repeat Steps 3-5 until the water bailed or pumped meets the required turbidity standard set forth in the Work Plan and/or UFP-QAPP. Values for pH, specific conductance, and temperature must not vary by more than 10 percent. (At a minimum, three to five times the volume of any water introduced during drilling and installation must be removed. Monitoring wells or piezometers that are purged dry during development activities will be purged dry three times and considered adequately developed).

7. If stabilization of the water quality parameters cannot be achieved, the site geologist will consult with the Project Manager and cease well development after a reasonable effort has been made. Stabilization of parameters is typically obtainable; however, elevated turbidity readings may persist due to a variety of circumstances.

8. Once well development is completed, a sample of the last development water withdrawn from the well will be collected in a clear glass jar and immediately photographed for documentation purposes. The color photograph will be back lit and taken within close range. A sample label must be visible in the photograph.

The disposal of well construction materials, drill cuttings, and development waters will be handled as investigation-derived wastes (IDW). The characterization and disposal of IDW is discussed in Bluestone's SOP No. 10 – *Storage and Sampling of IDW*.

7.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

8.0 DATA MANAGEMENT

Data collected during well construction and development activities (i.e., depth to groundwater, total well depth, volume of materials used for production of grout, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 04
GLOBAL POSITIONING SURVEY (GPS)

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the technical guidance and procedures that will be employed to conduct oversight of a Global Positioning Survey (GPS) Survey by a licensed Surveyor. Conventional land and GPS survey techniques will be used to survey monitoring well locations and elevations; and locations of a variety of sample types (i.e., soil, sediment, biological, surface water, etc.), borehole locations and elevations; geophysical and sampling grids, elevations and orientations; utility clearance (as applicable); and other surface and subsurface features. Bluestone will be responsible for the oversight and documentation of all surveying activities; however, the subcontractor will be responsible for the collection and interpretation of all survey data. In addition, the subcontractor must provide a SOP and perform the investigation in accordance with the policies and procedures set forth by their respective company.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with performing a GPS survey include working around heavy machinery, excessive noise, slip/trip/fall hazards, biological (i.e., poison ivy, bees/wasps, spiders, ticks), etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for the associated tasks, and may include a hard hat, safety glasses, safety shoes, leather gloves, high-visibility vests, and

hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 EQUIPMENT LIST

The following survey equipment may be required for conducting a GPS survey:

- Field logbook, field data sheets
- Indelible black-ink pens
- GPS Instrument set to the appropriate coordinate system
- Field map
- Measuring Tape
- Compass/Brunton
- Reference coordinates and datum

5.0 EQUIPMENT SET-UP AND OPERATION

This section provides a general summary of GPS surveying procedures for surveying sample locations and geophysical survey grid points. These procedures shall be supplemented by the specific survey instrument manufacturer's recommendations and generally accepted surveying practices.

Each operator will record the specific references (Universal Transverse Mercator [UTM] coordinates and datums) used for the survey points. It is imperative to identify the coordinate system used to complete the survey. The coordinate system must appear on the data entry form. UTM coordinates will be recorded as Easting and Northing. Land surveying control will be established from known National Geodetic Survey (NGS) benchmarks, using the New Mexico State Plane, Central Zone. The vertical datum will be the North American Vertical Datum of 1988. Details including the manufacturer, model, and unit number of the GPS used in the survey must be recorded in the field logbook.

If the coordinates at a survey location cannot be determined due to the presence of tree cover, or other sources of interference/obstructions that prohibit adequate signal reception, coordinates will be obtained at a minimum of two alternate locations (off-sets) in close proximity

to the original survey location. The distance and bearing from each of the alternate locations to the original survey location will be manually measured using a measuring tape and compass and recorded on the field sample form and the field logbook.

6.0 SURVEY DOCUMENTATION

Documentation of observations and data collected in the field provides a permanent record of field activities. Logbook entries must be made with waterproof ink in a bound weatherproof field logbook or on field data forms, in accordance with Bluestone's SOP No. 06 – *Field Documentation*. The survey location identifier (i.e., sample identification number) and respective coordinates must be recorded digitally, and in the logbook and/or field forms. The method used to determine elevation must be included with the data set.

Information that will be documented in the logbook includes:

- Project name and number
- Date and time (military)
- Datum and UTM coordinates used
- Surveying personnel
- Weather conditions
- Equipment used
- Waypoint number in the GPS
- Daily field verification information (i.e., benchmark identification and coordinates)
- Survey location identification
- Survey location coordinates (northing and easting UTM coordinates)
- Elevation and method to obtain (GPS, Topographic map, etc.)
- Descriptions and coordinates of alternate survey locations (offsets)
- Distance from alternate survey locations to original survey locations (collected manually)
- Description of any conditions that may affect data quality.

7.0 DATA MANAGEMENT

Data collected during the GPS Survey (i.e., location of off-set sampling points, obstructions, etc.) will be recorded in the field logbook and on field data sheets as necessary. Pre-existing conditions and post-work site restoration should be documented through photographs with

approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 05
DECONTAMINATION OF FIELD EQUIPMENT

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology used to perform decontamination activities during an environmental investigation. All sampling and investigation equipment that comes in contact with soil or groundwater must be decontaminated prior to use in the field, between soil borings, between sampling locations, and at the completion of field activities.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated decontamination activities include contact with potentially impacted media, splashing of decontamination fluids, heavy lifting, pinch points and slip/trip/fall hazards, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with decontamination tasks, and includes, safety glasses, safety shoes and nitrile gloves. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 DECONTAMINATION EQUIPMENT

Standard equipment and supplies necessary to perform decontamination:

- De-ionized or distilled water
- Non-chlorinated potable water
- Spray bottle
- 5-gallon pail
- Scrub brush
- Non-phosphate laboratory-grade detergent
- Nitric acid spray (10%) (for inorganics)
- Methanol spray (for organics)
- Plastic sheeting
- Contractor bags
- Nitrile gloves
- Paper towels

5.0 EQUIPMENT-SPECIFIC DECONTAMINATION PROCEDURES

5.1 Decontamination of Non-Dedicated Pumps

Well pumps will be decontaminated using to the following procedure:

1. Disassemble the pump and place on clean plastic sheeting. Note, plastic sheeting must be replaced between each location.

2. Use a distilled water and non-phosphate laboratory-grade detergent mixture to spray down the inner and outer pump and use a scrub brush to wipe away any visual sediment or residual contaminants.

3. Rinse all pieces of the pump with distilled water twice before reassembling. Containerize all decontamination water in a bucket.

4. Spray lightly with appropriate solvent (methanol for organics and nitric acid [10%] for inorganics, followed by a second distilled water rinse.

5. Place the equipment on clean plastic and allow it to air dry.

6. Wrap equipment in plastic or aluminum foil to store until next use.

7. Retain decontamination fluids for disposal as described in the Bluestone SOP No. 10 – *Storage and Sampling of IDW*.

5.2 Decontamination of Sampling Equipment

Non-disposable and other non-dedicated equipment used to collect soil and/or groundwater samples will be decontaminated prior to use. This equipment includes, but is not limited to, stainless-steel knives and spoons, split-spoon barrels, direct-push shoes and rods, and stainless-steel bowls used to homogenize soil samples.

Sampling equipment will be decontaminated using the following procedures:

1. Fill a non-metallic wash tub or bucket with approximately 6 inches of deionized/distilled water. Mix a detergent solution in the tub. The solution shall consist of approximately 1 tablespoon of non-phosphate laboratory-grade detergent (e.g. Liquinox®) per gallon of water.

2. Scrub all sampling equipment with a stiff-bristled brush and detergent solution.

3. Transfer the equipment to a separate tub partially filled with deionized/distilled water and thoroughly rinse each piece of equipment.

4. Spray lightly with appropriate solvent (methanol for organics and nitric acid [10%] for inorganics.

5. Perform a second rinse using fresh deionized/distilled water and collect fluid into the dedicated rinse tub.

6. Place the equipment on clean plastic sheeting and allow it to air dry.

7. Wrap equipment in plastic or aluminum foil to store until next use. Retain decontamination fluids for disposal as described in Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

5.3 Decontamination of Drilling Equipment and Heavy Machinery

Drilling tools including drill string, augers, drill bits, direct-push rods, etc., in addition to heavy equipment (i.e., drill rigs, excavators, etc.) will be decontaminated between each borehole according to the following procedures:

1. Construct a decontamination pad using heavy plastic sheeting other waterproof materials or use a decontamination trailer or tank.

2. Back the drill rig or direct-push rig into the decontamination pad/trailer/tank or place equipment in a rack off the ground inside the unit, as appropriate.

3. Remove all visible soil and contamination from equipment surfaces by steam cleaning. Include the inside of drill string, augers, and direct-push rods. If necessary, use a stiff-bristled brush to physically remove residual soils and contamination.

4. Transport the unit to an area free of IDW and known impacts and allow the equipment to air dry.

8. Retain all decontamination fluids for disposal as described in Bluestone's SOP No. 10 – *Storage and Sampling of IDW*.

6.0 EQUIPMENT BLANK SAMPLING

For quality control purposes, an equipment blank sample must be collected, typically at a frequency of one per every 10 to 20 field samples. Equipment blank samples are collected by pouring a volume of laboratory-prepared de-ionized water over a freshly decontaminated piece of equipment and into the sample bottle to determine the effectiveness of decontamination procedures. The equipment blank sample should be collected after sampling in contaminated areas, rather than after sampling background areas.

7.0 DATA MANAGEMENT

Pertinent information obtained during decontamination activities (i.e., times and methods used) should be recorded in the field logbook. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 6
FIELD DOCUMENTATION

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methods for recording and managing data obtained by performing tasks associated with an environmental investigation. Data management is a critical part of any field investigation, and requires diligence in maintaining accuracy, organization and confidentiality.

Every sample, field measurement and investigative activity conducted during a project must be properly documented using the official field logbook or approved field data sheets (i.e., geologic logs, well construction logs, soil boring logs, calibration forms, field sampling forms, and health and safety tailgate sheets, etc.). The use of electronic field data sheets is permitted at the approval of the Project Manager and Client. Field documentation must be able to support each step of the investigation, from the point of sample collect to final validated data reporting. Records must be available to identify, track and monitor individual samples and provide details for all investigative activities.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 APPROVED FIELD DOCUMENTS

3.1 Field Logbooks

Information pertinent to an investigation must be recorded in a bound logbook with consecutively numbered, water-resistant pages. All site activities must be recorded in the field logbook (i.e., arrival and departure times, a chain of events, progression of work tasks, site

visitors etc.). The field personnel responsible for the entries will sign and date each entry or page. All logbook entries will be made in indelible ink. The time and date of each entry will be noted in the logbook.

Field logbooks must be kept in the field personnel's possession or in a secure location during the investigation. Following the investigation, logbooks will become part of the project file, and will serve as the official site document in the event of legal action. The following list contains typical field logbook entries:

- Date
- Weather conditions
- Client
- Names of all field personnel, including subcontractors
- Site name, municipality, and State
- Location of samples (may include a sketch)
- Type of sample (soil, groundwater, etc.)
- Time (military) of sample collection
- Sample nomenclature
- Interval and depth of sample
- Sample collection methods
- Sample description (color, odor, etc.)
- Field observations
- Quality Control (QC) sample information
- Number assigned to chain-of-custody (COC)

In addition to pertinent sampling information, details with respect to anticipated health and safety hazards, required personal protective equipment (PPE) and a record of daily field safety briefings should be included in the field logbook.

3.2 Field Data Sheets

Sampling data will be recorded on either paper or electronic Field Sample Data Sheets (example data sheet provided below). The following information will be recorded:

- Sample ID
- Sample Location
- Sample Date/Time
- Sample Collection Method
- Sample Description
- Sample Type
- Sampled By

4.0 PHOTOGRAPHIC DOCUMENTATION

Photographs are a preferred method of documenting pre- and post-work conditions; however, the collection of photos must be approved by the Client. Project photos may include the sample, sample collection activities, surrounding areas, disturbances caused by access of heavy equipment or intrusive activities, and final site restoration. Photographs taken to document sampling points should include two or more reference points to facilitate a potential return to the sample point, if necessary.

5.0 SAMPLE DOCUMENTATION

5.1 Sample Nomenclature

In accordance with the Work Plan and/or Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP), each sample is assigned a unique sample identification number that appears on all sample labels, chains of custody (COCs), field logbooks, and all other applicable documentation forms.

The sample nomenclature typically consists of three to four alphanumeric code segments, each separated by hyphens. The sample tracking number identifies the site, sample type or medium, location, depth (for soil samples), and sampling date. QC sample designations will be added, as appropriate, following the nomenclature presented below. However, the Work Plan and/or UFP-QAPP should be consulted to determine the if there any site-specific requirements. Other

pertinent information regarding sample identification will be recorded in the field logbooks or on sample log sheets. The sample numbers will be in the following format: "AA-BBCCC_QEEEE_QC Indicator".

The sample tracking number format is designated as follows:

1) Location ID

AA: Area of Concern (AOC) identifier (AOC 1 = 01; AOC 3 = 03, etc.)
BB: Sample Media, including:
SS: Surface Soil
BS/BD: Subsurface Soil [S=shallow (1-5 ft bgs); D=deep (5-10 ft bgs)]
GW: Groundwater
PW: Pore water

2) Unique timeframe indicator

Q: Quarter identified (First Quarter = 1Q; Second Quarter = 2Q)
EEEE: Year (Example = 2019)

3) QC indicator (if applicable)

DUP: Field duplicate samples will be collected with 'DUP' in the 'AOC identifier'. The actual location of the duplicate sample will be noted in the field logbook.

MS: Matrix spike samples will be labeled with the standard sample convention, followed by 'MS' to clearly indicate to which sample the spike sample correlates.

MSD: Matrix spike duplicate (MSD) samples will be labeled with the standard sample convention, followed by 'MSD' to clearly indicate to which sample the spike sample correlates.

5.2 CCC Sequential Sample Location Number (001, 002, Etc.) Sample Labels and Tags

Each sample collected at a site and transported to a laboratory for analysis will be identified by a sample label, with specific information regarding the sample. Completed sticker labels must be securely attached to the sample container and include the following information:

- Date
- Time (military) of sample collection
- Type of analyses requested
- Sample number
- Sample collection depth

- Location of sample collection
- Type of preservative
- Initials of sampler

5.3 Chain-of-Custody Records

The Chain-of-Custody (COC) serves as physical evidence of sample custody over the life of the sample batch. Field personnel will initiate a COC at the time of sample collection. All custody transfers of the sample batch will be recorded on the COC by the individual relinquishing and the receiver of the samples, and signed, dated, and time stamped at the time of transfer. Each cooler is assigned a separate COC, on which only the samples packed in that cooler are listed.

Laboratory-specific COCs will be provided and in general include the following information:

- Sample identification numbers
- Signature(s) of field personnel
- Date of collection
- Time (military) of collection
- Sample type (solid, liquid, etc.)
- Sample ID
- Number of containers per sample
- Preservative(s) used
- Requested analytes
- Signatures of all sample handlers
- Inclusive dates and times of possession
- Description of compromised sample integrity (if applicable)
- Temperature of cooler upon receipt by laboratory

After completing the COC, the original will be enclosed in a plastic bag and taped to the inside lid of the cooler.

5.4 Custody Seals

Custody seals will be placed over the lid of the cooler and remain in place from the time the coolers are packed until they are opened by laboratory personnel in order to preserve the integrity of the cooler during shipment. Custody seals must be attached so that it is necessary to break the seals to open the cooler. The custody seals will be secured with clear tape. As long as

the COCs are sealed inside the sample cooler and custody seals remain intact, laboratory couriers are not required to sign the COC.

6.0 DAILY REPORT

A daily report will be generated at the end of each sampling day, and will include the following information:

- Date
- Prepared by
- Project and Site name
- Weather conditions
- Level of PPE
- Description of project activities
- Employees and sub-contractors present on site
- Documentation of tailgate safety meetings
- Daily equipment and material deliveries
- Material(s) shipped off site
- List of samples collected
- Submittals
- Planned activities for the next day

7.0 CORRECTIONS TO DOCUMENTATION

Data entries will be recorded with indelible ink. Accountable serialized documents will not be destroyed or discarded, even if they are illegible or contain inaccuracies that require a replacement document. Errors will be corrected by marking a single line through the error, entering the correct information, and initialing and dating the correction. The erroneous information will not be whited out. Any subsequent errors discovered later will be corrected, initialed, and dated by the individual who made the original entry.

8.0 COMMUNICATION AND TECHNICAL DIRECTION

Field personnel should support one another and maintain open communication during all aspects of the field activities. When a technical point is in question, field personnel should communicate with the Project Manager (PM) or technical lead for clarification and/or additional direction.

Deviations, exceptions, and/or omissions from the Work Plan or best management practices must be communicated to the Project Manager in a timely manner. Maintain records of all such communications in a field logbook, describing the issue, the outcome, and individuals involved in the decision.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 07
FIELD SCREENING SOILS USING A PHOTO-IONIZATION DETECTOR (PID)

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for screening soil cores and collecting headspace measurements using a photo-ionization detector (PID). The purpose of screening soils is to determine the presence (or absence) of organic vapors, and subsequently identify zones of subsurface contamination.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with field screening include inhalation of vapors, potentially explosive conditions, pinch points, and biological hazards (i.e., poison ivy, bees/wasps, spiders, ticks) associated with accessing a monitoring well. In addition, drilling hazards may apply (i.e., working around heavy machinery, overhead rotating parts, excessive noise, etc.) since PID screening is often performed in conjunction with drilling tasks. Modified Level D Personal Protective Equipment (PPE) is generally appropriate, and includes a hard hat, safety glasses, steel-toed boots, leather and/or nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 FIELD SCREENING METHODOLOGY

Soil cores may be screened for the presence of organic vapors using a photo-ionization detector (PID). The procedures for collecting measurements with a PID are provided below:

1. Calibrate the PID instrument (MiniRAE® 3000, or similar) as per the manufacturer's instructions.

2. Cut the acetate sleeve or open the split-spoon sampler (method specific) to expose the soil core and use a clean decontaminated soil knife or equivalent to split sections of the core. Position the PID approximately 1 to 2 inches away from soil surface and record measurements every 6 inches to 1 foot, and at any intervals with visible contamination.

3. This method is repeated for every soil core and may be used in the determination of sample collection intervals. An alternative method (headspace) may be used if readings or observations suggest high concentrations of volatile organic constituents (VOCs) and is described in the following section.

5.0 SOIL HEADSPACE FIELD SCREENING

Soil headspace measurements may be collected and analyzed using the following procedures:

1. Calibrate the PID unit as per the manufacturer's instructions.

2. Add a small volume of soil to a quart size Ziploc® bag (approximately ¼ to ½-full) and seal the bag. The amount of soil may be estimated but should be generally consistent for all headspace samples.

3. Allow the sample to sit for approximately five to ten minutes to reach the ambient temperature. Samples should be analyzed before significant condensation forms inside the bag.

4. After the volatilization period, open a 1 to 2-inch section of the bag to insert the sample probe of the PID unit. The highest PID measurement (recorded in parts per million [ppm])observed within the first twenty seconds will be recorded in the field logbook and/or on the drilling log form. It should be noted that headspace readings may be affected by low ambient temperatures. If low temperatures occur during headspace sampling, an alternative method to warm the samples may be required. Generally, if the ambient temperature is below 32 degrees Fahrenheit, the sample should be placed in a heated vehicle or building during the volatilization period and subsequent data recording.

6.0 DATA MANAGEMENT

Data collected during field screening activities (i.e., organic vapor concentrations and corresponding depths/locations) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 08
ELECTROMAGNETIC (EM) 31 GEOPHYSICAL SURVEY

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the technical guidance and procedures that will be employed to conduct oversight of an Electromagnetic Geophysical Survey (EM31) by a qualified subcontractor. The subcontractor will use an EM31-MK2 (electromagnetic terrain conductivity meter) or similar meter to perform the geophysical survey. Since Bluestone does not perform EM31 surveys, this SOP applies to field personnel providing oversight, documentation, and quality control (QC) during a GPR survey conducted by subcontractor personnel.

The requirements of this procedure apply to all project tasks that require the use of the EM31-MK2 device. The instrument is designed to collect transient electromagnetic signals from the subsurface to a maximum depth of approximately 16 feet. The data generated are collected concurrently with a navigational system (Differential Global Positioning System [DGPS], Real Time Kinematics [RTK], or total station) and stored on an Allegro field computer.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with conducting electromagnetic surveys include slip/trip/fall hazards, uneven ground, heavy lifting, electrical hazards, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work, and includes a safety glasses, safety boots with ankle

support, leather gloves, and high-visibility vests. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 INSTRUMENT COMPONENTS

The M31-MK2 Operating Manual, from Geonics Limited will be referenced as needed during the EM31 survey.

EM31-MK2 components include:

- Front Transmitter Tube
- Back Receiver Tube
- Console box with strap and batteries
- Allegro to Console Cable
- Allegro Data Logger (battery charger, data link, download cables, CF Card, PCMCIA CF Card Adaptor)

5.0 SET-UP AND OPERATION

This section outlines the steps for setting up the EM31-MK2 system in the field for the collection of geophysical data using a Global Positioning System (GPS). Be sure to follow all operating manuals for set-up and operation. Refer to **Photo 5-1** in the Operating Manual for the basic setup of the EM31-MK2.

The procedures for assembling the EM31-MK2, according to Operating Manuals include the following:

1. Interface the EM31-MK2 with a navigational system for precise location data. Connect the serial cable from the navigational system to Port 2 on the Allegro computer.

2. Set up the specific software (Dat31MK2 or NAV31MK2) on the Allegro computer.

3. Set the desired data collection rate or sampling rate to 10 hertz (this records a data point approximately every 1.5 to 2.5 feet and is ideal for activities such as buried drum mapping).

4. Set the EM31-MK2 to interface with COM1, and the navigation device to interface with COM2.

5. Adjust line increments and station start times as needed.

6. For most applications, set the mode to 'Vertical' and enable the Conductivity and In-phase features.

7. Perform a 10 to 15-minute warm-up of the EM31-MK2 device, based on weather conditions and ambient temperature.

8. Remove all metal objects from your person. Avoid the use of cell phones and radios within approximately 50 feet of the EM31 while in use, as these objects could potentially interfere with equipment readings.

9. Perform spot check readings to locate an area free of noise/interference to represent background conditions for calibrating the instrument and to perform function tests. Place a non-metallic pin flag in the ground at this location so that the same location be used to check readings throughout the day or over multiple days.

10. Perform pre-survey calibration checks (refer to the EM31 manual). These calibration checks typically consists of adjusting the following elements: In-phase meter readings (set the coarse and fine compensation controls to zero); Phase Check (remove the change in meter readings when course is turned 1/4); and Sensitivity Check (the meter should read between 22 to 26 millimhos/meter). Adjust range settings for areas with background conductivity higher than 30 millimhos/meter.

11. Once calibration checks have been performed, collect data according to operating manual instructions.

12. For QC purposes, at least two files should be created. One QC file must contain static and cable shake tests. For these field tests, the instrument should be placed on a stand or directly on the ground surface. The second file must contain the dynamic spike test, which incorporates a surveyed line with a large metal object to verify the instrument's response to an anomaly source.

13. For production surveys, data collection will be performed following the guidance established in the Site-Specific Geophysical Investigation Plan (GIP).

14. For all surveys, synchronize the clocks between the navigational system and the Allegro computer. Set the clock on the navigational system to 24-hr time. Prior to data collection data, create a file in the navigational system to record all positions during the survey.

6.0 DATA DOWNLOAD AND PROCESSING

This section outlines the steps for downloading raw EM-31 data (*.R31 or *.H31 files) and converting the data into 'xyz' files suitable for import into Geosoft Oasis Montaj software.

1. Remove CF memory card and connect with PC using the CF memory card adaptor. If the

computer is not equipped with a port for the CF memory card adapter, a USB card reader hardware will be required.

2. Copy files from CF memory card onto PC.
3. Convert .R31 files to .G31 files using DAT31W.
4. Open the .G31 files in DAT31W to view the profiles.
5. Convert .G31 files into .xyz. Convert .H31 files in Trackmaker31 to .xyz.
6. If GPS data was streamed to Allegro, simply use the GPS Positioning tool to convert.
7. If GPS information was collected separately and not downloaded onto the Allegro computer, use the Combine EM-31 and GPS Files tool to merge the data.

7.0 DATA MANAGEMENT

Pertinent information gathered during the geophysical surveys (i.e., approximate locations of subsurface features, anomalies and corresponding depths, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

9.0 REFERENCES

Geonics, Ltd., 1996, www.geonics.com/em31.html.

Geonics, Ltd. 1999. *Operating Manual for EM31-MK2*.

McNeill, J.D. 1980. *Electromagnetic Terrain Conductivity Measurements at Low Induction Number*. Technical Note TN-6, Geonics, Ltd., Mississauga, Ontario.

STANDARD OPERATING PROCEDURE NO. 09
GROUND-PENETRATING RADAR (GPR) GEOPHYSICAL SURVEY

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the procedures and technical guidance for performing a ground-penetrating radar (GPR) geophysical survey, and the operational use of the GSSI SIR 3000 unit. The theory and procedures are similar for other GPR vendor units with some minor modifications. Since Bluestone does not perform GPR surveys, this SOP applies to field personnel providing oversight, documentation and quality control (QC) during a GPR survey conducted by subcontractor personnel.

These procedures apply to all project tasks that require the use of a GSSI SIR 3000, GPR unit. GPR operates by transmitting an electromagnetic pulse or wave into the subsurface and receiving portions of the reflected wave, that produces a digital image of underground objects (i.e., buried drums, subsurface utilities, underground storage tanks [UST], etc.), and changes any in the subsurface media (i.e., lithologic contacts, former UST 'graves', etc.). The accuracy and range of GPR depends on the conductivity of subsurface media, with the greatest results achieved from low conductivity materials (i.e., dry sand or granite), where features up to 100 feet below ground surface (ft bgs) can be detected. High conductivity subsurface media (i.e., clay, shale or other saturated media) limit the accuracy and range of GPR to a depth of 3 ft bgs or less.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate

Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated GPR surveys include working around heavy machinery, excessive noise, slip/trip/fall hazards, uneven ground, heavy lifting, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work includes safety glasses, safety boots with ankle support, leather nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 PERFORMING A GPR SURVEY

4.1 Pre-Survey Site Reconnaissance

Prior to the start of the survey, the GPR technician should review information available for the site (i.e., driller's logs, soils surveys, utility maps, GPR Soils Suitability Map). The GPR survey should be designed to accommodate the site setting and project objectives, surface materials and subsurface soils, target depth, antennae frequency, and noise/background interferences.

4.2 Survey Design

Select the appropriate GPR instrumentation, antennae frequency (or multi-frequencies), and navigation/geo-reference points, based on project objectives and known subsurface conditions. Layout the survey paths in advance and clear any obstructions or vegetation.

4.3 GPR components

- GPR cart
- Mainframe – Field computer and data storage
- Radar antennae
- Acumen Data Bridge – Interface for Global Positioning System (GPS) (optional)
- GPS unite (i.e. Trimble GeoXH6000, R10, Robotic total system) (optional)
- Battery charger, data link, download cables, CF Card, PCMCIA CF Card Adaptor.

4.4 Set-Up and Operation

This section outlines the steps for setting up the GPR system in the field in preparation of geophysical data collection using a Global Positioning System (GPS). Be sure to follow all operating manuals for set-up and operation. The depth of penetration is dependent on the frequency of the transmitted signal and soil properties. Signal frequency specific to the antennae should be

employed. Lower frequencies penetrate deeper into the subsurface. The data generated are collected concurrently with a navigational system (Differential GPS [DGPS] or total station) and stored on a field computer.

Procedures for setting up a GPR System:

1. Assemble GPR cart according to manufacturer's operating manuals.
2. Affix Mainframe, Sunshield, and selected antennae.
3. Typical antennae frequencies – 270 and 400mhz moderate/shallow depths, 900mhz and 1.5ghz for concrete scanning.
4. Interface the GPR with a navigational system for precise location data. Connect serial cabling for Acumen Data Bridge and navigational system according to the schematic provided in the operations manual. (optional)
5. Acumen should be configured in the office prior to mobilizing.
6. Set-up the specific data collection parameters in the Mainframe field computer.
7. Set the appropriate profile window, data collection rate or scan rate.
8. Window setting – soils velocity, understanding dielectrics for site soil and calculation two-way travel time for initial window setting (i.e., T=2d* er [sq rt]).
9. Parameters: verify that the number of scans is sufficient for the size of the object.
10. Scan rate: the smaller rate, the more scans per foot of depth (i.e., 2-3 scans for small target, 10 to 20 scans for larger targets).
11. Set the GPR to interface with software-defined radio (SDR) and the navigation device to interface with COM2.
12. Adjust line increments and stations start as needed.
13. Perform a 5 to 10-minute warm-up of the GPR unit according to current weather conditions and ambient temperature.
14. When using the survey wheel perform wheel calibration following instructions in the operations manual. (A calibration constant for the survey wheel is typically determined by performing multiple passes along a 10-ft section of tape and entering the averaged value).
15. Soil properties tend to be highly variable and may impact the performance of GPR. The operator should perform pre-survey calibration scans and spot check readings to locate an area free of noise to represent background conditions. This area can be used for calibration and to perform function tests without the risk of signal interference.
16. Observe where noise is entering the profile and re-adjust the window as needed. (Note: highly attenuated signals may require switching to a lower frequency-antennae to

enhance depth penetration; however, the target resolution may be diminished).

17. Once calibration checks have been performed, collect data according to operating manual instructions.
18. For production surveys, data collection will be performed following the guidance established in the Site-Specific Geophysical Investigation Plan (GIP).

5.0 DATA MANAGEMENT

Pertinent information obtained during the survey (i.e., subsurface features, anomalies, and corresponding depths, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

7.0 REFERENCES

GPR Operating Manual, Geophysical Survey Systems, Inc.

GPR Soils Suitability Map - viewed on the National Soil Survey Center website at http://soils.usda.gov/gallery/main.htm; select Soil Geophysics, or the 2002 ESRI Map Library website at http://gallery.dcse.com/map_library/, use "search" for Map ID 20075. Contact Sharon W. Waltman, NRCS Soil Scientist, at 402-437-4007, or sharon.waltman@nssc.nrcs.usda.gov

Daniels, D.J. 1996. *Surface Penetrating Radar*. London, U.K.: The Institution of Electrical Engineers.

Bigman D. 2017 - Multiple Methods and Best Practices in GPR- An Interview with Brian Jones from GSSI.

Ground-Penetrating Radar Field Notes				
Date:			Project:	
Survey Inst.: GSSI, SIR 3000			Site Name:	
Weather:			Surface Conditions: Flat, scrub vegetation	
DATA COLLECTION PARAMETERS				
Antenna Frequency: 400 mhz			Transmission Rate:	
Mode of Survey (wheel, time):				
GPS Data Collected (filename):				
Number of Samples/Scan:			Format (bits):	Rate:
Range (nS):		Dielectric:	SCN/Unit:	
Filename	Start	Stop	Fiducial Increment	Comments
*****.dzt				

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 10
STORAGE AND SAMPLING OF INVESTIGATION-DERIVED WASTES (IDW)

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for characterization and handling of investigation-derived wastes (IDW). A field investigation typically generates solid and/or liquid wastes to be stored and characterized for disposal purposes. This material is classified as IDW, and typically includes soil cuttings generated through soil borings and monitoring well installations, groundwater sampling purge water, and associated decontamination fluids generated during the investigation or long-term monitoring activities. Waste management procedures for IDW are based on the requirements specified in Title 40 of the CFR, Part 262 (40 CFR 262) *Standards Applicable to Generators of Hazardous Waste* and industry best management practices. All sampling of IDW will be done in accordance with applicable state regulations. This SOP describes the proper on-site disposal, containerization, labeling, and storage of solid and liquid IDWs. Site-specific IDW disposal requirements will be detailed in the Work Plan, Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) and/or Accident Prevention Plan (APP).

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, at least one person on site must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Site Safety and Health Plan (SSHP), the Bluestone Corporate Safety and Health Program, and site and/or client-specific requirements. Potential health and safety

hazards associated with waste characterization and disposal include exposure to contaminants via direct contact, inhalation or splashing, heavy lifting, pinch points, and slip/trip/fall hazards. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with IDW sampling tasks, and includes a safety glasses, safety shoes, leather and/or nitrile gloves, and high-visibility vests. Additional details regarding site safety and health will be provided in the APP and/or SSHP.

4.0 IDW CHARACTERIZATION

4.1 Liquid IDW

Liquid IDW includes well development water, decontamination fluids, and groundwater monitoring purge waters. Liquid IDW may be characterized using associated groundwater quality data generated from the respective samples or direct sampling, and typically qualifies as non-hazardous waste. Liquid IDW may be stored in a bulk frac tank or containerized separately in drums.

On-site disposition of liquid IDW is acceptable in some states, provided the wastes qualify as non-hazardous and do not pose a threat to surrounding properties and authorization is received by the appropriate state agency. All liquid waste known or suspected to be hazardous waste must be contained. Drums and polyethylene totes may be used for interim storage and transport of liquid IDW. Labels will be prepared in accordance with applicable regulations and the guidelines outlined below. Containers must be kept closed and secured except when filling or disposing of the contents. Manufacturer Department of Transportation (DOT) specifications will be followed when sealing containers in preparation for transport.

United Nations (UN)-approved drums (49 CFR 173.3), polyethylene tanks, and 5-gallon pails may be used to collect liquid IDW, as task appropriate. Liquid IDW collected in 5-gallon pails will be transferred to drums or totes as soon as possible after collection. Hazardous (or suspected) liquid IDW must be containerized in UN-approved drums.

Containers of liquid IDW will be labeled to indicate the source and nature of the waste material.

The following information must be visible on the top or sides of each container: container number(s), facility name, associated monitoring well or borehole ID, date of generation, container contents, estimated quantity, and the client point of contact (POC).

Containers will be marked with 2-inch letters and numbers using a waterproof paint pen. A complete inventory of the IDW will be maintained by the Field Team Leader to facilitate identification and tracking of liquid IDW for appropriate disposal. This inventory will document the information listed above, in addition to the location of the container, and initials of the responsible POC. The total number of containers of liquid IDW generated will be recorded in the field logbook at the end of each workday. Containers of liquid IDW characterized as hazardous or non-hazardous transported off-site for disposal will be labeled in accordance with applicable State and Federal requirements including, but not limited to, Resource Conservation and Recovery Act (RCRA) guidelines, the Toxic Substances Control Act (TSCA), and DOT (40 CFR 171-179).

Containers of liquid IDW will be staged temporarily on site until characterization is complete. Containers of liquid IDW stored during winter months should be under-filled to allow for expansion during freezing. IDW should be stored in secure areas of the site, where containers are protected from flooding, traffic, and unauthorized access or tampering.

For IDW pending characterization, all containers must be properly sealed and labeled, and may be staged on pallets until characterization is complete. If possible, IDW containers should be secured with temporary chain-link fencing; however, caution tape and/or temporary orange construction fencing may also be used as need to protect the IDW containers.

Containers of liquid IDW should be characterized and disposed of accordingly in a timely manner. If liquid-filled containers remain in storage for 30 days or more, the containers must be staged on polyethylene sheeting that is surrounded by a retention berm (i.e. 2x4 lumber), and positioned with enough separation so all sides of the containers can be monitored for leaks.

4.2 Solid IDW

Solid IDW includes soil cuttings from sources such as, but not limited to, direct-push sampling, soil borings, and installation of monitoring wells. Solid IDW must be containerized and staged pending proper characterization. To characterize solid IDW, a composite sample consisting of aliquots from each container will be submitted for analysis. In addition, the quality of solid IDW will be evaluated based on data generated from primary sample and historical site data.

Expended personal protective equipment (PPE) may also be considered IDW, depending on site conditions and/or state regulations, in addition to disposable sampling equipment (i.e., bailers, string, acetate liners, etc.), depending on the nature of site contaminants. In most cases, expended PPE and sampling equipment may be double bagged and disposed with other municipal waste at a local sanitary landfill. All gross contamination removed from the PPE and disposable equipment will be added to the appropriate IDW drums/totes.

Solid IDW consisting of soil cuttings and excess soil volume, will be placed in a lined roll-off dumpster or in UN-approved drums. Five-gallon pails may be used for interim handling and transport of solid IDW. On-site disposition of solid IDW (soil cuttings and excess soil volume) may be appropriate, provided the material is characterized as non-hazardous and such disposition is acceptable under State and local regulations. Any soils suspected of being hazardous must be drummed rather than placed into a roll-off dumpster. Containers must be kept closed and sealed except when adding to or disposing of the contents. Manufacturer DOT specifications must be followed when sealing containers in preparation for transport.

Based on the size of the site, each area of concern (AOC) may require a staging area for solid IDW. Each staging area will be characterized separately, in accordance with the *Management of Investigation Derived Waste During Site Inspections*, EPA/540/G-91/009 (USEPA, 1991). However, solid IDW generated from multiple soil borings within a single AOC may be characterized as a single waste stream.

Containers of solid IDW must be labeled to indicate the source and nature of the waste media.

The following information will be marked on the top or sides of each container: container number(s), site name, associated monitoring well or soil boring ID, borehole number, date of generation, container contents, estimated quantity, and the client POC.

Containers will be marked with 2-inch letters and numbers using a waterproof paint pen. A complete inventory of IDW will be maintained by the Field Team Leader to facilitate identification and tracking of solid IDW for characterization and disposal. This inventory will document the details list above, in addition to the location of the container, and initials of the responsible POC. The IDW inventory and the total number of containers of solid IDW generated will be recorded in the field logbook at the end of each workday.

Solid IDW characterized as hazardous (based on laboratory analytical results) will be re-labeled in accordance with applicable State and Federal requirements including, but not limited to, RCRA, TSCA, and DOT. Containers of solid IDW will be staged temporarily at the site until characterization is complete. Solid IDW should be stored in secure areas of the site, where containers are protected from flooding, traffic, and unauthorized access or tampering. Secondary containment structures will be implemented as required for the storage of solid IDW.

For solid IDW pending characterization, all containers must be properly sealed and labeled, and may be staged on pallets until characterization is complete. If possible, IDW containers should be secured within temporary chain-link fencing; however, caution tape and/or temporary orange construction fencing may also be used as need to protect the IDW containers. Containers of solid IDW should be characterized and disposed of accordingly in a timely manner.

5.0 IDW CHARACTERIZATION SAMPLING

The sampling procedures for liquid and solid IDW contained in drums are described in the following sections. Within two weeks of the completion of field activities, an aliquot from each waste stream will be collected and composited except for samples to be analyzed for volatile organics constituents (VOCs). VOC analysis requires the collection of one representative grab sample from each source. For new monitoring well installations, the composite IDW sample will include aliquots only from drums associated with that well; however, aliquots from other composite samples may be grouped by AOC or field task. The list of analytes for the composite sample will match that of the primary investigation samples. If significant impacts are encountered during the field investigation, the IDW generated from these sampling locations will be stored in separate containers.

5.1 Liquid IDW Drum Composite Sampling Procedures

The procedures for collecting a sample from liquid IDW drums, with a known source are listed below:

1. Conduct field screening near drum storage area. If elevated concentrations are detected, then increase PPE level to C or B based on the APP.
2. Wearing clean nitrile gloves, remove bung or drum lid and store on plastic sheeting.
3. Dip sample collector/bailer into center of drum and lower the device into the middle section of the drum.
4. Slowly raise the sampling device and decant the appropriate volume into the bottleware.
5. Repeat Steps 3 and 4 until the correct sample volume has been collected. Cap the bottleware between sampling containers.
6. Replace bung or drum lid.
7. Dispose of or decontaminate the sampling device.

5.2 Solid IDW Drum Composite Sampling Procedures

The procedures for collecting a sample from solid IDW drums, with a known source are listed below: Conduct field screening near drum storage area. If elevated concentrations are detected, then increase PPE level to C or B based on the APP.

1. Wearing clean nitrile gloves, remove bung or drum lid and store on plastic sheeting.
2. Using a decontaminated trowel, gently scrape the top portion of the drum contents to one side.
3. Place sample collector into center of drum contents and slowly advance the device into the middle section of the drum to a depth of approximately four inches below the surface.
4. Extract the sampling device and transfer the soil to the sample jar.
5. Repeat Steps 4 and 5 until the correct sample volume has been collected. Cap the bottleware between sampling containers.
6. Replace bung or drum lid.
7. Decontaminate sampling device and dispose of plastic as solid PPE, as needed.

Procedures for appropriate sampling nomenclature are presented in Bluestone's SOP No. 06 – Field Documentation.

6.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

7.0 DATA MANAGEMENT

Pertinent information obtained during IDW characterization sampling and handling included, but not limited to, sample IDs, sample collection times, methods of collection, and PID screening results will be record in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – Field Documentation details the methods for data collection and management, and additional information should be specified in the Work Plan. Photographs are a preferred method of documenting pre- and post-work conditions; however, the collection of photographs must be in accordance with contract requirements and site-specific security requirements.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 11
GEOLOGIC LOGGING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for logging subsurface materials during drilling activities, including soil borings, groundwater monitoring well installations, and geotechnical borings, etc. All boreholes advanced during an environmental investigation must be logged by an experienced geologist in accordance with in the U.S. Army Corps of Engineers (USACE, 1994) Engineering Manual (EM) 1110 1 4000 Engineering and Design: *Monitoring Well Design, Installation, and Documentation at Hazardous, Toxic, and Radioactive Waste* [HTRW] *Sites*; the American Society for Testing and Materials (ASTM) Method D2488-17E1 (2017a): *Standard Practice for the Description and Identification of Soils (Visual-Manual Procedures)*; the ASTM Method 2487-17 (2017b): *Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)*; and ASTM Method D6286-19 (2019): *Standard Guide for Selection of Drilling and Direct Push Methods for Geotechnical and Environmental Subsurface Site Characterization*.

[NOTE: Soil Boring Log formats will be modified to accommodate specific project objectives and selected drilling methods. As such, the log format may deviate from EM 1110-1-4000; however, all pertinent or required information will be documented.]

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate

Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards created by drill rigs and associated drilling tasks include working around heavy machinery, excessive noise, overhead rotating parts, pinch points, heavy lifting, projectiles, biological hazards (i.e., poison ivy, bees/wasps, spiders, ticks), etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with drilling tasks, and includes a hard hat, safety glasses, steel-toed boots, leather and/or nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 GEOLOGIC LOGGING

The procedures and requirements listed below will be implemented (and modified as necessary) for drilling activities:

1. Geologic logging for soil borings and bedrock boreholes must be completed simultaneously with the progression of drilling. Geologic logs will be prepared using USACE HTRW Drilling logs known as MRK Forms 55 and 55-2.

2. The MRK forms must include all relevant and available information. 'NA' may be entered on the forms for data that are not applicable (e.g., 'NA' is recorded in the Depth to Bedrock Section if bedrock is not encountered in the borehole).

3. Stratigraphic or lithologic changes encountered in the borehole must be shown in Column C as a solid line. Gradational changes in stratigraphy and lithology will be shown as a dashed line in Column C. The scale of the log shall be one-inch equals one foot.

4. The bottom of the borehole will be represented on the form as a solid double line with the notation 'Bottom of Borehole.' Note in column H if the bottom of the borehole was at refusal.

5. Results of headspace air monitoring will be reported in Column D. Any evidence of contamination will be noted in the remarks section under Column H, including color, odor, or staining.

6. During the progression of drilling activities, depth-to-water measurements should be collected frequently and recorded in Column H with respective times of measurement. Any dry boreholes will be noted in Column H. The depth to water will be recorded in Column H and Box 15 at the time when the first water-bearing zone is encountered. Depth-to-water measurements will be recorded in Column H and in Boxes 16 and 17

after the completion of drilling and again after additional time has elapsed and groundwater elevation has stabilized.

7. The depth of each run (core or soil sample) will be measured with a weighted tape to the nearest 0.1 ft and recorded in Column H. Borehole depths and times of measurement should recorded frequently on the MRK form. In addition, the start and stop times of drilling should be recorded throughout each workday. The weighted tape must be constructed of materials that will not introduce contaminants into the borehole.

8. Intervals of no recovery (i.e., lost bedrock core or soil) will be measured and recorded in Column E. The length of the core or soil sample (recovery) will be measured to the nearest 0.1 ft and recorded in Column H.

9. The size and type of sampler or coring bit and barrel will be noted and recorded in the remarks section of Column H and Box 7. Geologic logs will indicate borehole and sample diameters in Column H, along with depths at which sampling methods or equipment change.

10. The source of the water used for coring or monitoring well installation will be recorded in Column H.

11. Drilling fluid volumes, loss or gain, brand and product name (as applicable) will be recorded in the remarks section in Column H.

12. If compressed air is used during the drilling process, the type of air filter will be recorded in Column H.

13. The depth and material of any temporary casing used during the well installation procedure will be recorded in Column H.

14. Depth intervals of borehole instability observed during drilling will be recorded in Column H.

15. Difficulties experienced during drilling (e.g., changes in drilling speed, rates, or downhole torque) and any issues in sampling will be noted in Column H, with corresponding descriptions of problem resolutions.

16. The depth interval over which samples are collected for lithologic analysis will be noted and recorded in Column E. The depth interval over which samples are collected for chemical analysis will be noted and recorded in Column F.

17. Each geologic log will be legibly signed by the preparer after proof-reading the log for completeness.

5.0 MEDIA-SPECIFIC LOGGING

5.1 Geologic Logging of Soil (Unconsolidated Material)

Unconsolidated material will be logged using the Unified Soil Classification System (USCS), as referenced above. All items in this section are noted in Column C of the MRK form.

1. The moisture content, in relative terms (i.e., dry, moist, wet/saturated), will be noted. If the sample is saturated (i.e., encountered groundwater), the groundwater level will be recorded to the nearest 0.1 ft as noted above.

2. The standardized color of the unconsolidated material will be logged using the Munsell Soil Color Chart.

3. The angularity, grain size, and grading of soil classified as coarse will be logged. An estimate, by percent of quantities of components (e.g., sand versus silt, silt versus clay, etc.), will be logged.

4. The consistency of materials classified as fine (e.g., ML or CH) and the density of materials classified as coarse (e.g., SW or GM) will be noted.

5. Bedding characteristics, evidence of bioturbation, root holes, and fractures will be noted and logged.

6. When known, the depositional type (i.e., alluvium, till, loess) will be noted.

5.2 Geologic Logging of Competent Bedrock (Consolidated Material)

All items from this section are noted in Column C of the MRK forms.

1. The geologic formation name will be noted.

2. The rock type (i.e., limestone, sandstone, shale) will be noted and logged.

3. The relative hardness of the consolidated material will be measured and logged.

4. The texture and grain angularity of the consolidated material will be examined with a hand lens and noted.

5. The standardized color of the consolidated material will be logged using a Munsell Rock Color Chart.

6. The consolidated material will be inspected for apparent weathering and noted accordingly.

7. The moisture content, in relative terms (i.e., dry, moist, wet/saturated), will be noted. If the sample is saturated (i.e., encountered groundwater), the groundwater level will be recorded to the nearest 0.1 ft as noted above.

8. Evidence of bedding, bedding planes, fractures, and joints will be noted and logged. The approximate angle of the dip of bedding, fracture, and joint planes will be noted.

9. Other significant features (i.e., fossils, crystalline minerals, voids, solution cavities, etc.) will be noted.

10. The reaction of the consolidated material to hydrochloric acid, if any, will be recorded.

11. Bedrock coring information shall be recorded in consecutively numbered runs in Column H and shall include the start and stop time of each run, depth to the top and bottom of each run, length of recovery, and size and type of coring bit and barrel.

6.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

7.0 DATA MANAGEMENT

Data collected during drilling activities (i.e., depth-to-water measurements, borehole depths, PID measurements, refusal depth, bedrock depth, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

An example of a modified HTRW Soil Boring Log template is provided below. It should be noted that the modifications shown in the template are designed for DPT drilling methods.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

Bluestone

HTRW DRILLING LOG

DISTRICT		DATE	BOREHOLE NO.
COMPANY NAME	DRILLING COMPANY		SHEET OF SHEETS
INSPECTOR	DRILLER / FOREMAN		
PROJECT	PROJECT NO.		
PROJECT LOCATION	BOREHOLE LOCATION		SURFACE EL.
DRILL METHOD		RIG TYPE	
START DATE	COMPLETION DATE	NOTES	
DEPTH TO GROUNDWATER	DEPTH TO BEDROCK		
DEPTH DRILLED INTO ROCK	TOTAL DEPTH OF BOREHOLE		
DEPTH TO WATER AND ELAPSE TIME AFTER DRILLING		OTHER WATER LEVEL MEASUREMENTS (SPECIFY)	
GEOTECHNICAL SAMPLES			
DISTURBED	UNDISTURBED		TOTAL NO. OF CORE BOXES
ENVIRONMENTAL SAMPLES			
VOCs	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	TOTAL RECOVERY
DESCRIPTION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	
SIGNATURE OF INSPECTOR			
LOCATION SKETCH / NOTES			SCALE
PROJECT			**BOREHOLE NO.**

Bluestone

HTRW DRILLING LOG

HTRW LOG CONTINUATION SHEET	DATE	BOREHOLE NO.
INSPECTOR	DRILLER / FOREMAN	SHEET OF SHEETS
SIGNATURE OF INSPECTOR		

Surface El.	Depth (ft bgs)	S-# / R-#	Recovery (ft / %)	USCS	Munsell	Moisture	PID (ppm)	Description	Notes / Sample

PROJECT	BOREHOLE NO.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 12
LOW-FLOW GROUNDWATER SAMPLING AND SAMPLING WITH A BAILER

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the procedures for the collection of groundwater samples using low-flow sampling techniques in both permanent and temporary groundwater monitoring wells. This procedure is consistent with the USEPA SOP: *Low Flow (Minimal Drawdown) Groundwater Sampling Procedures* (USEPA, 2017). Groundwater samples will be collected from monitoring wells using stainless-steel or polyvinyl chloride (PVC) bladder pumps equipped with Teflon bladders and either polyethylene or Teflon®-lined polyethylene high density tubing. Bladder pumps allow groundwater samples to be retrieved with little disturbance to the sample matrix and minimal exposure to the atmosphere, while employing low-flow techniques.

Groundwater and quality control (QC) samples will be collected and containerized in the order of the volatilization sensitivity of each constituent. If insufficient volume is available to collect the full analytical suite and designated QC samples, the available volume will be allocated at the discretion of the Project Manager and Project Chemist. This hierarchy of samples necessary for low yield/recharged wells will consider site-specific priorities, sample size, effect of turbidity on analytical results, etc.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with low-flow or bailer groundwater sampling include biological hazards (i.e., poison ivy, bees/wasps, spiders, ticks, etc.) associated with access the well, twisting and repetitive motion (bailing), heaving lifting, nicks and cuts, uneven ground, acid preservatives, contact with contaminated water, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with groundwater sampling, safety glasses, safety boots with ankle support, leather and/or nitrile gloves, and high-visibility vests. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 PRE-SAMPLING ACTIVITIES

4.1 Depth-to-Water and Total Well Depth Measurements

Prior to the start of sampling, a synoptic round of depth-to-water measurements will be collected to evaluate the potentiometric surface of groundwater at the site. An electric water-level indicator with an accuracy of +0.01 feet will be used to collect measurements. In addition, the total depth of each well will be measured, and described as a 'hard' or 'soft' bottom to indicate the presence of sediment that may have accumulated at the bottom of the well.

The procedures for collecting depth-to-water measurements include the following:

1. Measurements will be recorded in field logbooks. Depth-to-water and total depth measurements will be collected using the following procedures: Decontaminate the water level probe.

2. Check for proper instrument response by inserting the probe in cup of water. Replace the batteries, as needed.

3. Document observations with respect to the condition of the well pad, surface or protective casing, well locks, obstructions inside the well casing, total well depth, and other well conditions in the field logbook, or on a well assessment field form. Recommendations for maintenance actions should be reported.

4. Don a pair of clean nitrile gloves.
5. Unlock the well cover and remove the cap.
6. Locate the reference point on the riser pipe (notch, mark, or highest point on the riser pipe).
7. Slowly lower the probe down the well until the signal indicates that the water has been contacted.
8. Record the reading at the reference point as depth-to-water.
9. Withdraw the probe and repeat steps 7 & 8. Duplicate measurements should agree within 0.02 feet. If not, continue with measurements until 0.02 feet precision is achieved.
10. Turn the meter to the off position and lower the probe gently until the bottom of the well is encountered. Record the reading at the reference point as the total well depth and observe and record the condition of the bottom of the well (i.e., hard or soft).
11. Remove the probe from the well and decontaminate the unit.

4.2 Field Measurements for Groundwater Sampling

Immediately after removing the well cap, collect a headspace reading using a PID from the top 6 inches inside the well casing. This information will be utilized in conjunction with the site health and safety plan to determine the personal safety level required during sampling. The headspace readings must be recorded in the field logbook. If readings exceed background (0.0 parts per million [ppm]), air monitoring must continue for the duration of the sampling event. Once the headspace reading is recorded, the pump array may be set up and the pump lowered into the well at the target depth.

During the well purging process, water will pass through the flow-through cell at a controlled low rate, and water quality parameters (i.e., temperature, pH, specific conductance, dissolved oxygen (DO), oxidation-reduction potential (ORP), and turbidity) must be recorded every five minutes. The flow-through cell chamber and sample tubing should remain free of air bubbles during the purge process. Tighten or replace the sampling elbow if air bubbles are introduced.

5.0 LOW-FLOW SAMPLING EQUIPMENT

A list of equipment needed for the collection field measurements is summarized below:

- PID
- Water quality multi-parameter meter (i.e., YSI Pro Plus, or similar) capable of measuring pH, specific conductance, turbidity, temperature, DO, and ORP
- Flow-through cell
- Graduated cylinder
- High-density polyethylene tubing
- 5-gallon pails
- Nitrile gloves
- Pump control box
- Compressed air source

6.0 CALIBRATION OF MULTI-PARAMETER METER

The YSI Pro Plus (or similar) and PID unit must be calibrated at the start of each workday using the factory calibration standards provided by the manufacturer. In addition, ORP will be checked for accuracy using standard redox solution (200-275 millivolts [mV] at 25 degrees Celsius [°C]). Note: Standard Hydrogen Electrode (SHE) corrections for ORP field measurements will be completed at the end of the sampling event. Calibration checks will be conducted at the end of each workday, and any time the readings appear erroneous.

DO measurements will be verified both before and after each well. An accuracy check will be performed on the ferrous iron colorimeter once per day. The manual will be consulted if the reading is not within ±0.25 milligrams per liter (mg/L) of the standard solution.

All calibrations and calibration checks will be recorded in the field logbook and appropriate field forms. Extreme cold or hot weather may affect the performance of the YSI meter. In these conditions, the meter may require more frequent checks for instrument drift. Equipment manuals will be available for each instrument, and field personnel should consult the manual for

additional technical details. Additional information regarding calibration procedures is available in Bluestone's SOP No. 02 – *Equipment Calibration and Maintenance*.

7.0 LOW-FLOW SAMPLING TECHNIQUES

7.1 Bladder Pump Sampling

A freshly developed monitoring well must be allowed to stabilize for a minimum of 14 days prior to purging and sampling. To the extent possible, monitoring wells will be purged and sampled using bladder pumps. Non-dedicated bladder pumps will be decontaminated prior to and after each use. For bladder pumps used to collect volatile organic constituents (VOCs) or dissolved gas samples, the pump should be set to deliver Begin the task by removing the dedicated pump (if applicable) and collecting the depth-to-water and total well depth measurements, as described above. The pump should not be removed from the well except during the initial round of measurements. Leave the water level indicator in the well and connect the pump and hose assembly to a pump control box. Connect the controller to a compressed gas cylinder containing either nitrogen or carbon dioxide or to a portable air compressor. Connect the flow-through cell to the pump discharge tube so that the sample flows into the bottom of the flow-through cell. Direct the discharge from the flow-through cell into a 5-gallon pail. Groundwater purging will be conducted in accordance with applicable State and/or Federal regulations, as indicated below.

If the project objectives require metal concentrations to be field-filtered, an in-line filter (transparent housing preferred) will be used, in addition to the same low flow procedures. Pre-rinse the filter (0.45 µm) and verify that the filter is free of air bubbles prior to sample collection.

7.2 Procedures for a Sustainable Recovery Well

For the purpose of this SOP, a sustainable recovery well is defined as a well capable of maintaining a stable water level during pumping at a constant flow rate, at an elevation above the pump intake such that there is sufficient volume for all required samples (including any extra volume required for quality assurance/quality control (QA/QC) purposes), plus two additional sampling

system volumes. Excessive drawdown refers to drawdown of the water column at a constant flow rate, such that a stabilized water level cannot be obtained at an elevation above the pump intake.

7.3 Standard Sustainable Recovery Well

For this SOP, a standard sustainable recovery well is defined as a well in which stabilized water levels can be obtained at a pumping rate equal to or greater than 100 milliliters per minute (mL/min). The following procedures are used for low flow sampling:

1. Obtain well casing and borehole diameters, and filter pack percent-porosity from available well construction records (may be needed for calculations if the well is determined to be a low- recovery well).

2. Calibrate any electronic water-quality equipment as per manufacturer's instructions and record calibration data in the field logbook and on the calibration form .

3. Check the function of the electronic water level meter as per the manufacturer's instructions.

4. Assemble equipment at the well and perform field preparatory activities.

5. Measure the total well depth. Using the electronic water level meter, measure and document the depth of the well [to the nearest 0.01 foot (ft)] from the reference mark on the top of the inner well casing.

6. Pump type specific steps.

    a. If using a non-dedicated pump, the following steps are applicable:

        i. Measure water level. Using the electronic water level meter, measure and document the water level to the nearest 0.01 ft. from the reference mark on the top of the inner well casing.

        ii. Assemble the pump and sampling line components, taking care not to contact any of the components with potentially-contaminated media (i.e., ground surface), and ensure that the discharge line is affixed such that initial discharge is captured in either a graduated 5-gallon pail or a purge water collection/disposal drum.

        iii. Determine the depth of the portable pump intake. Measure length of pump from intake to tubing and cable attachment. Measure length of tubing and cable needed to set pump at target depth within the screened interval.

iv. Slowly lower the pump into the well casing to the target depth, taking care not to encounter the bottom of the well and cause unnecessary agitation of sediment. Affix the pump in this position by fastening the supporting cable. Record depth of pump intake from the reference mark on the top of the inner well casing.

b. If using a dedicated pump, the following steps are applicable:

i. Obtain well depth and depth of pump intake from well construction records.

ii. Measure water level with pump in place. Using the electronic water level meter, measure and document the depth to water (to the nearest 0.01 ft) from the reference mark on the top of the inner well casing.

7. Determine the saturated casing volume and saturated borehole volume (saturated casing volume + saturated filter pack volume). This may be needed for calculations if the well is determined to be a low recovery well.

8. Determine the saturated casing volume above the pump intake. This may be needed for calculations if the well is determined to be a low recovery well.

9. Determine sampling system volume (volume capacity of pump, tubing, and flow-through cell). This may be needed for calculations if the well is determined to be a low recovery well.

10. Determine volume necessary to collect all required samples, including QA/QC samples. This may be needed for calculations if the well is determined to be a low recovery well.

11. Connect the flow-through cell and multi-meter to the pump tubing.

12. If the sustainable flow rate is not known for the well, begin purging at 100 mL/min. For wells with historical sustainable flow rate data, use the historical rate.

13. Ensure that no air bubbles are entrained in the pump tubing. Raise the level of the flow-through cell above the well such that water must pump upward through the intake tubing of the cell. This will purge any bubbles through the tubing. After the cell fills with water, it may be lowered.

14. Measure and record the water level and an initial set of water quality parameter measurements.

15. Determine the initial purge flow rate from the well. Using a graduated cylinder, bucket, or other suitable container of known volume and a stopwatch, time the rate of filling.

16. Determine whether the initial purge flow rate causes excessive water level drawdown in the well. Measure and record the water level and water quality parameters at 500 mL or five-

minute intervals . The water level will be considered stable if water level readings do not decrease more than 0.3 ft over three successive measurements (it is acceptable for the water level to remain unchanged or to increase) and if the volume of water in the casing above the pump intake is equal to or greater than the volume needed for all required samples plus two sampling system volumes.

17. If the initial purge rate of 100 mL/min does not cause excessive drawdown and is an appropriate rate for project analytes and purposes, document that sustainable recovery has been achieved at this rate and skip to Number 21 and obtain stabilized water quality parameter readings.

18. If the initial purge rate of 100 mL/min does not cause excessive drawdown and a higher rate is desirable for project-specific reasons, adjust the flow rate and determine whether sustainable recovery can be obtained using the higher flow rate. Record each adjustment made to the pumping rate, the water level, and the multi-meter readings measured immediately after each adjustment. The water level and water quality parameters should be measured and recorded approximately every five minutes. When sustainable recovery has been documented at the higher flow rate, skip to Number 21 and obtain stabilized water quality parameter readings. (Note: Assuming a highly transmissive formation, one liter/minute is the maximum purge rate that will preserve laminar flow in the screened interval).

19. If the initial purge rate of 100 mL/min causes excessive drawdown and the well is less than 30 feet deep, the procedure may be repeated using a peristaltic pump to determine whether sustainable recovery can be obtained at flow rates less than 100 mL/min (See alternative sustainable recovery well section below).

20. If the initial purge rate of 100 mL/min causes excessive drawdown and alternative equipment with flow rates less than 100 mL/min cannot be used, see Low-Recovery Wells Section below.

21. Once a stabilized water level has been obtained, the field parameters will be monitored for stabilization. If the flow rate is equal to or greater than 100 mL/min, measure and record the water quality parameters at five-minute intervals. If the flow rate is less than 100 mL/min, record the water quality parameters at time intervals of 500 mL divided by the purge rate. Field parameters will be considered stable when three consecutive measurements within the following ranges are obtained:

    a. Turbidity: (10% for values greater than 5 Nephelometric turbidity units (NTU), if three turbidity values are less than 5 NTU, consider the values as stabilized),

    b. Dissolved Oxygen: (10% for values greater than 0.5 mg/L, if three DO values are less than 0.5 mg/L, consider the values as stabilized),

c. Oxidation/Reduction Potential: (+/- 10 millivolts),
d. Specific Conductance: (3%),
e. pH: (+/- 0.1 unit), and
f. Temperature: (3%).

22. Once water quality parameters have stabilized, the groundwater sample may be collected.
23. If parameters other than turbidity stabilize, but turbidity stabilization cannot be attained, the Project Manager will be consulted.

7.4 Alternative Sustainable Recovery Well

An alternative sustainable recovery well is defined as a well in which stabilized water levels can be obtained at a pumping rate less than 100 mL/min using alternative equipment capable of lower flow rates (e.g., peristaltic pump, mini bladder pump). The following procedures will be used for alternative sustainable recovery wells:

1. If stabilized water levels can be obtained at a pumping rate less than 100 mL/min using alternative equipment, refer to Number 21 above, and obtain stabilized water quality parameters. Note that for flow rates of less than 100 mL/min, parameter measurement interval is determined by 500 mL divided by the purge rate.

7.5 Procedures for Sampling Low-Recovery Wells

A low recovery well is defined as a well in which stabilized water levels cannot be obtained as described for sustainable wells, regardless of pumping rate or equipment type. The following procedures will be used for low recovery wells:

1. If a purge rate of 100 mL/min causes excessive drawdown and/or alternative equipment with flow rates less than 100 mL/min cannot be used, the following procedures should be used.
2. The following information (see previous steps 1, 7, 8, 9) is needed:

- Obtain well casing and borehole diameters, and filter pack percent-porosity from well construction records.
- Determine saturated casing volume and saturated borehole volume (casing volume + saturated filter pack volume).
- Determine saturated casing volume above the pump intake.

- Determine sampling system volume (volume capacity of pump, tubing, and flow-through cell).
- Determine volume necessary to collect all required samples, including QA/QC samples.
- Determine whether the saturated casing volume above the pump intake is sufficient for at least two sampling system volumes plus required samples.

3. If the casing volume above the pump intake is sufficient for at least two sampling system volumes plus required samples, purge slowly at a constant flow rate. measure and record water levels and field parameters every 500 mL until two (or available) system volumes have been removed; collect samples; document conditions and procedures. (Note: water level will not be stable (i.e., drawdown will occur) and water quality parameters may not be stable).

4. If the casing volume above the pump intake is not sufficient for at least two sampling system volumes plus required samples but is sufficient for at least one sampling system volumes plus required samples, purge slowly at a constant flow rate. measure and record water levels and water quality parameters every 500 mL until one (or available) system volumes have been removed; collect samples; document conditions and procedures. (Note: water level will not be stable (i.e., drawdown will occur) and water quality parameters may not be stable).

5. If the casing volume above the pump intake is sufficient for required samples only, determine whether it is acceptable to collect samples without purging. If this is acceptable for project purposes, collect samples at a constant flow rate without purging, document conditions and procedures.

6. If the casing volume above the pump intake is not sufficient for required samples, determine whether samples can be prioritized and if it is acceptable to collect priority samples without purging. If this is acceptable for project purposes, collect the priority samples at a constant flow rate without purging, document conditions and procedures.

7. If the casing volume above the pump intake is not sufficient for all required samples, samples cannot be prioritized, and/or it is not acceptable for project purposes to collect samples without purging, do not collect a sample, and document conditions.

8. If the well cannot be sampled using the low-recharge procedure:

   - Determine whether diffusion samplers or other passive methods are acceptable for project purposes and can be used.
   - Determine whether the well can be removed from the monitoring network.

8.0 GROUNDWATER SAMPLING DECISION TREES

The following decision tree will be used for purging monitoring wells (see Figures 1 and 2 below):

1. Wells with historical purge rate data:
    - For a consistent, sustainable recovery well (i.e., stabilized water level can always be achieved) using either standard or alternative equipment.
        - Use historical sustainable flow rate and equipment
        - Obtain stabilized water level
        - Obtain stabilized water quality parameters; and
        - Collect samples.
    - For a well with inconsistent recovery across multiple sampling events, determine whether a sustainable flow rate can be achieved during this sampling episode using standard or alternative equipment.
        - If yes, use sustainable rate and appropriate equipment; obtain stabilized water level; obtain stabilized water quality parameters; collect samples.
        - If no, use low recharge procedure.
2. Wells without historical purge rate data but with information from well development or redevelopment:
    - Does well development or redevelopment record indicate a sustainable recovery well using either standard or alternative equipment?
        - If yes, determine a sustainable flow rate and use sustainable rate procedures and appropriate equipment; obtain stabilized water level; obtain stabilized water quality parameters; collect samples.
        - If no, use low recharge procedures.
3. Wells without either historical purge rate or well development or redevelopment data:
    - Determine whether a sustainable flow rate can be achieved during this sampling event using standard or alternative equipment.
        - If yes, use the sustainable rate and appropriate equipment; obtain stabilized water level; obtain stabilized water quality parameters; collect samples.
        - If no, use low recharge procedures.

9.0 EQUIPMENT MALFUNCTION PROCEDURES

Every effort will be made to procure and maintain properly functioning equipment; however, equipment malfunctions may occur. In these instances, the field team leader will be contacted immediately, followed by the Project Manager. To the extent practicable, field crews will be equipped with backup sets of equipment. Any necessary replacement items will be ordered for next-day delivery. Since the measurement of DO is most likely to malfunction, subsequent wells will be purged and sampled at or below historical purge rates until 125 percent of the maximum volume purged during the previous three sampling events is removed from the well. All other stabilization parameters must also meet stabilization criteria prior to sample collection.

Any other equipment malfunctions will be brought to the attention of the Project Manager and a temporary site-specific sampling protocol will be implemented. In addition, any equipment malfunctions and remedies must be noted in the field logbook and on the daily report.

Figure 1 Groundwater Sampling Decision Flowchart - Wells Screened Below Water Table

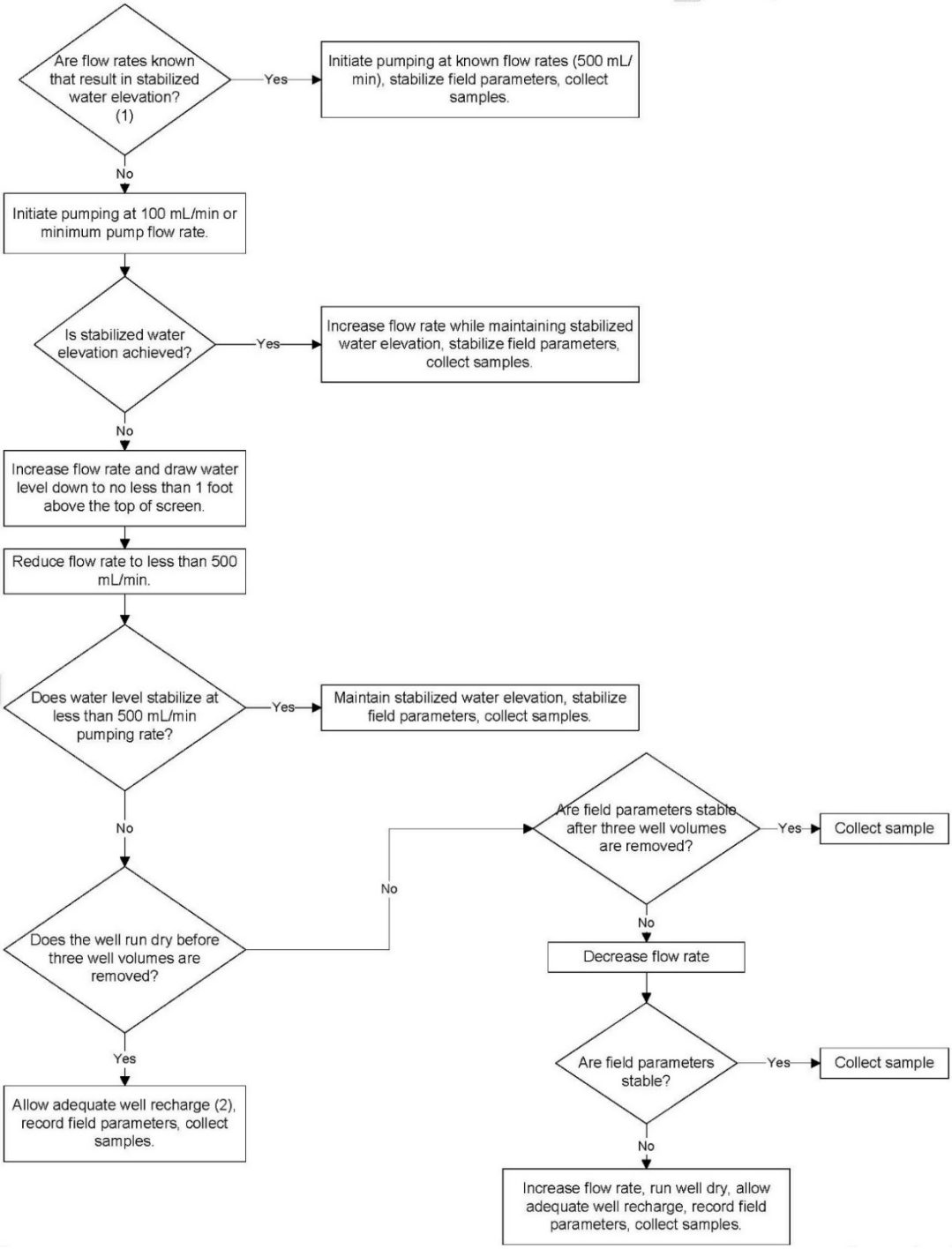
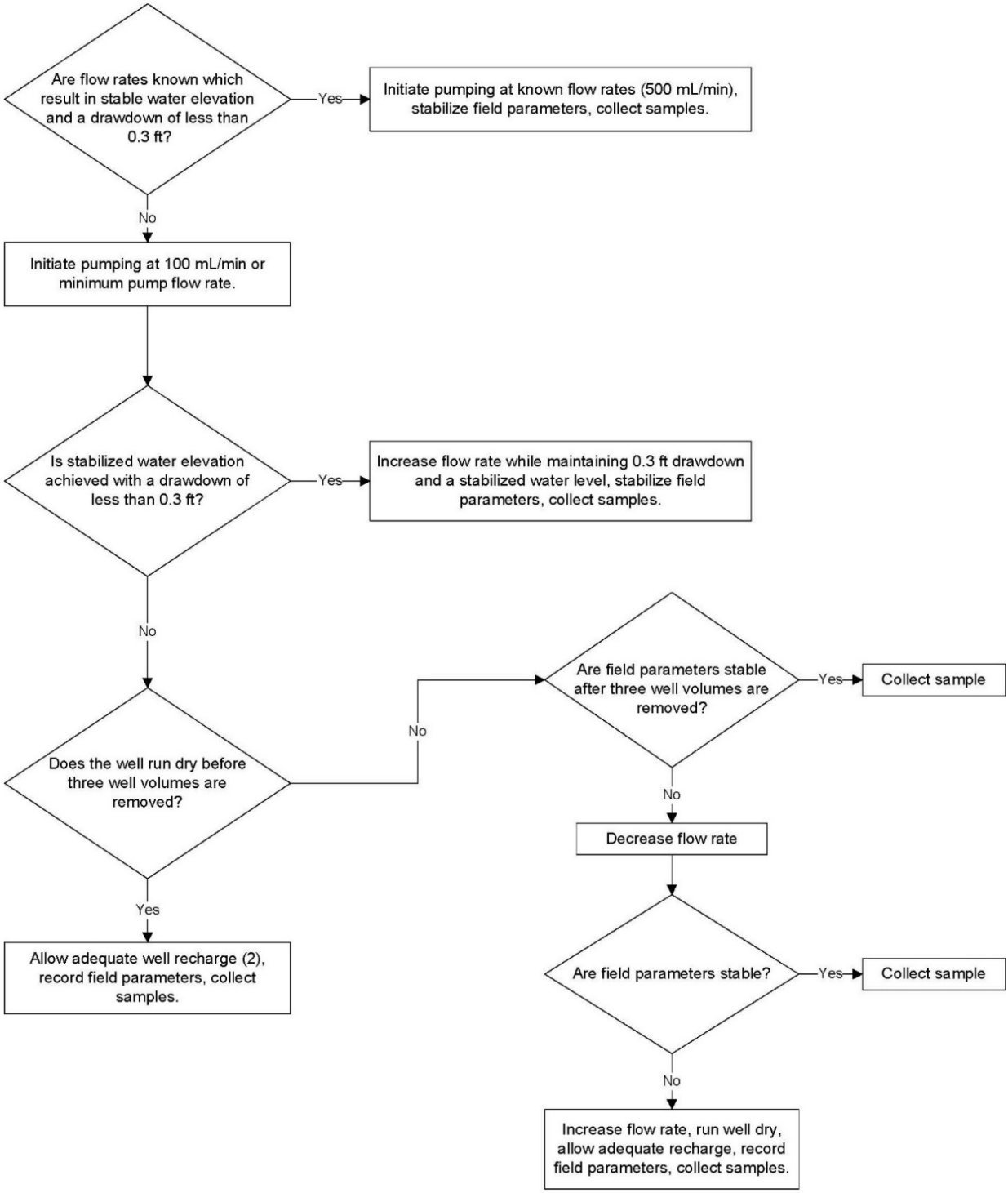


Figure 2 Groundwater Sampling Decision Flowchart - Wells Screened Across Water Table



10.0 GROUNDWATER SAMPLE COLLECTION USING A BAILER

For groundwater samples collected with a bailer, a standard cleaned closed-top polyethylene or Teflon® bailer with Teflon® coated stainless-steel leaders must be used. A new piece of nylon rope must be used for each bailer. The bailer and rope are lowered slowly into the well to the top of the water column, allowed to fill, and removed. It is critical that the bailer be slowly and gently lowered into the water column, particularly during the final stages of purging, to minimize turbidity and disturbance of any VOCs. A straight tube should be used to displace the check valve at the bottom of the bailer to decant the water into sample the appropriate sample containers.

11.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

12.0 DATA MANAGEMENT

Data collected during well sampling activities (i.e., depth-to-groundwater measurements, total well depth, sampling IDs, sample collection times, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

13.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 13
SAMPLE PACKAGING AND SHIPPING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the procedures for the proper packaging and shipping of environmental samples. All samples must be shipped priority overnight in accordance with the U.S. Environmental Protection Agency (USEPA) specifications and U.S. Department of Transportation (DOT) regulations (49 Code of Federal Regulations [CFR] Parts 172 and 173). Samples will be handled as a low hazard level, and packed and shipped within 24 hours of collection.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with packing and shipping samples include primarily nicks and cuts, and heavy lifting. Modified Level D is generally appropriate, and may be limited to nitrile gloves, safety glasses and safety shoes. High visibility vests should be worn when packing coolers in a parking lot. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 SAMPLE PACKAGING

The following procedure will be used to pack samples for overnight shipment:

1. To the extent possible, group samples by sample ID, with the exception of bottleware designated for volatile organic constituents (VOC) analysis. Group aqueous VOC samples in a common shipping container.

2. At the time of sample collection, wipe the outside of each sample bottle/jar with a paper towel and place a label on each container. Each glass bottle/jar will be wrapped with bubble wrap. Place all sample bottles in a Ziploc® bag. All VOC vials collected per sample shall be placed in the same plastic bag. Trip blank quality control (QC) samples will be packed in the same manner as the VOC bottleware.

3. Remove as much air as possible from the plastic bag prior to sealing.

4. Seal the drains on the shipping coolers with tape.

5. Place an absorbent pad in the bottom of the cooler, followed by a layer of bubble wrap.

6. Insert a plastic liner/contractor bag into the cooler.

7. Place the sample containers inside the liner in an upright position and place sections of bubble wrap in between sample bottles. Group all aqueous VOC samples in a common cooler. Place one trip blank set (two 40-mL volatile organic analysis [VOAs]) in each cooler containing aqueous VOC samples. Place one temperature blank in each cooler.

8. Preserve the samples with ice, using the method preferred by the Client. This may be completed by placing the ice chips directly inside the liner around all sample bottles; or by double-bagging ice chips in Ziploc® bags and placing the bags at the bottom and top of sample batch.

9. Sign and date the chain of custody (COC) and record the information in the field logbook.

10. If a laboratory courier service is arranged, the sampler and courier must both sign and date the COC at the time of transfer. The sampler must then take a photo of the COC before sealing it inside the cooler. Photographs of COCs will be provided to the sample manager at the end of each workday.

11. To properly seal the completed COC inside a cooler, separate the bottom copy and place the remaining pages inside a Ziploc® bag and tape the bag to the inside of the cooler lid.

12. Affix signed custody seals over lid openings (opposite corners of the cooler).

13. Seal both ends of the cooler by wrapping three times with clear packing tape.

14. For shipments through FedEx or UPS, provide the representative with the laboratory shipping and receiving addresses and the Bluestone or laboratory account number, depending on responsible party. In addition, provide any shipping procedures or restrictions the laboratory may require (i.e. no Saturday delivery).

5.0 DATA MANAGEMENT

Data associated with the release of samples to a courier service (i.e., time relinquished, receiving party) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERATING PROCEDURE NO. 15
SUBSURFACE SOIL SAMPLING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for collecting representative subsurface soil samples. Soil quality data generated from chemical analysis of subsurface soils will be used to determine the nature and extent of contamination, if present. In addition, the data will aid in the determination of associated risk posed to human health and the environment, and the most appropriate remedial measures. A subsurface soil investigation is often performed in conjunction with a shallow groundwater investigation, as a borehole may be converted into a temporary groundwater well, through which water quality and groundwater elevation may be evaluated.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated subsurface soil sampling are similar to drilling hazards and include working around heavy machinery, excessive noise, overhead rotating parts, pinch points, heavy lifting and twisting, projectiles, biological hazards (i.e., poison ivy, bees/wasps, spiders, ticks), etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with drilling tasks, and includes a hard hat, safety glasses, steel-toed boots, leather and/or nitrile

gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 PRE-SAMPLING ACTIVITIES

Locations for subsurface soil sampling will be selected based on project objectives, source location, suspected contaminant mobility, and available analytical data. Sample locations will be identified in the Work Plan, Sampling Plan, or Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP).

4.1 Discrete Soil Sampling Requirements

Subsurface soil samples may be collected using a Geoprobe® rig and direct-push technology (DPT), which retrieves soil cores in 4 or 5-foot barrels lined with acetate sleeves. Subsurface samples collected using higher-power drill rigs may be collected in 5 or 10-foot intervals. Soil cores must be logged by a qualified geologist, as described in Bluestone's SOP No. 11 – *Geologic Logging*.

Discrete subsurface soil samples typically target a known or suspected zone of contamination based on elevated readings on the photoionization detector (PID), staining, and/or odors. The sample interval should be centered on the area of the soil core exhibiting the highest PID readings (for volatile organic constituent [VOC] analysis) unless specified in the Work Plan or Uniform Federal Policy (UFP) Quality Assurance Project Plan (QAPP).

4.2 Background Samples

Based on project objectives, background subsurface soil samples may be collected to evaluate areas that are located topographically upgradient of source activities and are representative of site soils and subsurface conditions. Background samples will be collected using like procedures as other subsurface soil samples. Background samples will be analyzed for the same constituents as the remainder of site samples and will be used to evaluate the impact of source activities on surrounding areas.

4.3 Utility Clearance

Prior to any intrusive field activities, subsurface utility clearance must be performed to ensure that no buried utilities are damaged or compromised during a subsurface investigation. Clearance activities will be performed by the respective State service (i.e., One-Call, Dig-Safely, etc.) and by a third-party utility locator. Utility clearance may be completed using a variety of geophysical methods, as described in Bluestone's SOP No. 18 – *Utility Clearance*.

5.0 SUBSURFACE SOIL SAMPLING TECHNIQUES

5.1 Subsurface Sampling Using a Hand-Auger

Investigation of shallow soils may be conducted using a stainless-steel hand-auger. A hand-auger requires physical labor or an electrical source to advance a small-diameter sampling auger into unconsolidated subsurface material. Hand-auger sampling is generally reserved for depths less than 5 ft bgs, although greater depths are possible. All material extracted from the soil boring must be contained on plastic sheeting. Procedures for the disposal of cuttings will be described in the Work Plan or UFP-QAPP, in addition to soil sampling depth(s). Subsurface soil samples are recovered from the hand-auger using a clean, gloved hand, or a decontaminated soil knife. Subsurface soil samples submitted for chemical analysis will be collected from the intervals specified in the QAPP but will not be collected below the groundwater surface.

5.1.1 Equipment

The following equipment may be used for the collection of subsurface samples using DPT technology:

- Sample bottleware
- Camera
- Munsell Soil Color Chart
- Soil boring logs and field forms
- 6-foot measuring tape
- Field logbook
- Spray bottle
- Contractor bags
- Card table

- Duct tape
- Hand lens
- Caution tape
- Spray paint, flagging tape, pin flags
- Lath
- Aluminum foil
- Utility knife
- Stainless-steel bowl
- Stainless-sampling spoons
- Hand-auger with attachable extensions
- Decontamination supplies
- Safety equipment as needed (refer to the Project Accident Prevention Plan [APP] or Health and Safety Plan [HASP]).

5.1.2 Soil Sample Collection Procedures

Subsurface soil samples may be collected either at discrete intervals as specified in the Work Plan or UFP-QAPP, or continuously until refusal is encountered. If the water table is encountered prior to reaching the target depth, the saturated sample should be analyzed for geologic logging and headspace measurements only, and the subsurface soil sample will be collected from the interval directly above the water table. If insufficient volume is available, the sampler may reach into the borehole and scrape the sidewall with a scoop at the target depth or offset slightly and hand-auger a second boring to the target depth. PID measurements will be recorded every foot of the soil core.

5.2 Subsurface Soil Sampling Using Direct-Push Technology (DPT)

Investigation of shallow soils will be conducted using a Geoprobe® rig and direct-push technology. As discussed above, this method employs a hydraulically powered percussion/probing machine to advance 2 to 3-inch barrels lined with acetate sleeves (Macro-Core® or large-bore)into unconsolidated subsurface material. Subsurface soil samples will be collected at the depth(s) specified in the Work Plan or UFP-QAPP. The soil sample is recovered by removing the Macro-

Core® from the sampler and extracting soils from the target interval. No subsurface soil samples will be collected from below the groundwater surface.

5.2.1 Equipment

The following equipment may be needed for collection of subsurface samples using DPT methods:

- Sample bottleware
- Camera
- Munsell Soil Color Chart
- Soil boring logs and field forms
- 6-foot measuring tape
- Field logbook
- Spray bottle
- Contractor bags
- Hand lens
- Caution tape
- Spray paint, flagging tape, pin flags
- Lath
- Aluminum foil
- Utility knife
- Stainless-steel bowl
- Stainless-sampling knife and spoon
- Geoprobe® track rig
- Decontamination supplies
- Safety equipment as needed (refer to the APP/HASP).

5.2.2 Soil Sample Collection Procedures

DPT soil samples may be used for physical/geotechnical and/or chemical analyses. The type of liner used will be selected based on site conditions and project objectives. Acetate liners are suitable for most types of sampling and are less expensive and easier to use than Teflon® liners. However, they may potentially introduce phthalate contamination into the samples. Hence,

acetate liners will not be used at sites where phthalates are considered a constituent of potential concern (COPC), and instead Teflon® liners will be used.

Analytical samples may be collected at discrete intervals as specified in the Work Plan or UFP-QAPP, or continuously using the large-bore (1-3/8-inch outer-diameter [OD]) or Macro-Core® sleeves (2-inch OD). If the water table is encountered prior to reaching the target depth, the saturated portion of soil in the liners will be evaluated for logging purposes and headspace measurements only, and the sample will be collected from the interval directly above the water table. PID measurements will be recorded every 6 to 12 inches of the soil core. If an insufficient volume of soil is collected during sampling, the borehole will be offset slightly, and another soil boring will be advanced to extract the required soil volume.

5.3 Subsurface Soil Sampling Using Split-Barrel and Sonic Drilling

Additional methods for the collection of subsurface soil samples include split-barrel samplers or Shelby tubes, which provide geologic information for logging and/or samples for geotechnical and chemical analyses. Samples collected using a split-barrel sampler are ideal for the drilling of disturbed soils, and the project objectives include chemical analysis and physical characterization of sediment (i.e., grain size distribution, moisture content, etc.). Samples may also be collected using the Sonic drilling method, which is ideal in unconsolidated media.

5.3.1 Equipment

The following equipment may be needed to collect subsurface soil samples using split-barrel or Sonic drilling methods:

- Sample containers
- Camera
- Munsell Soil Color Chart
- Soil boring logs and field forms
- 6-foot measuring tape
- Field logbook
- Spray bottle
- Contractor bags

- Hand lens
- Caution tape
- Spray paint, flagging tape, pin flags
- Lath
- Aluminum foil
- Utility knife
- Stainless-steel bowl
- Stainless-steel sampling knife and spoon
- Drill rig (project-specific); must be capable of sampling the unconsolidated material for chemical analysis using split-barrel sampler
- Decontamination supplies
- Safety equipment as needed (refer to the APP or HASP).

5.3.2 Split – Barrel Sample Procedures

A spit-barrel (split-spoon) sampler operates similarly to DPT, in that no circular drilling is performed; however, it requires a higher-power rig capable of advancing to greater depths. Subsurface soil sampling using split-spoon) drilling methods are described below:

1. Depending on the COPCs, the ambient air surrounding the borehole should be monitored in accordance with Bluestone's SOP No. 07 – *Field Screening Methodology*. Air quality measurements will be recorded after each run, or more frequently if levels are increasing or the alarm is activated.

2. The split-spoon sampler will be removed from the borehole and placed on clean plastic sheeting on a table or ground surface.

3. The split-spoon is opened, and the soil core is screened for the presence of organic vapors using a PID and logged accordingly.

4. To collect a sample, the core may be extracted from the split-spoon using a decontaminated knife or spoon. Samples for VOC and/or total volatile petroleum hydrocarbons (TVPH) analyses will be collected first from the interval exhibiting the highest PID measurements or any visual signs of contamination. Samples for VOC analysis will be collected using sampling equipment identified in the Work Plan or UFP-QAPP directly from the core. Samples will be immediately transferred to the appropriate sample container and placed in an iced cooler to minimize volatilization. In the event no contamination or elevated PID readings are detected, the sample will be collected at the midpoint of the target interval.

5. For all other chemical analyses, the sample will be homogenized to generate a representative sample.by hand mixing (using nitrile gloves) or stirring the remaining soils in a stainless-steel bowl. The sample volume must be sufficient to fill all required sample containers including associated QA/QC analysis. Subsurface soil samples will be collected and preserved as described above.

6. Decontaminate all stainless-steel equipment (including split-spoon barrels) using methods described in Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

5.3.3 Sampling Procedures Using Sonic Drilling

Sonic drilling is ideal for the investigation of unconsolidated materials and is known for producing high-quality continuous soil samples. This technique employs two core barrels, the inner barrel of which is advanced into the subsurface first to obtain an undisturbed soil sample. The outer barrel is then advanced over the inner barrel to the same depth to provide stability of the surrounding formation while the inner barrel is extracted from the borehole. The advancement of the boreholes is accomplished through high-frequency resonant energy. The barrels are advanced in this manner until the target depth or refusal is encountered. Subsurface soil samples collected using Sonic drilling techniques will be follow the procedures listed below:

7. Depending on the COPCs, the ambient air surrounding the borehole should be monitored in accordance with Bluestone's SOP No. 07 – *Field Screening Methodology*. Air quality measurements will be recorded after each run, or more frequently if levels are increasing or the alarm is activated.

8. Soil/unconsolidated material is extracted from the inner barrel that is lined with a heavy-duty plastic sleeve. Sections of core are typically 10 feet in length and 3½-inch diameter. The plastic sleeve is pulled from the inner barrel and placed on plastic sheeting. Field sampling crews will split the lining and core vertically using a stainless-steel knife and log each section as described above.

9. Soil cores will be screened for the presence of VOCs using a PID, as described above.

10. The field sampler will select soil from the core at target sampling depths (and intervals of visual contamination, as applicable) and homogenize as described above.

11. For all other chemical analyses, the sample interval will be homogenized to generate a representative sample by hand mixing (using nitrile gloves) or stirring the remaining soils in a stainless-steel bowl. The sample volume must be sufficient to fill all required sample containers including associated QA/QC analysis. Subsurface soil samples will be collected

and preserved as described above.

12. Decontaminate all stainless-steel equipment (including split-spoon barrels) using methods described in Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

6.0 PRIORITY OF TARGET ANALYTES

Soil samples and QC samples will be collected in the order of the volatilization sensitivity of respective analytes. In general, the following order will be prioritized: VOCs, TVPH, (semi-volatile organic constituents (SVOCs), total extractable petroleum hydrocarbons (TEPH), pesticides, herbicides, inorganics, and lastly, soil properties).

Geotechnical analysis of soil properties will be conducted following chemical analysis. Soil aliquots intended for analysis of physical properties, including moisture content, will be placed in clean glass jars (if American Society for Testing and Materials [ASTM] Method D2216-19: *Standard Test Methods for Laboratory Determination of Water [Moisture] Content of Soil and Rock by Mass*) is required. Remaining sample material will be placed in sealable plastic bags, labeled with the date, associated soil boring ID, and depth. Depending on project objectives, the soil may be tested for Atterberg limits (ASTM Method D4318-17E1: *Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils*) or sieve analysis with U.S. Department of Agriculture (USDA) classification (ASTM D422-63): *Standard Test Method for Particle-Size Analysis of Soils*), if requested.

Many geotechnical analyses require undisturbed samples, which can be acquired using a Shelby tube sampler. Shelby tubes samples are ideal for collection subsurface soil samples in cohesive silts and soft clays. Once collected, the Shelby tube must remain upright and undisturbed during transport and analysis.

7.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

8.0 DATA MANAGEMENT

Data collected during subsurface soil sampling activities (i.e., sample ID, location, depths, media type, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 16
SURFACE SOIL SAMPLING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for the collecting representative surface soil samples for chemical analysis. Soil quality data generated from chemical analysis of surface soils will be used to determine the nature and extent of surface contamination, if present. In addition, the data will aid in the determination of associated risk posed to human health and the environment, and the most appropriate remedial measures.

2.0 PERONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with surface soil sampling may include working around heavy machinery, excessive noise, pinch points, heavy lifting and twisting, and biological hazards (i.e., poison ivy, bees/wasps, spiders, ticks) associated with accessing sample locations, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work and may include a hard hat, safety glasses, steel-toed boots, leather and/or nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 PRE-SAMPLING ACTIVITIES

Locations for surface soil sampling will be selected based on project objectives, source location, suspected contaminant mobility, and available analytical data. In general, surface samples are placed to provide adequate spatial coverage of the site or area of concern (AOC), with additional attention to areas of known or historical impacts. Sample locations will be identified in the Work Plan, Sampling Plan or Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP).

5.0 BACKGROUND SAMPLES

Based on project objectives, background surface soil samples may be collected to evaluate areas that are located topographically upgradient of source activities and are representative of site soils and surface conditions. Background samples will be collected using like procedures as other surface soil samples. Background samples will be analyzed for the same constituents as the remainder of site samples and will be used to evaluate the impact of source activities on surrounding areas.

6.0 SURFACE SOIL SAMPLING TECHNIQUES

6.1 (Grab) Surface Soil Sampling

Investigation of surface soils may be conducted using the grab sample method, as described below:

1. Clear an approximately one square-foot area of any obstructions, debris or surface vegetation.

2. Depending on the surface material (i.e., loose gravel, crushed stone, sod, etc.), remove the non-soil media to expose the soil beneath using a trowel or hand shovel. Collect the surface soil sample at the target depth, as specified in the Work Plan or UFP-QAPP.

3. Transfer the soil into a stainless-steel bowl and remove any non-soil materials (i.e., pebbles, sticks, leaves, debris, etc.).

4. Set aside the soil aliquot for volatile organic constituent (VOC) analysis; homogenize the remaining sample volume by stirring with a stainless-steel spoon or by hand mixing (using nitrile gloves).

5. Transfer soil into corresponding sample containers and label appropriately. Record relevant sampling information in the field logbook.

6. Backfill the surface depression/void with clean topsoil and replace the surface material (i.e., loose gravel, crushed stone, sod, etc.).

6.1.1 Equipment

The following equipment may be needed to collect a grab surface soil sample:

- Stainless-steel hand-auger, scoop, trowel, or shovel
- Sample bottleware
- Camera
- Munsell Soil Color Chart
- Field logbook
- Spray bottle
- Contractor bags
- Hand lens
- Caution tape
- Spray paint, flagging tape, pin flags
- Lath
- Aluminum foil
- Utility knife
- Stainless-steel bowl
- Stainless-steel sampling knife and spoon
- Decontamination supplies
- Applicable safety equipment (refer to the Project Accident Prevention Plan [APP] or Health and Safety Plan [HASP]).

6.1.2 Surface Soil Sampling Devices

Sampling devices, as detailed below, will be selected based on project objectives and site conditions.

Hand Scoops and Trowels: These devices may be laboratory-grade or landscape-variety and are typically constructed of stainless-steel or plastics (polyethylene, polystyrene, polycarbonate, etc.). The scoop or trowel is preferred when collecting grab surface samples in areas easily accessed.

Spades and Shovels: These tools may be used to collect surface soil samples but should be constructed with steel or stain-less steel. Care must be exercised to avoid the use of devices

plated with chrome, or other exterior coatings that may chemically alter the sample. These devices are easy to use and decontaminate, and work well for sampling; however, disposable boot covers may be necessary to avoid cross-contamination through footwear.

Tube Auger: A tube auger is typically constructed of stainless-steel and includes the auger bit, pole extension and handle. Depending on project objectives and surface soil conditions, an acetate sleeve liner may be inserted into the auger to contain the sample. The auger is driven into the surface soil with downward pressure to the target depth and retrieved slowly. Water collected at the top of the bucket/tube auger is carefully decanted to minimize the loss of fines-grained particles.

7.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

8.0 DATA MANAGEMENT

Data collected during surface soil sampling activities (i.e., sample ID, location, time of collection, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 18
UTILITY CLEARANCE

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for performing subsurface utility clearance activities. Since Bluestone does not self-perform utility clearance, these procedures describe the requirements and quality assurance (QC) protocols that must be followed during oversight of utility clearance subcontractors. In addition, most of the safety hazards and protocols apply to overhead utilities as well.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

Site personnel should be familiar with the appropriate telephone numbers, standard utility-specific color codes (provided below), and appropriate response actions in the event a line is compromised or damaged. All site personnel should be able to identify utility markings and communicate to emergency responders, if needed.

RED	ELECTRIC
YELLOW	GAS, OIL, STEAM
ORANGE	COMMUNICATIONS
BLUE	POTABLE WATER
PURPLE	RECLAIMED WATER
GREEN	SEWER / DRAINAGE
PINK	SURVEY MARKS
WHITE	PROPOSED EXCAVATION

3.0 HEALTH AND SAFETY HAZARDS

Field activities as detailed in this SOP will be performed in accordance with applicable safety related documents and requirements which may include, but are not limited to: Site Safety and Health Plans, the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with utility clearance activities include primarily trip hazards and possible electrocution. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with oversight of utility clearance, and includes a hard hat, safety glasses, safety shoes and high-visibility vests. Additional details regarding site safety and health must be provided in the Project Accident Prevention Plan (APP) or Site Health and Safety Plan (HASP).

4.0 UTILITY CLEARANCE ACTIVITIES

Prior to any intrusive field activities, subsurface utility clearance must be performed to ensure that no buried utilities are damaged or compromised during a subsurface investigation. Specific tasks include mark sample/work locations, notify the appropriate local/regional utility location services (i.e., PA One-Call, Dig-Safely New York, etc.), document and maintain the marked utilities. The call to local/regional utility locators must be placed a minimum of 72 hours in advance and should specify the length of intrusive work. The approval period may be extended beyond the typical 10 days or renewed as required for the duration of the project. Pertinent information, including the regional call ticket number, utilities notified and responded, period of approval, and names of all personnel granting clearance should be recorded in the field logbook, in accordance with Bluestone's SOP No. 06 – *Field Documentation*.

As an added measure of precaution, and to identify subsurface utilities on privately-owned parcels, a third-party utility locator will be subcontracted. Utility clearance will be performed using a variety of geophysical methods, including ground-penetrating radar (GPR), precision utility locating (PUL), and metered magnetic locating, etc.

5.0 INTRUSIVE ACTIVITIES

No intrusive activities will be performed within five feet of a marked utility. Any proposed sample locations, as specified in the Work Plan or Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP), within five feet of a utility will be shifted accordingly. In addition, drill rigs will be positioned so that the mast is kept a minimum of 30 lateral feet from overhead powerlines. Vehicles will be parked a minimum of 30 lateral feet from overhead utilities/powerlines to reduce the possibility of arcing. The minimum lateral distance may be adjusted based on known voltages of overhead lines, or if specified in the APP and/or HASP.

The relocation of soil borings to avoid subsurface utilities must be approved by the Field Team Leader (FTL) and documented in the field logbook. The FTL should notify the Project Manager within 24 hours; however, approval to proceed with work is not required.

6.0 EQUIPMENT AND SUPPLIES

Equipment used during a utility clearance survey includes:

- Project documents (Work Plan, UFP-QAPP, APP, HASP)
- Field logbook, field data sheets
- Hand-held GPS unit (TDS Trimble® Ranger 3L, or similar)
- Marking paint (designated industry colors)
- Pin flags, stakes, ribbon flagging (designated industry colors)
- Geophysical instruments (provided by the subcontractor) included but not limited to:
  - GSSI® Utility Scan Pro GPR, or similar
  - Schonstedt® Magnetic locator, or similar
  - PUL equipment (RD5100™) or similar.

7.0 DATA MANAGEMENT

Data collected during utility clearance activities (i.e., location and depth of utilities, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field

data and observations should be provided to the Project Manager periodically throughout the progression of work.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

STANDARD OPERATING PROCEDURE NO. 19
GROUNDWATER ELEVATION MONITORING

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) was prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the methodology for collecting depth-to-water measurements from monitoring wells and piezometers (terms used interchangeably). Hydrogeologic data gathered during a field investigation may be used to determine hydraulic gradient, interpret the migration rate and direction of contaminants (if present), produce groundwater contour maps, determine purge volumes for groundwater sampling, and design slug tests, packer tests and constant-rate pumping tests, etc.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY HAZARDS

Field activities as detailed in this SOP will be performed in accordance with applicable safety related documents and requirements which may include, but are not limited to: Site Safety and Health Plans, the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with groundwater elevation monitoring include biological hazards (i.e., poison ivy, wasps/bees, spiders, ticks, etc.) slip/trip/fall hazards and pinch points. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work associated with groundwater elevation monitoring, and includes a hard hat, safety glasses, leather/nitrile gloves, safety shoes and high-visibility vests. Additional details regarding site safety and health must be provided in the Project Accident Prevention Plan (APP) or Site Health and Safety Plan (HASP).

4.0 EQUIPMENT

Equipment used in the collection of depth-to-water measurements:

- Electronic water level indicator (Solinst® Model 101 P7 Water Level Meter or similar)
- Oil/water interface probe (Solinst® Model 122 or similar)
- Alconox®, Liquinox® or other non-phosphate concentrated laboratory grade soap
- De-ionized water
- Spray bottles
- PPE
- Air Monitoring instruments as required (MiniRAE®, Thermo TVA 1000 FID/PID, etc.)
- Field logbook, field data sheets
- Well keys
- Decontamination supplies
- Previous depth-to-water measurements (if available).

5.0 PROCEDURES FOR MEASURING DETH-TO-WATER

The procedures for collecting depth-to-water measurements include the following:

1. Record the condition of the well (protective casing, concrete collar, lock in place etc.).

2. Check the water level tape has no obvious kinks or damage.

3. Using nitrile gloves, decontaminate the water level meter in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

4. Stand upwind of the well; unlock and open the well, being cautious of biological hazards. Remove the well cap and collect a headspace reading using a PID. Record pertinent air monitoring data (sustained, dissipating, background, odor) in the field logbook in accordance with Bluestone's SOP No. 06 – *Field Documentation*.

5. Identify the survey notch on the riser or casing (if present). Record this location in the field logbook or field data sheet.

6. Activate the water level meter, check the audible indicator, reel the electronic probe into the well riser (with the increments visible) slowly until the meter sounds, grasp the tape with hand, withdraw the tape and lower it again slowly until the sound is again audible. Check the depth to water on the tape and make a mental note of the depth to within .01 feet. Lower the probe again slowly and repeat the measurement for accuracy, with care to measure from the correct direction.

7. Record the depth-to-water measurements in the field logbook or field data sheets and corresponding time of measurement.

8. Procedures implemented in the presence of free phase petroleum products (light non-aqueous phase liquids [LNAPL]) on the surface of the water table should be modified to include the use of the oil/water interface probe. The procedures during the use of this probe should be implemented similarly and by manufacturers' specifications. Depth-to-product and depth-to-water measurements will be recorded, which in turn provides the thickness of the free product layer.

6.0 EQUIPMENT DECONTAMINATION

Decontamination procedures for re-usable field equipment must be performed in accordance with Bluestone's SOP No. 05 – *Decontamination of Field Equipment*.

7.0 DATA MANAGEMENT

Data collected during groundwater elevation monitoring (i.e., depth-to-groundwater and total depth measurements, and corresponding times, etc.) will be recorded in the field logbook and on field data sheets as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

THIS PAGE LEFT BLANK INTENTIONALLY

STANDARD OPERTATING PROCEDURE NO. 21
INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

1.0 INTRODUCTION AND PURPOSE

This Standard Operating Procedure (SOP) has been prepared by Bluestone Environmental Group, Inc. (Bluestone) to describe the general approach for collecting soil samples (surface and subsurface) using incremental sampling methodology (ISM) for analysis of non-volatile constituents. The following regulatory guidance documents were referenced for the preparation of this SOP:

- Interstate Technology and Regulatory Council (ITRC) guidance document, *Incremental Sampling Methodology* (2012); and,
- U.S. Environmental Protection Agency (EPA), Standard Operating Procedure for Incremental Sampling Methodology for Soil (effective 12/15/15).

The rationale for the use of ISM is comprehensively described in the aforementioned documents. The ISM approach incorporates two principal components: 1) field sampling methodology and 2) laboratory preparation and processing. This SOP focuses on the field collection method. Preparation and processing at the laboratory are equally as important as the field collection procedures.

ISM sampling may be used to determine both the presence/absence of contaminants, and to evaluate human health and ecological risk, by controlling variability associated with the heterogenous distribution of contaminants in soil.

2.0 PERSONNEL QUALIFICATIONS

All Bluestone field personnel must complete the Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operators (HAZWOPER) training course and maintain the required 8-Hour annual refresher courses. In addition, site personnel must be certified in first aid and CPR.

3.0 HEALTH AND SAFETY

Field activities as detailed in this SOP will be performed in accordance with the Project Accident Prevention Plan (APP) and/or Health and Safety Plan (HASP), the Bluestone Corporate Safety and Health Program and site and/or client-specific requirements. Potential health and safety hazards associated with surface soil sampling may include working around heavy machinery, excessive noise, pinch points, heavy lifting and twisting, and biological hazards (e.g., poison ivy, bees/wasps, spiders, ticks) associated with accessing sample locations, etc. Modified Level D Personal Protective Equipment (PPE) is generally appropriate for work and may include a hard hat, safety glasses, steel-toed boots, leather and/or nitrile gloves, high-visibility vests, and hearing protection. Additional details regarding site safety and health must be provided in the APP and/or HASP.

4.0 DECISION UNITS

The objective of ISM is to determine a representative estimate of the mean concentration of a contaminant within a pre-determined area, referred to as a Decision Unit (DU). A DU represents the smallest volume of soil for which a characterization, risk-based, or remedial decision is made. DUs are based on project-specific needs and site-specific data quality objectives (DQOs). A DU may consist of one or more sampling units (SUs). SUs are subdivisions of DUs from which separate ISM samples are collected. SUs define the scale of the ISM sampling; whereas DUs define the scale of the decision based on the sampling (ITRC, 2012).

As noted by EPA (2015), DUs may be defined with a general understanding of a host of site characteristics, including the vertical and horizontal distribution of contaminants across a site, known presence of source areas, transport and migration pathways, geologic formations, human and/or ecological risk model requirements, soil types, property boundaries, property use (such as residential, farming, industrial), and sampling goals. In some cases, the entire site may be classified as a single DU; in other cases, the site may be partitioned into smaller DUs, each with its own sampling goals. For the purposes of this SOP, it is assumed that DUs and SUs have been previously identified and delineated.

5.0 SAMPLE INCREMENTS AND REPLICATES

For each area to be sampled (i.e., a DU or SU), a surface soil ISM sample will consist of a minimum of 30 increments that will be collected from the 0 to 1 foot below ground surface (ft bgs) interval. For quality control (QC) purposes, ISM samples are typically collected in triplicate sample sets, and organized as follows:

- Primary Sample (first path of travel);
- Duplicate Sample (second path of travel); and,
- Triplicate Sample (third path of travel).

Corresponding soil increments will be distinguished by a designated color of pin flag, and will be collected from a consistent location within each grid cell.

6.0 PROCEDURE FOR COLLECTING 30-INCREMENT SURFACE SOIL ISM SAMPLE (NON-VOLATILE ANALYSIS ONLY)

The steps below describe the general approach used to collect a 30-increment surface soil sample from one SU:

1. Locate the boundary of the SU using a hand-held Trimble® GPS unit, with pre-loaded coordinates, and mark the corners with wooden stakes and/or pin flags. For irregularly shaped SUs, stakes should mark the major vertices.

2. Assign one field team member to pace the length and width of the SU, by counting the paces for each direction. This information will be used to efficiently develop a grid for the SU, and ensures that the 30 sample increments are evenly distributed within the grid.

3. Assign a separate color pin flag to mark each of the three ISM samples, and place each color in a consistent location within each grid cell.

4. A random sample location will be identified in the first sampling grid cell. The same location will be replicated in each subsequent grid cell.

5. Placement of primary and replicate samples within each grid cell will be documented in the field logbook(s).

6. A serpentine pattern will be followed to collect primary and replicate samples. The serpentine pattern will alternate between starting in the X or Y direction for each replicate. Illustration 1 shows a simplified example of sample collection, in which it is

assumed that the 'X' symbol represents the primary sample, the 'O' symbol represents the duplicate sample, and the 'Δ' symbol represents the triplicate sample.

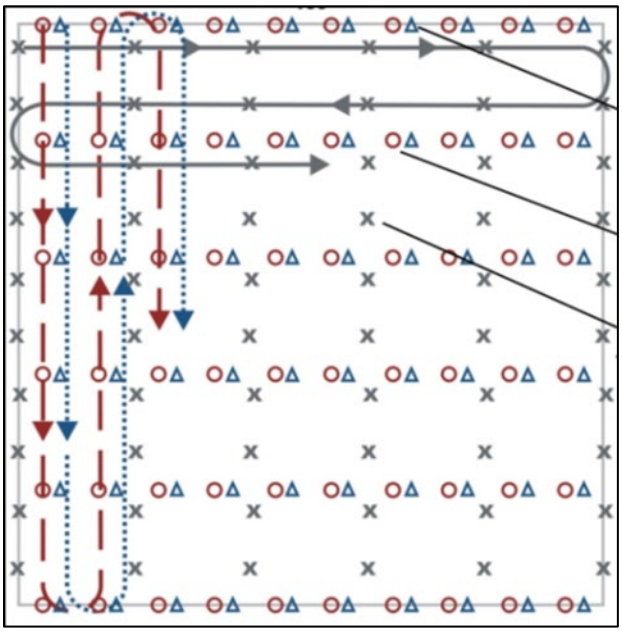


Illustration 1 – ISM serpentine pattern

7. Sample increments will be marked and verified during a trial run along the proposed paths to ensure spacing is adequate to collect 30 soil increments.

8. Collect soil increment using a dedicated volumetric sampling device (AMS® soil probe, or similar). Use care to obtain equal sample mass from each soil increment.

9. Complete field notes describing the sample increment, noting collection depth, location, a general description and if there is any odor.

10. Place soil increment into the designated sample container.

11. Continue to the next soil increment, and repeat steps #8 through 11. Repeat this process, following the designated pattern until all 30 increments for the primary sample are collected.

12. Place bulk sample into clean sample container. In order to comply with laboratory standards, each composited ISM sample should be a maximum volume approximately 1,000 grams. As such, the sampling tool must be conducive to collecting a volume of 30 to 35 grams of soil per increment. Soil increments will be transferred into the laboratory-prepared plastic bags for homogenization by the laboratory prior to chemical analysis.

13. Label each sample container and preserve in iced cooler.

The duplicate and triplicate surface soil ISM QC sample will be collected using the sample method.

6.1 Additional Surface Soil ISM Sampling Protocols

1. All surface vegetation or non-soil material/debris will be removed from the ground surface prior to sampling.

2. Sampling teams will don the appropriate personal protective equipment (PPE), at minimum, nitrile gloves, high-visibility vests, eye protection (nitrile gloves will be changed between samples and DU/SUs).

3. If refusal is encountered at a sampling location, the increment will be off-set slightly, but will remain as close as possible to the original location.

4. All surface sample locations will be backfilled to grade using clean topsoil.

7.0 PROCEDURE FOR COLLECTING SUBSURFACE SOIL ISM SAMPLE (NON-VOLATILE ANALYSIS ONLY)

The procedure for collecting subsurface ISM samples is relatively similar to the surface soil approach, with the exceptions noted below:

1. Establish the sampling grid using steps #1 through 7, as detailed in Section 6.0 above.

2. Use a Geoprobe® Direct-Push Technology (DPT) rig to advance soil borings. Extract the full, pre-determined length of soil cores prior to sampling.

3. Complete field notes describing the sample increment, noting collection depth, location, a general description and if there is any odor.

4. Once a soil core is collected, use the wedge method to collect the sample from the full length of the core. Place the wedge of soil in a sample container.

Note: If the sampling plan requires the collection of discrete volatile organic compound (VOC) samples, the VOC samples must be collected prior to disturbance of the core.

5. Mobilize the Geoprobe® rig to the next soil boring increment, and repeat steps #1 through 3. This will be repeated until wedges are collected from each section of core.

Repeat steps #2 through 5 to collect the duplicate and triplicate subsurface soil ISM samples.

7.1 Additional Subsurface Soil ISM Sampling Protocols

1. All surface vegetation or non-soil/debris material will be removed from the ground surface prior to sampling. Soil borings advanced through asphalt paving or concrete will be restored with like surface materials.

2. Sampling teams will don the appropriate personal protective equipment (PPE), at minimum, nitrile gloves, high-visibility vests, eye protection (nitrile gloves will be changed between samples and DU/SUs).

3. Place bulk wedge increments into designated sample container. In order to comply with laboratory standards, each composited ISM sample should be a maximum volume approximately 1,000 grams. As such, the sampling tool must be conducive to collecting a volume of 30 to 35 grams of soil per increment. A digital scale will be used to ensure that appropriate soil volumes are collected. Soil increments will be transferred into the laboratory-prepared plastic bags for homogenization by the laboratory prior to chemical analysis.

4. If refusal is encountered at a sampling location, the increment will be off-set slightly, but will remain as close as possible to the original location.

5. All borings will be backfilled to grade using clean topsoil or other like materials (i.e., asphalt or concrete).

8.0 DECONTAMINATION PROCEDURES

Decontamination of sampling equipment is necessary only between complete ISM sample sets.

Decontamination procedures are provided in Bluestone's SOP No. 05 – Decontamination of Field Equipment.

9.0 SOIL SAMPLING EQUIPMENT LIST

The following list of equipment and supplies may be needed during soil ISM sampling:

- Maps/plot plan
- Safety equipment
- Global positioning system (GPS)
- Survey stakes, flags, string
- Camera
- Stainless steel, plastic, or other appropriate composition bucket
- One-quart wide mouth jars w/Teflon® lined lids for non-volatile samples
- Ziploc® plastic bags
- Logbook or field data sheets

- Sample jar labels
- Chain of Custody records
- Chain of Custody seals
- Cooler(s)
- Ice
- Decontamination supplies/equipment
- AMS® soil probe or similar coring device
- Geoprobe® DPT Rig
- Applicable safety equipment (refer to the Project Accident Prevention Plan)
- Hand sprayer or wash bottle
- Aluminum foil
- Utility knife

10.0 SOIL SAMPLE COLLECTION – ADDITIONAL CRITERIA

- Field Measurement Procedures and Criteria: Any soil collected will be screened for the presence of organic vapors using a PID, and all results will be recorded in the field logbook.
- Investigation-Derived Waste (IDW) generated during the ISM sampling and/or decontamination procedures will be managed in accordance with the procedures detailed in Bluestone's SOP No. 10 – *Storage and Sampling of IDW*.

11.0 DATA MANAGEMENT

Data collected during ISM sampling activities (i.e., sample ID, location, time of collection, etc.) will be recorded in the field logbook and on field data sheets, as necessary. Bluestone's SOP No. 06 – *Field Documentation* details the methods for data collection and management, and additional information should be specified in the Work Plan. Pre-existing conditions and post-work site restoration should be documented through photographs with approval from the Client. Field data and observations should be provided to the Project Manager periodically throughout the progression of work.

12.0 QUALITY ASSURANCE/QUALITY CONTROL

Prior to the start of any field activity, Bluestone personnel must read and acknowledge the Work Plan, UFP-QAPP, and any applicable SOPs. Field personnel will complete a minimum of 40 hours of field training prior to working independently on environmental sites.

APPENDIX C

Laboratory Accreditation

Eurofins Test America Certifications

Denver

Accredited Laboratory

A2LA has accredited

TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Presented this 12th day of August 2019.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2021

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Roxanne Sullivan Phone: 303-736-0100
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2021 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Metals			
Aluminum	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Antimony	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Arsenic	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Barium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Beryllium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Boron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Cadmium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Calcium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Chromium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B

5202 Presidents Court, Suite 220 | Frederick, MD 21703-8398 | Phone: 301 644 3248 | Fax: 240 454 9449 | www.A2LA.org

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Cobalt	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Copper	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Iron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Lead	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Lithium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Magnesium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Manganese	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Mercury	EPA 245.1	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nickel	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Potassium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Selenium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Silica	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silicon	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silver	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sodium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Strontium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Thallium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Thorium	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Tin	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Titanium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Tungsten	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Uranium	EPA 200.8	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Vanadium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zinc	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nutrients			
Nitrate (as N)	By calculation	EPA 300.0 EPA 9056/9056A By Calculation/Nitrate by Calc	EPA 9056/9056A By Calculation/Nitrate by Calc
Nitrate-nitrite (as N)	EPA 353.2	EPA 300.0 EPA 353.2 EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	EPA 353.2 SM 4500-NO_2 B	EPA 300.0 EPA 353.2 EPA 9056/9056A SM 4500-NO_2 B	EPA 353.2 EPA 9056/9056A

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Orthophosphate (as P)	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Total Phosphorus	EPA 365.1	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Demands			
Total Organic Carbon	----------	EPA 9060/9060A	EPA 9060/9060A
Total Organic Halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinty)	SM 2320B-1997	SM 2320B	SM 2320B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chloride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	----------
Conductivity	----------	EPA 9050/9050A	EPA 9050/9050A
Cyanide	----------	EPA 9012A/9012B	EPA 9012A/9012B
Ferrous iron	SM 3500Fe B, D	SM 3500Fe B, D	----------
Fluoride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Flashpoint	----------	EPA 1010A	----------
Hexavalent chromium	----------	EPA 7196A	EPA 7196A
pH	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Oil and Grease (HEM and SGT-HEM)	----------	EPA 1664A/1664B	EPA 9071B
Percent moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	----------
Solids, total	----------	----------	SM 2540B
Solids, Total Suspended	SM 2540D	SM 2540D	SM 2540D
Solids, Total Dissolved	SM 2540C	SM 2540C	SM 2540C
Sulfate	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Sulfide, Total	SM 4500S2 D	EPA 9034/SM 4500S2 D	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	EPA 351.2
Purgeable Organics (Volatiles)			
Acetone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Acetonitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
Acrolein	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Acrylonitrile	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Allyl Chloride	----------	EPA 8260B/8260C	EPA 8260B/8260C
Benzene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C/ AK101/OK DEQ GRO
Benzyl Chloride	----------	EPA 8260B/8260C/8260C	EPA 8260B/8260C/8260C

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Bromobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Bromochloromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Bromodichloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Bromoform	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Bromomethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Butadiene	----------	----------	----------
2-Butanone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
n-Butyl alcohol	----------	EPA 8260B/8260C EPA 8015B/8015C	EPA 8260B/8260C EPA 8015B/8015C
tert-Butyl alcohol (2-Methyl-2-propanol)	----------	EPA 8260B/8260C	EPA 8260B/8260C
n-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
sec-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
tert-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Carbon disulfide	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Carbon tetrachloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Chloro-1,3-butadiene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Chloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Chloroethyl vinyl ether	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chloroform	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1-Chlorohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Chloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chloroprene	----------	EPA 8260B/8260C	EPA 8260B/8260C
4-Chlorotoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
2-Chlorotoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Cyclohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Cyclohexanone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Dibromochloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
Dibromochloromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Dichlorodifluoromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Dibromomethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2 Dibromoethane (EDB)	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
1,2-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Dichlorodifluoromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Dichlorofluoromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloropropane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichloropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
2,2-Dichloropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloropropene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Diethyl ether	----------	EPA 8260B/8260C	EPA 8260B/8260C
Di-isopropylether	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Dioxane	EPA 624/624.1	EPA 8260B/8260C EPA 8260B/8260C SIM	EPA 8260B/8260C EPA 8260B/8260C SIM
Ethanol	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl acetate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl benzene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
Ethyl methacrylate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl tert-butyl ether	----------	----------	----------
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx
Hexane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Hexanone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Hexachlorobutadiene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isopropyl alcohol	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isopropylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Isopropyltoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Iodomethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methacrylonitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl acetate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl cyclohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methylene chloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Methyl ethyl ketone (MEK)	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl isobutyl ketone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl methacrylate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl tert-butyl ether (MtBE)	EPA 624/624.1	EPA 8260B/8260C OK DEQ GRO	EPA 8260B/8260C OK DEQ GRO
4-Methyl-2-pentanone (MIBK)	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Naphthalene	EPA 624/624.1	EPA 8260B/8260C OK DEQ GRO	EPA 8260B/8260C OK DEQ GRO
2-Nitropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Pentachloroethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
2-Pentanone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Propionitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
n-Propylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Styrene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
1,1,1,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1,2,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Tetrachloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Tetrahydrofuran	----------	EPA 8260B/8260C	EPA 8260B/8260C
Toluene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	EPA 1664A/1664B	EPA 1664A/1664B	----------
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3,5-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1,1-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1,2-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Trichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Trichlorofluoromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,4-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trichloropropane	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,4-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3,5-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Vinyl acetate	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Vinyl chloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Xylenes, Total	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
1,2-Xylene (o-Xylene)	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
m+p-Xylene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/ K DEQ GRO
Methane	----------	RSK-175	----------
Ethane	----------	RSK-175	----------
Ethylene (Ethene)	----------	RSK-175	----------
Acetylene	----------	RSK-175	----------
Acetylene ethane	----------	RSK-175	----------
Extractable Organics (Semivolatiles)			
Acenaphthene	EPA 625/625.1/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acenaphthylene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acetophenone	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Acetylaminofluorene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Alachlor	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Aniline	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Aramite	----------	EPA 8270C/8270D	EPA 8270C/8270D
Atrazine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Azobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzaldehyde	----------	EPA 8270C 8270D	EPA 8270C/8270D
Benzidine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzo(a)anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(b)fluoranthene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(k)fluoranthene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(ghi)perylene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(a)pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzyl alcohol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,1-Biphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethoxy) methane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethyl) ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroisopropyl) ether (2,2'Oxybis(1-chloropropane)	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Ethylhexyl) phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Bromophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
butyl Benzyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-sec-butyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Caprolactam	----------	EPA 8270C/8270D	EPA 8270C/8270D
Carbazole	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroanilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Chlorobenzilate	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-chloro-3-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1-Chloronaphthalene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Chrysene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Cresols	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diallate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Dibenzo (a,h) anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Dibenzofuran	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dichlorobenzidine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
2,4-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate	----------	EPA 8270C/8270D	EPA 8270C/8270D
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D	EPA 8270C/8270D
alpha-, alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dimethylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
di-n-butyl Phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
di-n-octyl Phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4,6-Dinitro-2-methylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dinoseb	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dioxane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Diphenylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx
Ethyl Methanesulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Famphur	----------	EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene	EPA 625/625.1	EPA 8270C/8270D EPA8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Fluorene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Hexachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorobutadiene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloroethane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorophene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloropropene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Isodrin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Isophorone	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole	----------	EPA 8270C/8270D	EPA 8270 C/8270D
Methapyrilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-methyl-4,6-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
methyl Methane sulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
3+4-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
1,4-Naphthoquinone	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiphenylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomorpholine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
5-nitro-o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,2-oxybis(1-chloropropane)	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, methyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloroethane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol	EPA 625/625.1	EPA 8270C/8270D EPA 8321A/8321B	EPA 8270C/8270D EPA 8321A/8321B
Phenacetin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Phenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
p-Phenylene Diamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Phorate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pronamide	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Pyridine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Safrole	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Sulfotepp	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Thionazin	----------	EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Tribromophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Tributyl phosphate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4,5-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
o,o,o-triethyl Phosphorothioate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C/8015D AK103/OK DEQ RRO	EPA 8015B/ 8015C/8015D AK103/ OK DEQ RRO
Pesticides/Herbicides/PCBs			
Aldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
beta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-Chlordane	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4-D	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dalapon	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
2,4-DB	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
4,4'-DDD	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dichloroprop	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Dieldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb	----------	EPA 8151A EPA 8321A	EPA 8321A
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endosulfan I	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 608/608.3	EPA 8081A /8081B	EPA 8081A/8081B
Endonsulfan sulfate	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl Parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Heptachlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
MCPP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA8321A
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1242	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Total PCBs	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Picrolam	----------	EPA 8151A	EPA 8151A
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8141A/8141B	EPA 8141A/8141B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-T	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-TP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Toxaphene	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-Triethylphos Phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-amino-4,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-amino-2,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
DNX	----------	EPA 8330A/8330B	EPA 8330A/8330B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
MNX	----------	EPA 8330A/8330B	EPA 8330A/8330B
TNX	----------	EPA 8330A/8330B	EPA 8330A/8330B
Explosives LC/MS/MS			
1,3,5-Trinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Diamino-4-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Diamino-6-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Amino-4,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Amino-2,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
DNX (hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
MNX (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroglycerin	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8321A/8321B	EPA 8321A/8321B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8321A/8321B	EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tris(o-cresyl)phosphate	----------	EPA 8321A/8321B	EPA 8321A/8321B
Chemical Warfare Agents			
Thiodiglycol (2,2'-Thiodiethanol)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Hazardous Waste Characteristics			
Conductivity	SM 2510B	EPA 9050A	EPA 9050A
Corrosivity	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Paint filter liquids test	----------	EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	EPA 1311	EPA 1311
Turbidity	EPA 180.1	----------	----------
Organic Prep Methods			
Separatory funnel liquid-liquid extraction	----------	EPA 3510C	----------

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Continuous liquid-liquid extraction	----------	EPA 3520C	----------
Soxhlet extraction	----------	----------	EPA 3540C
Microwave extraction	----------	----------	EPA 3546
Ultrasonic extraction	----------	----------	EPA 3550B/3550C
Waste dilution	----------	EPA 3580A	EPA 3580A
Solid phase extraction	----------	EPA 3535A	----------
Volatiles purge and trap	----------	EPA 5030B	EPA 5030A EPA 5035/5035A
Organic Cleanup Procedures			
Florisil cleanup	----------	EPA 3620B	EPA 3620B
Florisil cleanup	----------	EPA 3620C	EPA 3620C
Sulfur cleanup	----------	EPA 3660A	EPA 3660A
Sulfuric acid/Permanganate cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid digestion total recoverable or dissolved metals	----------	EPA 3005A	----------
Acid digestion for total metals	----------	EPA 3010A	----------
Acid digestion for total metals	----------	EPA 3020A	----------
Acid digestion of sediments, sludges and soils	----------	----------	EPA 3050B

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

WYOMING STORAGE TANK PROGRAM

Parameter/Analyte	Method(s)
Metals	
Cadmium	EPA 6010C/6010D
Chromium	EPA 6010C/6010D
Lead	EPA 6010C/6010D
Wet Chemistry	
Hexavalent chromium	EPA 7196A
Pureable Organics (Volatiles)	
tert-Amyl Methyl Ether	EPA 8260B/8260C
Benzene	EPA 8260B/8260C
tert-Butyl alcohol (2-Methyl-2-propanol)	EPA 8260B/8260C
1,2-Dichloroethane	EPA 8260B/8260C
Di-isopropylether	EPA 8260B/8260C
Ethyl benzene	EPA 8260B/8260C
Ethyl tert-butyl ether	EPA 8260B/8260C
Gas Range Organics (GRO)	EPA 8015B/8015C/8015D

Parameter/Analyte	**Method(s)**
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C
Naphthalene	EPA 8260B/8260C
Toluene	EPA 8260B/8260C
Xylenes, total	EPA 8260B/8260C
1,2-Xylene	EPA 8260B/8260C
M+P-Xylene	EPA 8260B/8260C
Extractable Organics (Semivolatiles)	
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D (WY: C10-C32)
Organic Prep Methods	
Volatiles purge and trap	EPA 5030B (water) /5030A (solids)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Trihalomethanes	
Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2
Volatile Aromatics	
1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
Volatile Aromatics	
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2

Serial No.: 61819

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Volatile Halocarbons

Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

Serial No.: 61819

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
	EPA 624.1
Acrylonitrile	EPA 8260C
	EPA 624.1
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Methapyrilene	EPA 8270D
Pronamide	EPA 8270D
Propionitrile	EPA 8260C
Pyridine	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 625.1
	EPA 8270D
3,3'-Dimethylbenzidine	EPA 8270D

Benzidines	
Benzidine	EPA 625.1
	EPA 8270D
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 8081B
	EPA 608.3
4,4'-DDE	EPA 8081B
	EPA 608.3
4,4'-DDT	EPA 8081B
	EPA 608.3
Aldrin	EPA 8081B
	EPA 608.3
alpha-BHC	EPA 8081B
	EPA 608.3
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
	EPA 608.3
Chlordane Total	EPA 8081B
	EPA 608.3
Chlorobenzilate	EPA 8081B
	EPA 8270D
delta-BHC	EPA 8081B
	EPA 608.3
Diallate	EPA 8081B
	EPA 8270D
Dicofol	EPA 8081B

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorinated Hydrocarbon Pesticides	
Dieldrin	EPA 8081B
	EPA 608.3
Endosulfan I	EPA 8081B
	EPA 608.3
Endosulfan II	EPA 8081B
	EPA 608.3
Endosulfan sulfate	EPA 8081B
	EPA 608.3
Endrin	EPA 8081B
	EPA 608.3
Endrin aldehyde	EPA 8081B
	EPA 608.3
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
	EPA 608.3
Heptachlor epoxide	EPA 8081B
	EPA 608.3
Isodrin	EPA 8081B
	EPA 8270D
Kepone	EPA 8081B
Methoxychlor	EPA 8081B
	EPA 608.3
Mirex	EPA 8081B
PCNB	EPA 8270D
Toxaphene	EPA 8081B
Chlorinated Hydrocarbon Pesticides	
Toxaphene	EPA 608.3
Chlorinated Hydrocarbons	
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 625.1
	EPA 8270D
2-Chloronaphthalene	EPA 625.1
	EPA 8270D
Hexachlorobenzene	EPA 8081B
	EPA 625.1
	EPA 8270D
Hexachlorobutadiene	EPA 625.1
	EPA 8270D
Hexachlorocyclopentadiene	EPA 625.1
	EPA 8270D
Hexachloroethane	EPA 625.1
	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
	EPA 8321B
2,4,5-TP (Silvex)	EPA 8151A
	EPA 8321B
2,4-D	EPA 8151A
	EPA 8321B

Serial No.: 61627

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorophenoxy Acid Pesticides	
2,4-DB	EPA 8151A
	EPA 8321B
Dalapon	EPA 8151A
	EPA 8321B
Dicamba	EPA 8151A
	EPA 8321B
Dichloroprop	EPA 8151A
	EPA 8321B
Dinoseb	EPA 8151A
	EPA 8270D
	EPA 8321B
Pentachlorophenol	EPA 8151A
Demand	
Biochemical Oxygen Demand	SM 5210B-2011
Carbonaceous BOD	SM 5210B-2011
Chemical Oxygen Demand	EPA 410.4, Rev. 2.0 (1993)
Dissolved Gases	
Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175
Fuel Oxygenates	
Methyl tert-butyl ether	EPA 8260C
Fuel Oxygenates	
Methyl tert-butyl ether	EPA 624.1
Haloethers	
2,2'-Oxybis(1-chloropropane)	EPA 625.1
	EPA 8270D
4-Bromophenylphenyl ether	EPA 625.1
	EPA 8270D
4-Chlorophenylphenyl ether	EPA 625.1
	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 625.1
	EPA 8270D
Bis(2-chloroethyl)ether	EPA 625.1
	EPA 8270D
Low Level Halocarbons	
1,2-Dibromo-3-chloropropane, Low Level	EPA 8011
1,2-Dibromoethane, Low Level	EPA 8011
Low Level Polynuclear Aromatics	
Acenaphthene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Acenaphthylene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM

Serial No.: 61627

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Low Level Polynuclear Aromatics	
Anthracene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Benzo(a)anthracene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Benzo(a)pyrene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Benzo(b)fluoranthene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Benzo(g,h,i)perylene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Benzo(k)fluoranthene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Chrysene Low Level	EPA 8310
	EPA 610

Low Level Polynuclear Aromatics	
Chrysene Low Level	EPA 8270D
	EPA 8270D SIM
Dibenzo(a,h)anthracene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Fluoranthene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Fluorene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Naphthalene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Phenanthrene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Low Level Polynuclear Aromatics	
Pyrene Low Level	EPA 8310
	EPA 610
	EPA 8270D
	EPA 8270D SIM
Metals I	
Barium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Cadmium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Calcium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Chromium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Copper, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Iron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Metals I	
Lead, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Magnesium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Manganese, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Nickel, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Potassium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Silver, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Sodium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Strontium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Metals II	
Aluminum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Antimony, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Arsenic, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Beryllium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Chromium VI	EPA 7196A
	SM 3500-Cr B-2011
Mercury, Total	EPA 245.1, Rev. 3.0 (1994)
	EPA 7470A
Selenium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Vanadium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Metals II	
Zinc, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Metals III	
Cobalt, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Molybdenum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Thallium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Tin, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Titanium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Uranium (Mass)	EPA 200.8, Rev. 5.4 (1994)
Mineral	
Acidity	SM 2310B-2011
Alkalinity	SM 2320B-2011

Serial No.: 61627

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Mineral	
Chloride	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Fluoride, Total	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Hardness, Total	SM 2340C-2011
	EPA 200.7, Rev. 4.4 (1994)
	SM 2340B-2011
Sulfate (as SO4)	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Miscellaneous	
Boron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Bromide	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Color	SM 2120B-2011
Cyanide, Total	EPA 335.4, Rev. 1.0 (1993)
	EPA 9012B
Oil and Grease Total Recoverable (HEM)	EPA 1664A
	EPA 1664B
Organic Carbon, Total	SM 5310B-2011
	EPA 9060A
Perchlorate	EPA 6860
Phenols	EPA 420.1 (Rev. 1978)
	EPA 420.4, Rev. 1.0 (1993)
	EPA 9066
Miscellaneous	
Silica, Dissolved	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Specific Conductance	SM 2510B-2011
Sulfide (as S)	SM 4500-S2- F-2011
	EPA 9034
	SM 4500-S2- D-2011
Total Organic Halides	EPA 9020B
Turbidity	EPA 180.1, Rev. 2.0 (1993)
Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8270D
	EPA 8330B
1,3-Dinitrobenzene	EPA 8330B
1,4-Naphthoquinone	EPA 8270D
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 625.1
	EPA 8270D
	EPA 8330B
2,6-Dinitrotoluene	EPA 625.1
	EPA 8270D
	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3,5-Dinitroaniline	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Nitroaromatics and Isophorone	
4-Nitroquinoline-1-oxide	EPA 8270D
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Isophorone	EPA 625.1
	EPA 8270D
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 625.1
	EPA 8270D
	EPA 8330B
Nitroglycerine	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B
Pentaerythritol tetranitrate	EPA 8330B
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 625.1
	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 625.1
	EPA 8270D
N-Nitrosodiphenylamine	EPA 625.1
	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D

Nutrient	
Ammonia (as N)	EPA 350.1, Rev. 2.0 (1993)
Kjeldahl Nitrogen, Total	EPA 351.2, Rev. 2.0 (1993)
Nitrate (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Nitrite (as N)	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-NO2 B-2011
	EPA 9056A
Orthophosphate (as P)	EPA 365.1, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Phosphorus, Total	EPA 365.1, Rev. 2.0 (1993)
Organophosphate Pesticides	
Atrazine	EPA 8141B
Azinphos methyl	EPA 8141B
Chlorpyriphos	EPA 8141B
Demeton-O	EPA 8141B
Demeton-S	EPA 8141B
Diazinon	EPA 8141B
Dimethoate	EPA 8141B
	EPA 8270D
Disulfoton	EPA 8141B
	EPA 8270D
Famphur	EPA 8141B
	EPA 8270D

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Organophosphate Pesticides	
Malathion	EPA 8141B
Parathion ethyl	EPA 8141B
	EPA 8270D
Parathion methyl	EPA 8141B
	EPA 8270D
Phorate	EPA 8141B
	EPA 8270D
Simazine	EPA 8141B
Sulfotepp	EPA 8141B
Thionazin	EPA 8141B
	EPA 8270D
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015C
Gasoline Range Organics	EPA 8015C
Phthalate Esters	
Benzyl butyl phthalate	EPA 625.1
	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 625.1
	EPA 8270D
Diethyl phthalate	EPA 625.1
	EPA 8270D
Dimethyl phthalate	EPA 625.1
	EPA 8270D
Di-n-butyl phthalate	EPA 625.1
	EPA 8270D
Phthalate Esters	
Di-n-octyl phthalate	EPA 625.1
	EPA 8270D
Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
	EPA 608.3
Aroclor 1221 (PCB-1221)	EPA 8082A
	EPA 608.3
Aroclor 1232 (PCB-1232)	EPA 8082A
	EPA 608.3
Aroclor 1242 (PCB-1242)	EPA 8082A
	EPA 608.3
Aroclor 1248 (PCB-1248)	EPA 8082A
	EPA 608.3
Aroclor 1254 (PCB-1254)	EPA 8082A
	EPA 608.3
Aroclor 1260 (PCB-1260)	EPA 8082A
	EPA 608.3
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
Polynuclear Aromatics	
2-Acetylaminofluorene	EPA 8270D
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 625.1
	EPA 8270D

Serial No.: 61627

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Polynuclear Aromatics	
Acenaphthylene	EPA 625.1
	EPA 8270D
Anthracene	EPA 625.1
	EPA 8270D
Benzo(a)anthracene	EPA 625.1
	EPA 8270D
Benzo(a)pyrene	EPA 625.1
	EPA 8270D
Benzo(b)fluoranthene	EPA 625.1
	EPA 8270D
Benzo(g,h,i)perylene	EPA 625.1
	EPA 8270D
Benzo(k)fluoranthene	EPA 625.1
	EPA 8270D
Chrysene	EPA 625.1
	EPA 8270D
Dibenzo(a,h)anthracene	EPA 8270D
Fluoranthene	EPA 625.1
	EPA 8270D
Fluorene	EPA 625.1
	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 625.1
	EPA 8270D
Naphthalene	EPA 625.1
	EPA 8270D
Phenanthrene	EPA 625.1

Polynuclear Aromatics	
Phenanthrene	EPA 8270D
Pyrene	EPA 625.1
	EPA 8270D
Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 625.1
	EPA 8270D
2,4-Dichlorophenol	EPA 625.1
	EPA 8270D
2,4-Dimethylphenol	EPA 625.1
	EPA 8270D
2,4-Dinitrophenol	EPA 625.1
	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 625.1
	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 625.1
	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 625.1
	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 625.1
	EPA 8270D

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Priority Pollutant Phenols	
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 625.1
	EPA 8270D
Cresols, Total	EPA 8270D
Pentachlorophenol	EPA 625.1
	EPA 8270D
Phenol	EPA 625.1
	EPA 8270D
Residue	
Settleable Solids	SM 2540 F-2011
Solids, Total	SM 2540 B-2011
Solids, Total Dissolved	SM 2540 C-2011
Solids, Total Suspended	SM 2540 D-2011
Semi-Volatile Organics	
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
4-Amino biphenyl	EPA 8270D
Acetophenone	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Semi-Volatile Organics	
Methyl methanesulfonate	EPA 8270D
O,O,O-Triethyl phosphorothioate	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260C
1,2-Dichlorobenzene	EPA 8260C
	EPA 624.1
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 8260C
	EPA 624.1
1,4-Dichlorobenzene	EPA 8260C
	EPA 624.1
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 624.1
Chlorobenzene	EPA 624.1
Ethyl benzene	EPA 8260C
	EPA 624.1
Isopropylbenzene	EPA 8260C
m/p-Xylenes	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Volatile Aromatics	
o-Xylene	EPA 8260C
p-Isopropyltoluene (P-Cymene)	EPA 8260C
sec-Butylbenzene	EPA 8260C
tert-Butylbenzene	EPA 8260C
Toluene	EPA 8260C
	EPA 624.1
Total Xylenes	EPA 8260C
	EPA 624.1
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
	EPA 624.1
1,1,2,2-Tetrachloroethane	EPA 8260C
	EPA 624.1
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
	EPA 624.1
1,1-Dichloroethane	EPA 8260C
	EPA 624.1
1,1-Dichloroethene	EPA 8260C
	EPA 624.1
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C

Volatile Halocarbons	
1,2-Dichloroethane	EPA 8260C
	EPA 624.1
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
2-Chloroethylvinyl ether	EPA 8260C
	EPA 624.1
3-Chloropropene (Allyl chloride)	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
	EPA 624.1
Bromoform	EPA 8260C
	EPA 624.1
Bromomethane	EPA 8260C
	EPA 624.1
Carbon tetrachloride	EPA 8260C
	EPA 624.1
Chloroethane	EPA 8260C
	EPA 624.1
Chloroform	EPA 8260C
	EPA 624.1
Chloromethane	EPA 8260C
	EPA 624.1
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C

Serial No.: 61627

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Volatile Halocarbons

cis-1,3-Dichloropropene	EPA 624.1
Dibromochloromethane	EPA 8260C
	EPA 624.1
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methylene chloride	EPA 8260C
	EPA 624.1
Tetrachloroethene	EPA 8260C
	EPA 624.1
trans-1,2-Dichloroethene	EPA 8260C
	EPA 624.1
trans-1,3-Dichloropropene	EPA 8260C
	EPA 624.1
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
	EPA 624.1
Trichlorofluoromethane	EPA 8260C
	EPA 624.1
Vinyl chloride	EPA 8260C
	EPA 624.1

Volatiles Organics

1,4-Dioxane	EPA 8260C
	EPA 8260C SIM
2-Butanone (Methylethyl ketone)	EPA 8260C

Volatiles Organics

2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
	EPA 624.1
Acetone	EPA 8260C
	EPA 624.1
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C
Di-ethyl ether	EPA 8260C
Ethyl Acetate	EPA 8260C
Isobutyl alcohol	EPA 8260C
n-Butanol	EPA 8260C
Vinyl acetate	EPA 8260C
	EPA 624.1

Sample Preparation Methods

EPA 5030C
EPA 3010A
EPA 3005A
EPA 3510C
EPA 3520C
EPA 3020A
EPA 3535A

Serial No.: 61627

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1,2-Diphenylhydrazine	EPA 8270D
1,4-Phenylenediamine	EPA 8270D
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Methapyrilene	EPA 8270D
Pronamide	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 8270D
3,3'-Dimethylbenzidine	EPA 8270D
Benzidine	EPA 8270D

Carbamate Pesticides	
Aldicarb	EPA 8321B
Carbofuran	EPA 8321B
Characteristic Testing	
Ignitability	EPA 1010A
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311
Chlorinated Hydrocarbon Pesticides	
2,4'-DDD (Mitotane)	EPA 8081B
4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
Chlorobenzilate	EPA 8081B
delta-BHC	EPA 8081B
Diallate	EPA 8081B
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B

Serial No.: 61628

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Chlorinated Hydrocarbon Pesticides	
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Isodrin	EPA 8270D
Kepone	EPA 8081B
Methoxychlor	EPA 8081B
Pentachloronitrobenzene	EPA 8270D
Simazine	EPA 8141B
Toxaphene	EPA 8081B
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 8270D
1-Chloronaphthalene	EPA 8270D
2-Chloronaphthalene	EPA 8270D
Hexachlorobenzene	EPA 8270D
Hexachlorobutadiene	EPA 8270D
Hexachlorocyclopentadiene	EPA 8270D
Hexachloroethane	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
	EPA 8321B

Chlorophenoxy Acid Pesticides	
2,4,5-TP (Silvex)	EPA 8151A
	EPA 8321B
2,4-D	EPA 8151A
	EPA 8321B
2,4-DB	EPA 8151A
	EPA 8321B
Dalapon	EPA 8151A
	EPA 8321B
Dicamba	EPA 8151A
	EPA 8321B
Dichloroprop	EPA 8151A
	EPA 8321B
Dinoseb	EPA 8321B
MCPA	EPA 8151A
	EPA 8321B
MCPP	EPA 8151A
	EPA 8321B
Haloethers	
2,2'-Oxybis(1-chloropropane)	EPA 8270D
4-Bromophenylphenyl ether	EPA 8270D
4-Chlorophenylphenyl ether	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 8270D
Low Level Polynuclear Aromatic Hydrocarbons	
Acenaphthene Low Level	EPA 8310

Serial No.: 61628

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene Low Level	EPA 8270D
Acenaphthylene Low Level	EPA 8310
	EPA 8270D
Anthracene Low Level	EPA 8310
	EPA 8270D
Benzo(a)anthracene Low Level	EPA 8310
	EPA 8270D
Benzo(a)pyrene Low Level	EPA 8310
	EPA 8270D
Benzo(b)fluoranthene Low Level	EPA 8310
	EPA 8270D
Benzo(g,h,i)perylene Low Level	EPA 8310
	EPA 8270D
Benzo(k)fluoranthene Low Level	EPA 8310
	EPA 8270D
Chrysene Low Level	EPA 8310
	EPA 8270D
Dibenzo(a,h)anthracene Low Level	EPA 8310
	EPA 8270D
Fluoranthene Low Level	EPA 8310
	EPA 8270D
Fluorene Low Level	EPA 8310
	EPA 8270D
Indeno(1,2,3-cd)pyrene Low Level	EPA 8310
	EPA 8270D
Naphthalene Low Level	EPA 8310

Low Level Polynuclear Aromatic Hydrocarbons

Naphthalene Low Level	EPA 8270D
Phenanthrene Low Level	EPA 8310
	EPA 8270D
Pyrene Low Level	EPA 8310
	EPA 8270D

Metals I

Barium, Total	EPA 6010C
	EPA 6020A
Cadmium, Total	EPA 6010C
	EPA 6020A
Calcium, Total	EPA 6010C
Chromium, Total	EPA 6010C
	EPA 6020A
Copper, Total	EPA 6010C
	EPA 6020A
Iron, Total	EPA 6010C
Lead, Total	EPA 6010C
	EPA 6020A
Magnesium, Total	EPA 6010C
Manganese, Total	EPA 6010C
	EPA 6020A
Nickel, Total	EPA 6010C
	EPA 6020A
Potassium, Total	EPA 6010C
Silver, Total	EPA 6010C

Serial No.: 61628

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Metals I	
Silver, Total	EPA 6020A
Sodium, Total	EPA 6010C
Strontium, Total	EPA 6010C
Metals II	
Aluminum, Total	EPA 6010C
Antimony, Total	EPA 6010C
	EPA 6020A
Arsenic, Total	EPA 6010C
	EPA 6020A
Beryllium, Total	EPA 6010C
	EPA 6020A
Chromium VI	EPA 7196A
Lithium, Total	EPA 6010C
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010C
	EPA 6020A
Vanadium, Total	EPA 6010C
	EPA 6020A
Zinc, Total	EPA 6010C
	EPA 6020A
Metals III	
Cobalt, Total	EPA 6010C
	EPA 6020A
Molybdenum, Total	EPA 6010C
	EPA 6020A
Metals III	
Silica, Dissolved	EPA 6010C
Thallium, Total	EPA 6010C
	EPA 6020A
Tin, Total	EPA 6010C
Titanium, Total	EPA 6010C
Minerals	
Bromide	EPA 9056A
Fluoride, Total	EPA 9056A
Sulfate (as SO4)	EPA 9056A
Miscellaneous	
Boron, Total	EPA 6010C
Cyanide, Total	EPA 9012B
Perchlorate	EPA 6860
Sulfide (as S)	EPA 9034
Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8270D
	EPA 8330B
1,3-Dinitrobenzene	EPA 8270D
	EPA 8330B
1,4-Dinitrobenzene	EPA 8270D
1,4-Naphthoquinone	EPA 8270D
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 8270D
	EPA 8330B

Serial No.: 61628

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Nitroaromatics and Isophorone	
2,6-Dinitrotoluene	EPA 8270D
	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Nitroquinoline-1-oxide	EPA 8270D
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Isophorone	EPA 8270D
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 8270D
	EPA 8330B
Nitroglycerine	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B
Pentaerythritol tetranitrate	EPA 8330B
Pyridine	EPA 8270D
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 8270D
N-Nitrosodiphenylamine	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
Nitrosoamines	
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D
Nutrients	
Nitrate (as N)	EPA 9056A
Nitrite (as N)	EPA 9056A
Orthophosphate (as P)	EPA 9056A
Organophosphate Pesticides	
Azinphos ethyl	EPA 8141B
Azinphos methyl	EPA 8141B
Bolstar	EPA 8141B
Carbophenothion	EPA 8141B
Chlorpyriphos	EPA 8141B
Coumaphos	EPA 8141B
Demeton-O	EPA 8141B
Demeton-S	EPA 8141B
Diazinon	EPA 8141B
Dimethoate	EPA 8141B
	EPA 8270D
Disulfoton	EPA 8141B
	EPA 8270D
EPN	EPA 8141B
Ethoprop	EPA 8141B
Famphur	EPA 8141B
	EPA 8270D
Fensulfothion	EPA 8141B

Serial No.: 61628

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Organophosphate Pesticides	
Fenthion	EPA 8141B
Malathion	EPA 8141B
Mevinphos	EPA 8141B
NALED	EPA 8141B
Parathion ethyl	EPA 8141B
	EPA 8270D
Parathion methyl	EPA 8141B
	EPA 8270D
Phorate	EPA 8141B
	EPA 8270D
Ronnel	EPA 8141B
Sulfotepp	EPA 8141B
	EPA 8270D
Thionazin	EPA 8141B
	EPA 8270D
Tokuthion	EPA 8141B
Trichloronate	EPA 8141B
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015C
Gasoline Range Organics	EPA 8015C
Oil and Grease Total Recoverable (HEM)	EPA 9071B (Solvent:Hexane)
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 8270D
Diethyl phthalate	EPA 8270D
Phthalate Esters	
Dimethyl phthalate	EPA 8270D
Di-n-butyl phthalate	EPA 8270D
Di-n-octyl phthalate	EPA 8270D
Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
Polynuclear Aromatic Hydrocarbons	
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 8270D
Acenaphthylene	EPA 8270D
Anthracene	EPA 8270D
Benzo(a)anthracene	EPA 8270D
Benzo(a)pyrene	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D
Benzo(g,h,i)perylene	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 8270D

Serial No.: 61628

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Polynuclear Aromatic Hydrocarbons	
Dibenzo(a,h)anthracene	EPA 8270D
Dibenzo(a,j)acridine	EPA 8270D
Fluoranthene	EPA 8270D
Fluorene	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D
Naphthalene	EPA 8270D
Phenanthrene	EPA 8270D
Pyrene	EPA 8270D
Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 8270D
2,4-Dichlorophenol	EPA 8270D
2,4-Dimethylphenol	EPA 8270D
2,4-Dinitrophenol	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 8270D
Pentachlorophenol	EPA 8270D
Priority Pollutant Phenols	
Phenol	EPA 8270D
Semi-Volatile Organics	
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
4-Amino biphenyl	EPA 8270D
Acetophenone	EPA 8270D
Aramite	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Methyl methanesulfonate	EPA 8270D
O,O,O-Triethyl phosphorothioate	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260C
1,2-Dichlorobenzene	EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 8260C
1,4-Dichlorobenzene	EPA 8260C

Serial No.: 61628

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Volatile Aromatics

2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 8260C
Bromobenzene	EPA 8260C
Chlorobenzene	EPA 8260C
Ethyl benzene	EPA 8260C
Isopropylbenzene	EPA 8260C
m/p-Xylenes	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C
o-Xylene	EPA 8260C
p-Isopropyltoluene (P-Cymene)	EPA 8260C
sec-Butylbenzene	EPA 8260C
Styrene	EPA 8260C
tert-Butylbenzene	EPA 8260C
Toluene	EPA 8260C
Total Xylenes	EPA 8260C

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 8260C
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
1,1-Dichloroethane	EPA 8260C

Volatile Halocarbons

1,1-Dichloroethene	EPA 8260C
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloroethane	EPA 8260C
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
2-Chloroethylvinyl ether	EPA 8260C
3-Chloropropene (Allyl chloride)	EPA 8260C
Bromodichloromethane	EPA 8260C
Bromoform	EPA 8260C
Bromomethane	EPA 8260C
Carbon tetrachloride	EPA 8260C
Chloroethane	EPA 8260C
Chloroform	EPA 8260C
Chloromethane	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
Dibromochloromethane	EPA 8260C
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methylene chloride	EPA 8260C

Serial No.: 61628

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. SCOTT HALL
EUROFINS TESTAMERICA INC. - DENVER
4955 YARROW STREET
ARVADA, CO 80002

NY Lab Id No: 11964

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Volatile Halocarbons	
Tetrachloroethene	EPA 8260C
trans-1,2-Dichloroethene	EPA 8260C
trans-1,3-Dichloropropene	EPA 8260C
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
Trichlorofluoromethane	EPA 8260C
Vinyl chloride	EPA 8260C
Volatile Organics	
1,4-Dioxane	EPA 8260C
	EPA 8260C SIM
2-Butanone (Methylethyl ketone)	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C
Di-ethyl ether	EPA 8260C
Ethyl Acetate	EPA 8260C
Isobutyl alcohol	EPA 8260C
Methyl tert-butyl ether	EPA 8260C
Propionitrile	EPA 8260C
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 5035A-L
	EPA 5035A-H
	EPA 9030B

Sample Preparation Methods
EPA 3010A
EPA 3005A
EPA 3050B
EPA 3550C
EPA 3020A
EPA 3546

Serial No.: 61628

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

Eurofins Test America Certifications

Savannah

CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Eurofins TestAmerica Savannah

5102 LaRoche Avenue
Savannah, GA 31404

Fulfills the requirements of

ISO/IEC 17025:2017

and the

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 22 September 2024
Certificate Number: L2463

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 AND U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.3)

Eurofins TestAmerica Savannah
5102 LaRoche Avenue
Savannah, GA 31404
Kim Chamberlain
912-354-7858

TESTING

Valid to: **September 22, 2024** Certificate Number: **L2463**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
General Chemistry	EPA 1664A	Oil and Grease
General Chemistry	EPA 1664A	Total Petroleum Hydrocarbons
General Chemistry	EPA 1664B	Oil and Grease
General Chemistry	EPA 1664B	Total Petroleum Hydrocarbons
ICP	EPA 6010C	Aluminum
ICP	EPA 6010C	Antimony
ICP	EPA 6010C	Arsenic
ICP	EPA 6010C	Barium
ICP	EPA 6010C	Beryllium
ICP	EPA 6010C	Boron
ICP	EPA 6010C	Cadmium
ICP	EPA 6010C	Calcium
ICP	EPA 6010C	Chromium
ICP	EPA 6010C	Cobalt
ICP	EPA 6010C	Copper

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 6010C	Iron
ICP	EPA 6010C	Lead
ICP	EPA 6010C	Magnesium
ICP	EPA 6010C	Manganese
ICP	EPA 6010C	Molybdenum
ICP	EPA 6010C	Nickel
ICP	EPA 6010C	Potassium
ICP	EPA 6010C	Selenium
ICP	EPA 6010C	Silica
ICP	EPA 6010C	Silicon
ICP	EPA 6010C	Silver
ICP	EPA 6010C	Sodium
ICP	EPA 6010C	Strontium
ICP	EPA 6010C	Thallium
ICP	EPA 6010C	Tin
ICP	EPA 6010C	Titanium
ICP	EPA 6010C	Vanadium
ICP	EPA 6010C	Zinc
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium
ICP	EPA 6010D	Boron
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Copper
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead
ICP	EPA 6010D	Magnesium
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium
ICP	EPA 6010D	Silica
ICP	EPA 6010D	Silicon
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Strontium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Titanium
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Mercury
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Boron
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Mercury
ICP/MS	EPA 6020B	Molybdenum
ICP/MS	EPA 6020B	Nickel
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Strontium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Tin
ICP/MS	EPA 6020B	Titanium
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
Colorimetry	EPA 7196A	Chromium 3+
Colorimetry	EPA 7196A	Chromium 6+
CVAA	EPA 7470A	Mercury
GC/ECD	EPA 8011	1,2,3-Trichloropropane
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/FID	EPA 8015C	#2 Diesel Fuel
GC/FID	EPA 8015C	Diesel Range Organics
GC/FID	EPA 8015C	Gasoline Range Organics

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C	Kerosene
GC/FID	EPA 8015C	Mineral Spirits
GC/FID	EPA 8015C	Motor Oil
GC/FID	EPA 8015C	Oil Range Organics
GC/FID	EPA 8015C-DAI	2,2'-Oxybisethanol
GC/FID	EPA 8015C-DAI	2-Butoxyethanol
GC/FID	EPA 8015C-DAI	2-Propoxy ethanol
GC/FID	EPA 8015C-DAI	Cellosolve acetate
GC/FID	EPA 8015C-DAI	Di-propylene glycol
GC/FID	EPA 8015C-DAI	Di-propylene glycol methyl ether
GC/FID	EPA 8015C-DAI	Ethanol
GC/FID	EPA 8015C-DAI	Ethyl acetate
GC/FID	EPA 8015C-DAI	Ethylene glycol
GC/FID	EPA 8015C-DAI	Isoamyl acetate
GC/FID	EPA 8015C-DAI	Isobutanol
GC/FID	EPA 8015C-DAI	Isobutyl acetate
GC/FID	EPA 8015C-DAI	Isopropanol
GC/FID	EPA 8015C-DAI	Isopropyl acetate
GC/FID	EPA 8015C-DAI	Methanol
GC/FID	EPA 8015C-DAI	Methyl acetate
GC/FID	EPA 8015C-DAI	n-Butanol
GC/FID	EPA 8015C-DAI	n-Butyl acetate
GC/FID	EPA 8015C-DAI	n-Heptanol
GC/FID	EPA 8015C-DAI	n-Propanol
GC/FID	EPA 8015C-DAI	n-Propyl acetate
GC/FID	EPA 8015C-DAI	Phenol
GC/FID	EPA 8015C-DAI	Propylene glycol
GC/FID	EPA 8015C-DAI	sec-Butanol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C-DAI	sec-Butyl acetate
GC/FID	EPA 8015C-DAI	Tert-amyl alcohol
GC/FID	EPA 8015C-DAI	tert-Butyl alcohol
GC/FID	EPA 8015C-DAI	Tetraethylene glycol
GC/FID	EPA 8015C-DAI	Triethylene glycol
GC/ECD	EPA 8081B	2,4’ DDE
GC/ECD	EPA 8081B	2,4’-DDD
GC/ECD	EPA 8081B	2,4’-DDT
GC/ECD	EPA 8081B	4,4’ DDE
GC/ECD	EPA 8081B	4,4’-DDD
GC/ECD	EPA 8081B	4,4’-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC
GC/ECD	EPA 8081B	beta-BHC
GC/ECD	EPA 8081B	Chlordane (alpha)
GC/ECD	EPA 8081B	Chlordane (gamma)
GC/ECD	EPA 8081B	Chlordane (technical)
GC/ECD	EPA 8081B	Chlorobenzilate
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I (alpha)
GC/ECD	EPA 8081B	Endosulfan II (beta)
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	gamma-BHC
GC/ECD	EPA 8081B	Heptachlor

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Isodrin
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Toxaphene
GC/ECD	EPA 8082A	PCB-1016
GC/ECD	EPA 8082A	PCB-1221
GC/ECD	EPA 8082A	PCB-1232
GC/ECD	EPA 8082A	PCB-1242
GC/ECD	EPA 8082A	PCB-1248
GC/ECD	EPA 8082A	PCB-1254
GC/ECD	EPA 8082A	PCB-1260
GC/ECD	EPA 8082A	PCB-1262
GC/ECD	EPA 8082A	PCB-1268
GC/ECD	EPA 8082A	PCBs, Total
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4,6-Trichlorophenol
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	2,6-Dichlorophenol
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	DCPA (Dacthal)
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Picloram
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethene
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane
GC/MS	EPA 8260B	1,2-Dichloroethene, Total
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,3-Dichloropropene, Total
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	2-Butanone
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone
GC/MS	EPA 8260B	3-Chloro-1-propene
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Isopropyltoluene
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonitrile
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	BTEX, Total
GC/MS	EPA 8260B	Carbon disulfide
GC/MS	EPA 8260B	Carbon tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane
GC/MS	EPA 8260B	Diethyl ether
GC/MS	EPA 8260B	Ethanol
GC/MS	EPA 8260B	Ethyl benzene
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Furan
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutanol
GC/MS	EPA 8260B	Isopropyl ether
GC/MS	EPA 8260B	Isopropylbenzene
GC/MS	EPA 8260B	m & p-Xylene
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl acetate
GC/MS	EPA 8260B	Methyl cyclohexane
GC/MS	EPA 8260B	Methyl isobutyl ketone
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260B	Methylene chloride
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Heptane
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	o-Xylene
GC/MS	EPA 8260B	Pentachloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Propionitrile
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene
GC/MS	EPA 8260B	Trichlorofluoromethane
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl chloride
GC/MS	EPA 8260B	Xylenes, total
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethene, Total
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropene, Total
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	3-Chloro-1-propene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	4-Isopropyltoluene
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	BTEX, Total
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Chloroprene
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethanol
GC/MS	EPA 8260C	Ethyl benzene
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Furan
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	Hexane
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropyl ether
GC/MS	EPA 8260C	Isopropylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	m & p-Xylene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl cyclohexane
GC/MS	EPA 8260C	Methyl isobutyl ketone
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	n-Heptane
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	Vinyl acetate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	Xylenes, total
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 8260D	1,2-Dichloroethene, Total
GC/MS	EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,3-Dichloropropene, Total
GC/MS	EPA 8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260D	1,4-Dioxane
GC/MS	EPA 8260D	1-Chlorohexane
GC/MS	EPA 8260D	2,2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	2-Butanone
GC/MS	EPA 8260D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone
GC/MS	EPA 8260D	3-Chloro-1-propene
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Isopropyltoluene
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	Bromomethane
GC/MS	EPA 8260D	BTEX, Total
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloromethane
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	Cyclohexane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Dibromochloromethane
GC/MS	EPA 8260D	Dibromomethane
GC/MS	EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diethyl ether
GC/MS	EPA 8260D	Ethanol
GC/MS	EPA 8260D	Ethyl benzene
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Furan
GC/MS	EPA 8260D	Hexachlorobutadiene
GC/MS	EPA 8260D	Hexane
GC/MS	EPA 8260D	Iodomethane
GC/MS	EPA 8260D	Isobutanol
GC/MS	EPA 8260D	Isopropyl ether
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	m & p-Xylene
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl acetate
GC/MS	EPA 8260D	Methyl cyclohexane
GC/MS	EPA 8260D	Methyl isobutyl ketone
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	n-Heptane
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	o-Xylene
GC/MS	EPA 8260D	Pentachloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Propionitrile
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	Styrene
GC/MS	EPA 8260D	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260D	tert-Butylbenzene
GC/MS	EPA 8260D	Tetrachloroethene
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260D	Trichloroethene
GC/MS	EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride
GC/MS	EPA 8260D	Xylenes, total
GC/MS	EPA 8270D	1,1-Biphenyl
GC/MS	EPA 8270D	1,2,3-Trichlorobenzene
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,2-Diphenylhydrazine
GC/MS	EPA 8270D	1,3,5-Trichlorobenzene
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dioxane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Methylnaphthalene
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	2,3,6-Trichlorophenol
GC/MS	EPA 8270D	2,3-Dimethylphenol
GC/MS	EPA 8270D	2,3-Xylenol
GC/MS	EPA 8270D	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,5-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	2-Picoline

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270D	3 & 4-Methylphenol
GC/MS	EPA 8270D	3,3-Dichlorobenzidine
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	3,4-Dimethylphenol
GC/MS	EPA 8270D	3,4-Xylenol
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	4-Bromophenylphenyl ether
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	4-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	alpha-, alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	alpha-Pinene
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite, Total
GC/MS	EPA 8270D	Atrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo (a) anthracene
GC/MS	EPA 8270D	Benzo (a) pyrene
GC/MS	EPA 8270D	Benzo (b) fluoranthene
GC/MS	EPA 8270D	Benzo (ghi) perylene
GC/MS	EPA 8270D	Benzo (k) fluoranthene
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270D	Bis (2-chloroethyl) ether
GC/MS	EPA 8270D	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270D	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols
GC/MS	EPA 8270D	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h) anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	Diphenyl ether

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methane sulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Hexachlrobutadiene
GC/MS	EPA 8270D	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	Methyl methane sulfonate
GC/MS	EPA 8270D	Methylbenzoate
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	o,o',o''-Triethylphosphorothioate
GC/MS	EPA 8270D	Parathion ethyl
GC/MS	EPA 8270D	Parathion methyl
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Pentachlronitrobenzene
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	Phenyl ether
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole, Total
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270D	2,4-Dinitrochlorobenzene
GC/MS	EPA 8270E	1,1-Biphenyl
GC/MS	EPA 8270E	1,2,3-Trichlorobenzene
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270E	1,2-Dichlorobenzene
GC/MS	EPA 8270E	1,2-Diphenylhydrazine
GC/MS	EPA 8270E	1,3,5-Trichlorobenzene
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	1,3-Dichlorobenzene
GC/MS	EPA 8270E	1,3-Dinitrobenzene
GC/MS	EPA 8270E	1,4-Dichlorobenzene
GC/MS	EPA 8270E	1,4-Dioxane
GC/MS	EPA 8270E	1,4-Naphthoquinone
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,3,6-Trichlorophenol
GC/MS	EPA 8270E	2,3-Dimethylphenol
GC/MS	EPA 8270E	2,3-Xylenol
GC/MS	EPA 8270E	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene
GC/MS	EPA 8270E	2,5-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	2-Picoline
GC/MS	EPA 8270E	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270E	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270E	3 & 4-Methylphenol
GC/MS	EPA 8270E	3,3-Dichlorobenzidine
GC/MS	EPA 8270E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270E	3,4-Dimethylphenol
GC/MS	EPA 8270E	3,4-Xylenol
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	4-Bromophenylphenyl ether
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenol
GC/MS	EPA 8270E	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270E	4-Nitroaniline
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270E	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	alpha-, alpha-Dimethylphenethylamine
GC/MS	EPA 8270E	alpha-Pinene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Aniline
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Aramite, Total
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Benzaldehyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E	Benzo (a) anthracene
GC/MS	EPA 8270E	Benzo (a) pyrene
GC/MS	EPA 8270E	Benzo (b) fluoranthene
GC/MS	EPA 8270E	Benzo (ghi) perylene
GC/MS	EPA 8270E	Benzo (k) fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270E	Bis (2-chloroethyl) ether
GC/MS	EPA 8270E	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270E	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270E	Butyl benzyl phthalate
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	Cresols
GC/MS	EPA 8270E	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 8270E	Dibenz(a,h) anthracene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethyl phthalate
GC/MS	EPA 8270E	Dimethoate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Dimethyl phthalate
GC/MS	EPA 8270E	Di-n-butyl phthalate
GC/MS	EPA 8270E	Di-n-octyl phthalate
GC/MS	EPA 8270E	Diphenyl ether
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Ethyl methane sulfonate
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachlorophene
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 8270E	Hexachlrobutadiene
GC/MS	EPA 8270E	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Methapyrilene
GC/MS	EPA 8270E	Methyl methane sulfonate
GC/MS	EPA 8270E	Methylbenzoate
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	N-Nitrosodiethylamine
GC/MS	EPA 8270E	N-Nitrosodimethylamine
GC/MS	EPA 8270E	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270E	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270E	N-Nitrosodiphenylamine
GC/MS	EPA 8270E	N-Nitrosomethylethylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	N-Nitrosomorpholine
GC/MS	EPA 8270E	N-Nitrosopiperidine
GC/MS	EPA 8270E	N-Nitrosopyrrolidine
GC/MS	EPA 8270E	o,o',o''-Triethylphosphorothioate
GC/MS	EPA 8270E	Parathion ethyl
GC/MS	EPA 8270E	Parathion methyl
GC/MS	EPA 8270E	p-Dimethylaminoazobenzene
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachlorophenol
GC/MS	EPA 8270E	Pentachlronitrobenzene
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Phenyl ether
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	p-Phenylene diamine
GC/MS	EPA 8270E	Pronamide
GC/MS	EPA 8270E	Pyrene
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	Safrole, Total
GC/MS	EPA 8270E	Sulfotepp
GC/MS	EPA 8270E	Thionazin
GC/MS	EPA 8270E	2,4-Dinitrochlorobenzene
General Chemistry	EPA 9012B	Cyanide
General Chemistry	EPA 9013 EPA 9012B	Cyanide amenable to chlorination
General Chemistry	EPA 9020B	Total organic halides
General Chemistry	EPA 9030B EPA 9034	Sulfide

Non-Potable Water		
Technology	**Method**	**Analyte**
General Chemistry	EPA 9038	Sulfate
General Chemistry	EPA 9040C	pH
General Chemistry	EPA 9050A	Specific conductance
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate (as N)
IC	EPA 9056A	Nitrate (as NO3)
IC	EPA 9056A	Nitrate-nitrite (as N)
IC	EPA 9056A	Nitrate-nitrite (as NO3-NO2)
IC	EPA 9056A	Nitrite (as N)
IC	EPA 9056A	Nitrite (as NO2)
IC	EPA 9056A	Sulfate
General Chemistry	EPA 9060A	Dissolved carbon
General Chemistry	EPA 9060A	Dissolved inorganic carbon
General Chemistry	EPA 9060A	Dissolved organic carbon
General Chemistry	EPA 9060A	Total carbon
General Chemistry	EPA 9060A	Total inorganic carbon
General Chemistry	EPA 9060A	Total organic carbon
General Chemistry	EPA 9065A	Phenols
General Chemistry	EPA 9251	Chloride
GC/FID/TCD	RSK-175	Ethane (FID)
GC/FID/TCD	RSK-175	Ethene (FID)
GC/FID/TCD	RSK-175	Methane (FID)
GC/FID/TCD	RSK-175	Methane (TCD)
Preparation	**Method**	**Type**
Organic Extraction	EPA 3520C	Continuous Liquid-Liquid Extraction
TLCP Preparation	EPA 1311	Toxicity Characteristics Leaching Procedure

Non-Potable Water		
Technology	**Method**	**Analyte**
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Purge & Trap	EPA 5030B	Purge & Trap for Aqueous volatile
Acid Digestion	EPA 3005A	Metals Prep
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Distillation	EPA 9030B	Sulfide

Drinking Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 504.1	1,2,3-Trichloropropane
GC/ECD	EPA 504.1	1,2-Dibromo-3-chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2-Dibromoethane (EDB)
GC/MS	EPA 524.2	1,1,1,2-Tetrachloroethane
GC/MS	EPA 524.2	1,1,1-Trichloroethane
GC/MS	EPA 524.2	1,1,2,2-Tetrachloroethane
GC/MS	EPA 524.2	1,1,2-Trichloroethane
GC/MS	EPA 524.2	1,1-Dichloroethane
GC/MS	EPA 524.2	1,1-Dichloroethylene
GC/MS	EPA 524.2	1,1-Dichloropropene
GC/MS	EPA 524.2	1,2,3-Trichlorobenzene
GC/MS	EPA 524.2	1,2,3-Trichloropropane
GC/MS	EPA 524.2	1,2,4-Trichlorobenzene
GC/MS	EPA 524.2	1,2,4-Trimethylbenzene
GC/MS	EPA 524.2	1,2-Dichlorobenzene
GC/MS	EPA 524.2	1,2-Dichloroethane
GC/MS	EPA 524.2	1,2-Dichloropropane
GC/MS	EPA 524.2	1,3,5-Trimethylbenzene
GC/MS	EPA 524.2	1,3-Dichlorobenzene

Drinking Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 524.2	1,3-Dichloropropane
GC/MS	EPA 524.2	1,4-Dichlorobenzene
GC/MS	EPA 524.2	2,2-Dichloropropane
GC/MS	EPA 524.2	2-Butanone (Methyl ethyl ketone, MEK)
GC/MS	EPA 524.2	2-Chlorotoluene
GC/MS	EPA 524.2	2-Hexanone
GC/MS	EPA 524.2	4-Chlorotoluene
GC/MS	EPA 524.2	4-Isopropyltoluene
GC/MS	EPA 524.2	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 524.2	Acetone
GC/MS	EPA 524.2	Benzene
GC/MS	EPA 524.2	Bromobenzene
GC/MS	EPA 524.2	Bromochloromethane
GC/MS	EPA 524.2	Bromodichloromethane
GC/MS	EPA 524.2	Bromoform
GC/MS	EPA 524.2	Carbon tetrachloride
GC/MS	EPA 524.2	Chlorobenzene
GC/MS	EPA 524.2	Chloroethane
GC/MS	EPA 524.2	Chloroform
GC/MS	EPA 524.2	cis-1,2-Dichloroethylene
GC/MS	EPA 524.2	cis-1,3-Dichloropropene
GC/MS	EPA 524.2	Dibromochloromethane
GC/MS	EPA 524.2	Dibromomethane
GC/MS	EPA 524.2	Dichlorodifluoromethane
GC/MS	EPA 524.2	Dichloromethane (DCM, Methylene chloride)
GC/MS	EPA 524.2	Ethylbenzene
GC/MS	EPA 524.2	Hexachlorobutadiene
GC/MS	EPA 524.2	Isopropylbenzene

Drinking Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 524.2	m+p-Xylenes
GC/MS	EPA 524.2	Methyl bromide (Bromomethane)
GC/MS	EPA 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 524.2	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 524.2	Naphthalene
GC/MS	EPA 524.2	n-Butylbenzene
GC/MS	EPA 524.2	n-Propylbenzene
GC/MS	EPA 524.2	o-Xylene
GC/MS	EPA 524.2	sec-Butylbenzene
GC/MS	EPA 524.2	Styrene
GC/MS	EPA 524.2	tert-Butylbenzene
GC/MS	EPA 524.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 524.2	Toluene
GC/MS	EPA 524.2	Total trihalomethanes
GC/MS	EPA 524.2	trans-1,2-Dichloroethylene
GC/MS	EPA 524.2	trans-1,3-Dichloropropene
GC/MS	EPA 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 524.2	Trichlorofluoromethane
GC/MS	EPA 524.2	Vinyl chloride
GC/MS	EPA 524.2	Xylene (total)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
General Chemistry	EPA 1030	Ignitability
ICP	EPA 6010C	Aluminum
ICP	EPA 6010C	Antimony
ICP	EPA 6010C	Arsenic
ICP	EPA 6010C	Barium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010C	Beryllium
ICP	EPA 6010C	Boron
ICP	EPA 6010C	Cadmium
ICP	EPA 6010C	Calcium
ICP	EPA 6010C	Chromium
ICP	EPA 6010C	Cobalt
ICP	EPA 6010C	Copper
ICP	EPA 6010C	Iron
ICP	EPA 6010C	Lead
ICP	EPA 6010C	Magnesium
ICP	EPA 6010C	Manganese
ICP	EPA 6010C	Molybdenum
ICP	EPA 6010C	Nickel
ICP	EPA 6010C	Potassium
ICP	EPA 6010C	Selenium
ICP	EPA 6010C	Silver
ICP	EPA 6010C	Sodium
ICP	EPA 6010C	Strontium
ICP	EPA 6010C	Thallium
ICP	EPA 6010C	Tin
ICP	EPA 6010C	Titanium
ICP	EPA 6010C	Vanadium
ICP	EPA 6010C	Zinc
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Boron
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt
ICP	EPA 6010D	Copper
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead
ICP	EPA 6010D	Magnesium
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Strontium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Titanium
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Mercury
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Boron

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Mercury
ICP/MS	EPA 6020B	Molybdenum
ICP/MS	EPA 6020B	Nickel
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Strontium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Tin
ICP/MS	EPA 6020B	Titanium
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
CVAA	EPA 7471B	Mercury
GC/FID	EPA 8015C	#2 Diesel Fuel
GC/FID	EPA 8015C	Diesel Range Organics
GC/FID	EPA 8015C	Gasoline Range Organics
GC/FID	EPA 8015C	Kerosene
GC/FID	EPA 8015C	Mineral Spirits

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C	Motor Oil
GC/FID	EPA 8015C	Oil Range Organics
GC/FID	EPA 8015C	2,2'-Oxybisethanol
GC/FID	EPA 8015C	2-Butoxyethanol
GC/FID	EPA 8015C	2-Propoxy ethanol
GC/FID	EPA 8015C	Cellosolve acetate
GC/FID	EPA 8015C	Di-propylene glycol
GC/FID	EPA 8015C	Di-propylene glycol methyl ether
GC/FID	EPA 8015C	Ethanol
GC/FID	EPA 8015C	Ethanol
GC/FID	EPA 8015C	Ethyl acetate
GC/FID	EPA 8015C	Ethylene glycol
GC/FID	EPA 8015C	Isoamyl acetate
GC/FID	EPA 8015C	Isobutanol
GC/FID	EPA 8015C	Isobutyl acetate
GC/FID	EPA 8015C	Isopropanol
GC/FID	EPA 8015C	Isopropyl acetate
GC/FID	EPA 8015C	Methanol
GC/FID	EPA 8015C	Methyl acetate
GC/FID	EPA 8015C	n-Butanol
GC/FID	EPA 8015C	n-Butyl acetate
GC/FID	EPA 8015C	n-Heptanol
GC/FID	EPA 8015C	n-Propanol
GC/FID	EPA 8015C	n-Propyl acetate
GC/FID	EPA 8015C	Phenol
GC/FID	EPA 8015C	Propylene glycol
GC/FID	EPA 8015C	sec-Butanol
GC/FID	EPA 8015C	sec-Butyl acetate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C	Tert-amyl alcohol
GC/FID	EPA 8015C	tert-Butyl alcohol
GC/FID	EPA 8015C	Tetraethylene glycol
GC/FID	EPA 8015C	Triethylene glycol
GC/ECD	EPA 8081B	2,4’ DDE
GC/ECD	EPA 8081B	2,4’-DDD
GC/ECD	EPA 8081B	2,4’-DDT
GC/ECD	EPA 8081B	4,4’ DDE
GC/ECD	EPA 8081B	4,4’-DDD
GC/ECD	EPA 8081B	4,4’-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC
GC/ECD	EPA 8081B	beta-BHC
GC/ECD	EPA 8081B	Chlordane (alpha)
GC/ECD	EPA 8081B	Chlordane (gamma)
GC/ECD	EPA 8081B	Chlordane (technical)
GC/ECD	EPA 8081B	Chlorobenzilate
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I (alpha)
GC/ECD	EPA 8081B	Endosulfan II (beta)
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	gamma-BHC
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Isodrin
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Toxaphene
GC/ECD	EPA 8082A	PCB-1016
GC/ECD	EPA 8082A	PCB-1221
GC/ECD	EPA 8082A	PCB-1232
GC/ECD	EPA 8082A	PCB-1242
GC/ECD	EPA 8082A	PCB-1248
GC/ECD	EPA 8082A	PCB-1254
GC/ECD	EPA 8082A	PCB-1260
GC/ECD	EPA 8082A	PCB-1262
GC/ECD	EPA 8082A	PCB-1268
GC/ECD	EPA 8082A	PCBs, Total
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4,6-Trichlorophenol
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	2,6-Dichlorophenol
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	DCPA (Dacthal)
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	Picloram
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethene
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane
GC/MS	EPA 8260B	1,2-Dichloroethene, Total
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,2-Xylene
GC/MS	EPA 8260B	1,3 & 1,4-Xylene
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,3-Dichloropropene, Total
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	1-Chlorohexane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone
GC/MS	EPA 8260B	3-Chloro-1-propene
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Isopropyltoluene
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonitrile
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	BTEX, Total
GC/MS	EPA 8260B	Carbon disulfide
GC/MS	EPA 8260B	Carbon tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene
GC/MS	EPA 8260B	cis-1,3-Dichloropropene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane
GC/MS	EPA 8260B	Diethyl ether
GC/MS	EPA 8260B	Ethanol
GC/MS	EPA 8260B	Ethyl benzene
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Furan
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutanol
GC/MS	EPA 8260B	Isopropyl ether
GC/MS	EPA 8260B	Isopropylbenzene
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl acetate
GC/MS	EPA 8260B	Methyl cyclohexane
GC/MS	EPA 8260B	Methyl isobutyl ketone
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260B	Methylene chloride
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Heptane
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	Pentachloroethane
GC/MS	EPA 8260B	Propionitrile

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene
GC/MS	EPA 8260B	Trichlorofluoromethane
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl chloride
GC/MS	EPA 8260B	Xylenes, total
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane (DBCP)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethene, Total
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,2-Xylene
GC/MS	EPA 8260C	1,3 & 1,4-Xylene
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropene, Total
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	3-Chloro-1-propene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	4-Isopropyltoluene
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	BTEX, Total
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Chloroprene
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethanol
GC/MS	EPA 8260C	Ethyl benzene
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Furan
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	Hexane
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropyl ether
GC/MS	EPA 8260C	Isopropylbenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl cyclohexane
GC/MS	EPA 8260C	Methyl isobutyl ketone
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	n-Heptane
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	Xylenes, total

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 8260D	1,2-Dichloroethene, Total
GC/MS	EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,2-Xylene
GC/MS	EPA 8260D	1,3 & 1,4-Xylene
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,3-Dichloropropene, Total
GC/MS	EPA 8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260D	1,4-Dioxane
GC/MS	EPA 8260D	1-Chlorohexane
GC/MS	EPA 8260D	2,2-Dichloropropane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	2-Butanone
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone
GC/MS	EPA 8260D	3-Chloro-1-propene
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Isopropyltoluene
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	Bromomethane
GC/MS	EPA 8260D	BTEX, Total
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloromethane
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Dibromochloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Dibromomethane
GC/MS	EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diethyl ether
GC/MS	EPA 8260D	Ethanol
GC/MS	EPA 8260D	Ethyl benzene
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Furan
GC/MS	EPA 8260D	Hexachlorobutadiene
GC/MS	EPA 8260D	Hexane
GC/MS	EPA 8260D	Iodomethane
GC/MS	EPA 8260D	Isobutanol
GC/MS	EPA 8260D	Isopropyl ether
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl acetate
GC/MS	EPA 8260D	Methyl cyclohexane
GC/MS	EPA 8260D	Methyl isobutyl ketone
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	n-Heptane
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	Propionitrile
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	Styrene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260D	tert-Butylbenzene
GC/MS	EPA 8260D	Tetrachloroethene
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260D	Trichloroethene
GC/MS	EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride
GC/MS	EPA 8260D	Xylenes, total
GC/MS	EPA 8270D	1,1-Biphenyl
GC/MS	EPA 8270D	1,2,3-Trichlorobenzene
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,2-Diphenylhydrazine
GC/MS	EPA 8270D	1,3,5-Trichlorobenzene
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Methylnaphthalene
GC/MS	EPA 8270D	1-Naphthylamine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	2,3,6-Trichlorophenol
GC/MS	EPA 8270D	2,3-Dimethylphenol
GC/MS	EPA 8270D	2,3-Xylenol
GC/MS	EPA 8270D	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,5-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270D	3 & 4-Methylphenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	3,3-Dichlorobenzidine
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	3,4-Dimethylphenol
GC/MS	EPA 8270D	3,4-Xylenol
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	4-Bromophenylphenyl ether
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	4-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	alpha-, alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	alpha-Pinene
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite, Total
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo (a) anthracene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Benzo (a) pyrene
GC/MS	EPA 8270D	Benzo (b) fluoranthene
GC/MS	EPA 8270D	Benzo (ghi) perylene
GC/MS	EPA 8270D	Benzo (k) fluoranthene
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270D	Bis (2-chloroethyl) ether
GC/MS	EPA 8270D	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270D	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols
GC/MS	EPA 8270D	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h) anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	Diphenyl ether
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methane sulfonate
GC/MS	EPA 8270D	Famphur

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Hexachlrobutadiene
GC/MS	EPA 8270D	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	Methyl methane sulfonate
GC/MS	EPA 8270D	Methylbenzoate
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	o,o',o''-Triethylphosphorothioate
GC/MS	EPA 8270D	Parathion ethyl
GC/MS	EPA 8270D	Parathion methyl

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Pentachlronitrobenzene
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	Phenyl ether
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole, Total
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270D	2,4-Dinitrochlorobenzene
GC/MS	EPA 8270E	1,1-Biphenyl
GC/MS	EPA 8270E	1,2,3-Trichlorobenzene
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270E	1,2-Dichlorobenzene
GC/MS	EPA 8270E	1,2-Diphenylhydrazine
GC/MS	EPA 8270E	1,3,5-Trichlorobenzene
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene
GC/MS	EPA 8270E	1,3-Dichlorobenzene
GC/MS	EPA 8270E	1,3-Dinitrobenzene
GC/MS	EPA 8270E	1,4-Dichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	1,4-Dioxane
GC/MS	EPA 8270E	1,4-Naphthoquinone
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,3,6-Trichlorophenol
GC/MS	EPA 8270E	2,3-Dimethylphenol
GC/MS	EPA 8270E	2,3-Xylenol
GC/MS	EPA 8270E	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene
GC/MS	EPA 8270E	2,5-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	2-Picoline
GC/MS	EPA 8270E	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270E	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270E	3 & 4-Methylphenol
GC/MS	EPA 8270E	3,3-Dichlorobenzidine
GC/MS	EPA 8270E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270E	3,4-Dimethylphenol
GC/MS	EPA 8270E	3,4-Xylenol
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	4-Bromophenylphenyl ether
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenol
GC/MS	EPA 8270E	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270E	4-Nitroaniline
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270E	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	alpha-, alpha-Dimethylphenethylamine
GC/MS	EPA 8270E	alpha-Pinene
GC/MS	EPA 8270E	Aniline
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Aramite, Total

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Benzaldehyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E	Benzo (a) anthracene
GC/MS	EPA 8270E	Benzo (a) pyrene
GC/MS	EPA 8270E	Benzo (b) fluoranthene
GC/MS	EPA 8270E	Benzo (ghi) perylene
GC/MS	EPA 8270E	Benzo (k) fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270E	Bis (2-chloroethyl) ether
GC/MS	EPA 8270E	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270E	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270E	Butyl benzyl phthalate
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	Cresols
GC/MS	EPA 8270E	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 8270E	Dibenz(a,h) anthracene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethyl phthalate
GC/MS	EPA 8270E	Dimethoate
GC/MS	EPA 8270E	Dimethyl phthalate
GC/MS	EPA 8270E	Di-n-butyl phthalate
GC/MS	EPA 8270E	Di-n-octyl phthalate

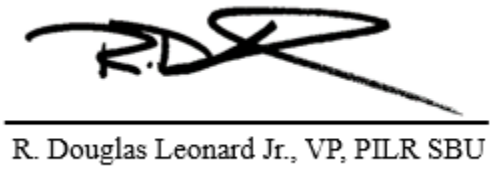
Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Diphenyl ether
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Ethyl methane sulfonate
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachlorophene
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 8270E	Hexachlrobutadiene
GC/MS	EPA 8270E	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Methapyrilene
GC/MS	EPA 8270E	Methyl methane sulfonate
GC/MS	EPA 8270E	Methylbenzoate
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	N-Nitrosodiethylamine
GC/MS	EPA 8270E	N-Nitrosodimethylamine
GC/MS	EPA 8270E	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270E	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270E	N-Nitrosodiphenylamine
GC/MS	EPA 8270E	N-Nitrosomethylethylamine
GC/MS	EPA 8270E	N-Nitrosomorpholine
GC/MS	EPA 8270E	N-Nitrosopiperidine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	N-Nitrosopyrrolidine
GC/MS	EPA 8270E	o,o',o''-Triethylphosphorothioate
GC/MS	EPA 8270E	Parathion ethyl
GC/MS	EPA 8270E	Parathion methyl
GC/MS	EPA 8270E	p-Dimethylaminoazobenzene
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachlorophenol
GC/MS	EPA 8270E	Pentachlronitrobenzene
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Phenyl ether
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	p-Phenylene diamine
GC/MS	EPA 8270E	Pronamide
GC/MS	EPA 8270E	Pyrene
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	Safrole, Total
GC/MS	EPA 8270E	Sulfotepp
GC/MS	EPA 8270E	Thionazin
GC/MS	EPA 8270E	2,4-Dinitrochlorobenzene
General Chemistry	EPA 9012B	Cyanide
General Chemistry	EPA 9013 EPA 9012B	Cyanide amenable to chlorination
General Chemistry	EPA 9030B EPA 9034	Sulfide
General Chemistry	EPA 9038	Sulfate
General Chemistry	EPA 9045D	pH
General Chemistry	EPA 9050A	Specific conductance

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate (as N)
IC	EPA 9056A	Nitrate (as NO3)
IC	EPA 9056A	Nitrate-nitrite (as N)
IC	EPA 9056A	Nitrate-nitrite (as NO3-NO2)
IC	EPA 9056A	Nitrite (as N)
IC	EPA 9056A	Nitrite (as NO2)
IC	EPA 9056A	Sulfate
General Chemistry	EPA 9065A	Phenols
General Chemistry	EPA 9071B	Oil and Grease
General Chemistry	EPA 9071B	Total Petroleum Hydrocarbons
General Chemistry	EPA 9095B	Free Liquid
General Chemistry	EPA 9251	Chloride
Preparation	**Method**	**Type**
Organic preparation	EPA 3546	Microwave Extraction
TCLP preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching procedure
Purge & Trap	EPA 5035A	Volatiles Prep
Acid Digestion	EPA 3050B	Metals Prep
Preparation	EPA 5050	Bomb Prep
Distillation	EPA 9030B	Sulfide

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2463.



R. Douglas Leonard Jr., VP, PILR SBU

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Chlorinated Acids	
2,4,5-TP (Silvex)	EPA 515.1
2,4-D	EPA 515.1
Dalapon	EPA 515.1
Dicamba	EPA 515.1
Dinoseb	EPA 515.1
Pentachlorophenol	EPA 515.1
Picloram	EPA 515.1
Disinfection By-products	
Bromate	EPA 300.1 Rev. 1.0
Bromide	EPA 300.1 Rev. 1.0
Chlorate	EPA 300.1 Rev. 1.0
Chlorite	EPA 300.1 Rev. 1.0
Dibromoacetic acid	EPA 552.2
Dichloroacetic acid	EPA 552.2
Monobromoacetic acid	EPA 552.2
Monochloroacetic acid	EPA 552.2
Trichloroacetic acid	EPA 552.2
Fuel Additives	
Methyl tert-butyl ether	EPA 524.2
Naphthalene	EPA 524.2
Metals I	
Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Metals I	
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 200.8 Rev. 5.4
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Metals II	
Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Serial No.: 61192

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Metals II	
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Metals III	
Boron, Total	EPA 200.7 Rev. 4.4
Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4
Methylcarbamate Pesticides	
3-Hydroxy Carbofuran	EPA 531.1
Aldicarb	EPA 531.1
Aldicarb Sulfone	EPA 531.1
Aldicarb Sulfoxide	EPA 531.1
Carbaryl	EPA 531.1
Carbofuran	EPA 531.1
Methomyl	EPA 531.1
Oxamyl	EPA 531.1
Microextractables	
1,2-Dibromo-3-chloropropane, Low Level	EPA 504.1
1,2-Dibromoethane, Low Level	EPA 504.1
Miscellaneous	
Benzo(a)pyrene	EPA 525.2
Bis(2-ethylhexyl) phthalate	EPA 525.2
Di (2-ethylhexyl) adipate	EPA 525.2
Diquat	EPA 549.2
Endothall	EPA 548.1
Glyphosate	EPA 547
Hexachlorobenzene	EPA 525.2
Hexachlorocyclopentadiene	EPA 525.2
Odor	SM 21-23 2150 B (-97)
Organic Carbon, Dissolved	SM 21-23 5310B (-00)
Organic Carbon, Total	SM 21-23 5310B (-00)
Turbidity	SM 21-23 2130 B (-01)
	EPA 180.1 Rev. 2.0
UV 254	SM 21-23 5910B (-00,-11)
Non-Metals	
Alkalinity	SM 21-23 2320B (-97)
Calcium Hardness	SM 18-22 2340C (-97)
	SM 18-22 2340B (-97)
Chloride	EPA 300.0 Rev. 2.1
	SM 21-22 4500-Cl- E (-97)
Color	SM 21-23 2120B (-01)
Corrosivity	SM 18-22 2330
Cyanide	SM 20, 21-23 4500-CN E
	EPA 335.4 Rev. 1.0
Fluoride, Total	EPA 300.0 Rev. 2.1

Serial No.: 61192

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below.

Non-Metals	
Nitrate (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
Nitrite (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
Orthophosphate (as P)	EPA 365.1 Rev. 2.0
	SM 19, 21-23 4500-P F (-99)
Silica, Dissolved	EPA 200.7 Rev. 4.4
Solids, Total Dissolved	SM 21-23 2540C (-97)
Specific Conductance	SM 21-23 2510B (-97)
Sulfate (as SO4)	EPA 300.0 Rev. 2.1
Organohalide Pesticides	
Alachlor	EPA 525.2
Aldrin	EPA 508
	EPA 525.2
Atrazine	EPA 525.2
Butachlor	EPA 525.2
Chlordane Total	EPA 508
Dieldrin	EPA 508
	EPA 525.2
Endrin	EPA 508
	EPA 525.2
Heptachlor	EPA 508
	EPA 525.2
Heptachlor epoxide	EPA 508
	EPA 525.2

Organohalide Pesticides	
Lindane	EPA 508
	EPA 525.2
Methoxychlor	EPA 508
	EPA 525.2
Metolachlor	EPA 525.2
Metribuzin	EPA 525.2
Propachlor	EPA 525.2
Simazine	EPA 525.2
Toxaphene	EPA 508
Trifluralin	EPA 525.2
Polychlorinated Biphenyls	
PCB Screen	EPA 508
Trihalomethanes	
Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2
Total Trihalomethanes	EPA 524.2
Volatile Aromatics	
1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2

Serial No.: 61192

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Volatile Aromatics	
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2

Volatile Halocarbons	
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

Serial No.: 61192

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
	EPA 624.1
Acrylonitrile	EPA 8260C
	EPA 624.1
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1,4-Phenylenediamine	EPA 8270D
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
a,a-Dimethylphenethylamine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Methapyrilene	EPA 8270D
Pronamide	EPA 8270D
Pyridine	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 625.1
	EPA 8270D
Benzidines	
3,3'-Dimethylbenzidine	EPA 8270D
Benzidine	EPA 625.1
	EPA 8270D
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 8081B
	EPA 608.3
4,4'-DDE	EPA 8081B
	EPA 608.3
4,4'-DDT	EPA 8081B
	EPA 608.3
Aldrin	EPA 8081B
	EPA 608.3
alpha-BHC	EPA 8081B
	EPA 608.3
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
	EPA 608.3
Chlordane Total	EPA 8081B
	EPA 608.3
Chlorobenzilate	EPA 8081B
delta-BHC	EPA 8081B
	EPA 608.3
Dieldrin	EPA 8081B
	EPA 608.3
Endosulfan I	EPA 8081B

Serial No.: 61193

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorinated Hydrocarbon Pesticides	
Endosulfan I	EPA 608.3
Endosulfan II	EPA 8081B
	EPA 608.3
Endosulfan sulfate	EPA 8081B
	EPA 608.3
Endrin	EPA 8081B
	EPA 608.3
Endrin aldehyde	EPA 8081B
	EPA 608.3
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
	EPA 608.3
Heptachlor epoxide	EPA 8081B
	EPA 608.3
Isodrin	EPA 8081B
Kepone	EPA 8081B
Lindane	EPA 8081B
	EPA 608.3
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
Toxaphene	EPA 8081B
	EPA 608.3
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C

Chlorinated Hydrocarbons	
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 625.1
	EPA 8270D
2-Chloronaphthalene	EPA 625.1
	EPA 8270D
Hexachlorobenzene	EPA 625.1
	EPA 8270D
Hexachlorobutadiene	EPA 625.1
	EPA 8270D
Hexachlorocyclopentadiene	EPA 625.1
	EPA 8270D
Hexachloroethane	EPA 625.1
	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
	EPA 8270D

Serial No.: 61193

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorophenoxy Acid Pesticides	
Pentachlorophenol	EPA 8151A
Demand	
Biochemical Oxygen Demand	SM 5210B-2011
Carbonaceous BOD	SM 5210B-2011
Chemical Oxygen Demand	EPA 410.4, Rev. 2.0 (1993)
	SM 5220D-2011
Dissolved Gases	
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Fuel Oxygenates	
Ethanol	EPA 8260C
	EPA 8015C
Methyl tert-butyl ether	EPA 8260C
tert-butyl alcohol	EPA 8260C
	EPA 8015C
Haloethers	
2,2'-Oxybis(1-chloropropane)	EPA 625.1
	EPA 8270D
4-Bromophenylphenyl ether	EPA 625.1
	EPA 8270D
4-Chlorophenylphenyl ether	EPA 625.1
	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 625.1
Haloethers	
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 625.1
	EPA 8270D
Low Level Halocarbons	
1,2-Dibromo-3-chloropropane, Low Level	EPA 8011
1,2-Dibromoethane, Low Level	EPA 8011
Metals I	
Barium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Cadmium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Calcium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Chromium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Copper, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)

Serial No.: 61193

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Metals I		Metals II	
Iron, Total	EPA 200.7, Rev. 4.4 (1994)	Aluminum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C		EPA 6010C
Lead, Total	EPA 200.7, Rev. 4.4 (1994)		EPA 6020A
	EPA 6010C		EPA 200.8, Rev. 5.4 (1994)
	EPA 6020A	Antimony, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 200.8, Rev. 5.4 (1994)		EPA 6010C
Magnesium, Total	EPA 200.7, Rev. 4.4 (1994)		EPA 6020A
	EPA 6010C		EPA 200.8, Rev. 5.4 (1994)
Manganese, Total	EPA 200.7, Rev. 4.4 (1994)	Arsenic, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C		EPA 6010C
	EPA 6020A		EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)		EPA 200.8, Rev. 5.4 (1994)
Nickel, Total	EPA 200.7, Rev. 4.4 (1994)	Beryllium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C		EPA 6010C
	EPA 6020A		EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)		EPA 200.8, Rev. 5.4 (1994)
Potassium, Total	EPA 200.7, Rev. 4.4 (1994)	Chromium VI	EPA 7196A
	EPA 6010C	Mercury, Total	EPA 245.1, Rev. 3.0 (1994)
Silver, Total	EPA 200.7, Rev. 4.4 (1994)		EPA 7470A
	EPA 6010C	Selenium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6020A		EPA 6010C
	EPA 200.8, Rev. 5.4 (1994)		EPA 6020A
Sodium, Total	EPA 200.7, Rev. 4.4 (1994)		EPA 200.8, Rev. 5.4 (1994)
	EPA 6010C	Vanadium, Total	EPA 200.7, Rev. 4.4 (1994)
Strontium, Total	EPA 200.7, Rev. 4.4 (1994)		EPA 6010C
	EPA 6010C		EPA 6020A

Serial No.: 61193

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Metals II	
Vanadium, Total	EPA 200.8, Rev. 5.4 (1994)
Zinc, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Metals III	
Cobalt, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Molybdenum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Thallium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
	EPA 6020A
	EPA 200.8, Rev. 5.4 (1994)
Tin, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Titanium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Mineral	
Alkalinity	SM 2320B-2011
Chloride	EPA 300.0, Rev. 2.1 (1993)
Mineral	
Chloride	SM 4500-Cl- E-2011
	EPA 9056A
Fluoride, Total	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Hardness, Total	SM 2340C-2011
	SM 2340B-2011
Sulfate (as SO4)	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Miscellaneous	
Boron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010C
Bromide	EPA 300.1, Rev. 1.0 (1997)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Color	SM 2120B-2011
Corrosivity	SM 2330
Cyanide, Total	SM 4500-CN E-2011
	EPA 335.4, Rev. 1.0 (1993)
Formaldehyde	EPA 8315A
non-Polar Extractable Material (TPH)	EPA 1664A
Oil and Grease Total Recoverable (HEM)	EPA 1664A
Organic Carbon, Total	SM 5310B-2011
Phenols	EPA 420.1 (Rev. 1978)
	EPA 9065
Silica, Dissolved	EPA 200.7, Rev. 4.4 (1994)

Serial No.: 61193

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Miscellaneous	
Specific Conductance	EPA 120.1 (Rev. 1982)
	SM 2510B-2011
	EPA 9050A
Sulfide (as S)	SM 4500-S2- F-2011
	EPA 9034
Surfactant (MBAS)	SM 5540C-2011
Total Organic Halides	EPA 9020B
Turbidity	SM 2130 B-2011
	EPA 180.1, Rev. 2.0 (1993)
Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8270D
1,3-Dinitrobenzene	EPA 8270D
1,4-Naphthoquinone	EPA 8270D
2,4-Dinitrotoluene	EPA 625.1
	EPA 8270D
2,6-Dinitrotoluene	EPA 625.1
	EPA 8270D
Isophorone	EPA 625.1
	EPA 8270D
Nitrobenzene	EPA 625.1
	EPA 8270D
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 625.1
	EPA 8270D

Nitrosoamines	
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 625.1
	EPA 8270D
N-Nitrosodiphenylamine	EPA 625.1
	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D
Nutrient	
Ammonia (as N)	EPA 350.1, Rev. 2.0 (1993)
Kjeldahl Nitrogen, Total	EPA 351.2, Rev. 2.0 (1993)
Nitrate (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Orthophosphate (as P)	EPA 365.1, Rev. 2.0 (1993)
	SM 4500-P F-2011 or G-2011
Phosphorus, Total	EPA 365.4 (Issued 1974)
Organophosphate Pesticides	
Disulfoton	EPA 8270D
Famphur	EPA 8270D
Parathion ethyl	EPA 8270D

Serial No.: 61193

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12.01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Organophosphate Pesticides	
Parathion methyl	EPA 8270D
Phorate	EPA 8270D
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015C
Gasoline Range Organics	EPA 8015C
Phthalate Esters	
Benzyl butyl phthalate	EPA 625.1
	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 625.1
	EPA 8270D
Diethyl phthalate	EPA 625.1
	EPA 8270D
Dimethyl phthalate	EPA 625.1
	EPA 8270D
Di-n-butyl phthalate	EPA 625.1
	EPA 8270D
Di-n-octyl phthalate	EPA 625.1
	EPA 8270D
Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
	EPA 608.3
Aroclor 1221 (PCB-1221)	EPA 8082A
	EPA 608.3
Aroclor 1232 (PCB-1232)	EPA 8082A

Polychlorinated Biphenyls	
Aroclor 1232 (PCB-1232)	EPA 608.3
Aroclor 1242 (PCB-1242)	EPA 8082A
	EPA 608.3
Aroclor 1248 (PCB-1248)	EPA 8082A
	EPA 608.3
Aroclor 1254 (PCB-1254)	EPA 8082A
	EPA 608.3
Aroclor 1260 (PCB-1260)	EPA 8082A
	EPA 608.3
Polynuclear Aromatics	
2-Acetylaminofluorene	EPA 8270D
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 625.1
	EPA 8270D
Acenaphthylene	EPA 625.1
	EPA 8270D
Anthracene	EPA 625.1
	EPA 8270D
Benzo(a)anthracene	EPA 625.1
	EPA 8270D
Benzo(a)pyrene	EPA 625.1
	EPA 8270D
Benzo(b)fluoranthene	EPA 625.1
	EPA 8270D

Serial No.: 61193

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Polynuclear Aromatics	
Benzo(g,h,i)perylene	EPA 625.1
	EPA 8270D
Benzo(k)fluoranthene	EPA 625.1
	EPA 8270D
Chrysene	EPA 625.1
	EPA 8270D
Dibenzo(a,h)anthracene	EPA 625.1
	EPA 8270D
Fluoranthene	EPA 625.1
	EPA 8270D
Fluorene	EPA 625.1
	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 625.1
	EPA 8270D
Naphthalene	EPA 625.1
	EPA 8270D
Phenanthrene	EPA 625.1
	EPA 8270D
Pyrene	EPA 625.1
	EPA 8270D
Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 625.1
	EPA 8270D

Priority Pollutant Phenols	
2,4-Dichlorophenol	EPA 625.1
	EPA 8270D
2,4-Dimethylphenol	EPA 625.1
	EPA 8270D
2,4-Dinitrophenol	EPA 625.1
	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 625.1
	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 625.1
	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 625.1
	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 625.1
	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 625.1
	EPA 8270D
Pentachlorophenol	EPA 625.1
	EPA 8270D
Phenol	EPA 625.1
	EPA 8270D

Serial No.: 61193

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Residue	
Settleable Solids	SM 2540 F-2011
Solids, Total	SM 2540 B-2011
Solids, Total Dissolved	SM 2540 C-2011
Solids, Total Suspended	SM 2540 D-2011
Semi-Volatile Organics	
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
2-Picoline	EPA 8270D
4-Amino biphenyl	EPA 8270D
Acetophenone	EPA 8270D
Aramite	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Methyl methanesulfonate	EPA 8270D
O,O,O-Triethyl phosphorothioate	EPA 8270D
p-Dimethylaminoazobenzene	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
Volatile Aromatics	
1,2,4-Trimethylbenzene	EPA 8260C
1,2-Dichlorobenzene	EPA 8260C
	EPA 624.1
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 8260C
	EPA 624.1
1,4-Dichlorobenzene	EPA 8260C
	EPA 624.1
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 8260C
	EPA 624.1
Bromobenzene	EPA 8260C
Chlorobenzene	EPA 8260C
	EPA 624.1
Ethyl benzene	EPA 8260C
	EPA 624.1
Isopropylbenzene	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C
p-Isopropyltoluene (P-Cymene)	EPA 8260C
sec-Butylbenzene	EPA 8260C
Styrene	EPA 8260C
tert-Butylbenzene	EPA 8260C
Toluene	EPA 8260C

Serial No.: 61193

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Volatile Aromatics	
Toluene	EPA 624.1
Total Xylenes	EPA 8260C
	EPA 624.1
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
	EPA 624.1
1,1,2,2-Tetrachloroethane	EPA 8260C
	EPA 624.1
1,1,2-Trichloroethane	EPA 8260C
	EPA 624.1
1,1-Dichloroethane	EPA 8260C
	EPA 624.1
1,1-Dichloroethene	EPA 8260C
	EPA 624.1
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloroethane	EPA 8260C
	EPA 624.1
1,2-Dichloropropane	EPA 8260C
	EPA 624.1
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C

Volatile Halocarbons	
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
2-Chloroethylvinyl ether	EPA 8260C
	EPA 624.1
3-Chloropropene (Allyl chloride)	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
	EPA 624.1
Bromoform	EPA 8260C
	EPA 624.1
Bromomethane	EPA 8260C
	EPA 624.1
Carbon tetrachloride	EPA 8260C
	EPA 624.1
Chloroethane	EPA 8260C
	EPA 624.1
Chloroform	EPA 8260C
	EPA 624.1
Chloromethane	EPA 8260C
	EPA 624.1
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
	EPA 624.1
Dibromochloromethane	EPA 8260C
	EPA 624.1
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C

Serial No.: 61193

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Volatile Halocarbons	
Hexachlorobutadiene, Volatile	EPA 8260C
Methyl iodide	EPA 8260C
Methylene chloride	EPA 8260C
	EPA 624.1
Tetrachloroethene	EPA 8260C
	EPA 624.1
trans-1,2-Dichloroethene	EPA 8260C
	EPA 624.1
trans-1,3-Dichloropropene	EPA 8260C
	EPA 624.1
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
	EPA 624.1
Trichlorofluoromethane	EPA 8260C
	EPA 624.1
Vinyl chloride	EPA 8260C
	EPA 624.1
Volatiles Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260C
2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C

Volatiles Organics	
Di-ethyl ether	EPA 8260C
Ethyl Acetate	EPA 1666A
	EPA 8015C
Isobutyl alcohol	EPA 8260C
	EPA 8015C
Isopropyl Acetate	EPA 1666A
n-Amyl Acetate	EPA 1666A
n-Butyl Acetate	EPA 1666A
o-Toluidine	EPA 8270D
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 5030C
	EPA 9030B
	EPA 3010A
	EPA 3005A
	EPA 3520C
	EPA 3020A

Serial No.: 61193

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1,2-Diphenylhydrazine	EPA 8270D
1,4-Phenylenediamine	EPA 8270D
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
a,a-Dimethylphenethylamine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Methapyrilene	EPA 8270D
Pronamide	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 8270D
3,3'-Dimethylbenzidine	EPA 8270D
Benzidine	EPA 8270D

Characteristic Testing	
Free Liquids	EPA 9095B
Ignitability	EPA 1030
Synthetic Precipitation Leaching Proc	EPA 1312
TCLP	EPA 1311
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
Chlorobenzilate	EPA 8081B
delta-BHC	EPA 8081B
Dialiate	EPA 8270D
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B

Serial No.: 61194

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Chlorinated Hydrocarbon Pesticides	
Heptachlor epoxide	EPA 8081B
Kepone	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Pentachloronitrobenzene	EPA 8270D
Toxaphene	EPA 8081B
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 8270D
2-Chloronaphthalene	EPA 8270D
Hexachlorobenzene	EPA 8270D
Hexachlorobutadiene	EPA 8270D
Hexachlorocyclopentadiene	EPA 8270D
Hexachloroethane	EPA 8270D
Hexachlorophene	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A

Chlorophenoxy Acid Pesticides	
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
	EPA 8270D
MCPA	EPA 8151A
MCPP	EPA 8151A
Pentachlorophenol	EPA 8151A
Haloethers	
2,2'-Oxybis(1-chloropropane)	EPA 8270D
4-Bromophenylphenyl ether	EPA 8270D
4-Chlorophenylphenyl ether	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 8270D
Metals I	
Barium, Total	EPA 6010C
	EPA 6020A
Cadmium, Total	EPA 6010C
	EPA 6020A
Calcium, Total	EPA 6010C
Chromium, Total	EPA 6010C
	EPA 6020A
Copper, Total	EPA 6010C
	EPA 6020A
Iron, Total	EPA 6010C
Lead, Total	EPA 6010C
	EPA 6020A

Serial No.: 61194

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Metals I	
Magnesium, Total	EPA 6010C
Manganese, Total	EPA 6010C
	EPA 6020A
Nickel, Total	EPA 6010C
	EPA 6020A
Potassium, Total	EPA 6010C
Silver, Total	EPA 6010C
	EPA 6020A
Sodium, Total	EPA 6010C
Strontium, Total	EPA 6010C
Metals II	
Aluminum, Total	EPA 6010C
	EPA 6020A
Antimony, Total	EPA 6010C
	EPA 6020A
Arsenic, Total	EPA 6010C
	EPA 6020A
Beryllium, Total	EPA 6010C
	EPA 6020A
Chromium VI	EPA 7196A
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010C
	EPA 6020A
Vanadium, Total	EPA 6010C
	EPA 6020A
Metals II	
Zinc, Total	EPA 6010C
	EPA 6020A
Metals III	
Cobalt, Total	EPA 6010C
	EPA 6020A
Molybdenum, Total	EPA 6010C
	EPA 6020A
Thallium, Total	EPA 6010C
	EPA 6020A
Tin, Total	EPA 6010C
Titanium, Total	EPA 6010C
Minerals	
Bromide	EPA 9056A
Chloride	EPA 9056A
Fluoride, Total	EPA 9056A
Sulfate (as SO4)	EPA 9038
	EPA 9056A
Miscellaneous	
Boron, Total	EPA 6010C
Phenols	EPA 9065
Sulfide (as S)	EPA 9034
Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8270D
1,3-Dinitrobenzene	EPA 8270D

Serial No.: 61194

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12.01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Nitroaromatics and Isophorone	
1,4-Naphthoquinone	EPA 8270D
2,4-Dinitrotoluene	EPA 8270D
2,6-Dinitrotoluene	EPA 8270D
4-Dimethylaminoazobenzene	EPA 8270D
4-Nitroquinoline-1-oxide	EPA 8270D
Isophorone	EPA 8270D
Nitrobenzene	EPA 8270D
Pyridine	EPA 8270D
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 8270D
N-Nitrosodiphenylamine	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D
Nutrients	
Nitrate (as N)	EPA 9056A
Nitrite (as N)	EPA 9056A
Organophosphate Pesticides	
Dimethoate	EPA 8270D
Disulfoton	EPA 8270D
Organophosphate Pesticides	
Famphur	EPA 8270D
Parathion ethyl	EPA 8270D
Parathion methyl	EPA 8270D
Phorate	EPA 8270D
Sulfotepp	EPA 8270D
Thionazin	EPA 8270D
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015C
Gasoline Range Organics	EPA 8015C
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 8270D
Diethyl phthalate	EPA 8270D
Dimethyl phthalate	EPA 8270D
Di-n-butyl phthalate	EPA 8270D
Di-n-octyl phthalate	EPA 8270D
Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A

Serial No.: 61194

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Polynuclear Aromatic Hydrocarbons	
2-Acetylaminofluorene	EPA 8270D
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 8270D
Acenaphthylene	EPA 8270D
Anthracene	EPA 8270D
Benzo(a)anthracene	EPA 8270D
Benzo(a)pyrene	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D
Benzo(g,h,i)perylene	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 8270D
Dibenzo(a,h)anthracene	EPA 8270D
Fluoranthene	EPA 8270D
Fluorene	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D
Naphthalene	EPA 8270D
Phenanthrene	EPA 8270D
Pyrene	EPA 8270D

Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 8270D
2,4-Dichlorophenol	EPA 8270D
2,4-Dimethylphenol	EPA 8270D

Priority Pollutant Phenols	
2,4-Dinitrophenol	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 8270D
Pentachlorophenol	EPA 8270D
Phenol	EPA 8270D

Semi-Volatile Organics	
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
2-Picoline	EPA 8270D
4-Amino biphenyl	EPA 8270D
Acetophenone	EPA 8270D
Aramite	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D

Serial No.: 61194

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Semi-Volatile Organics	
Isosafrole	EPA 8270D
Methyl methanesulfonate	EPA 8270D
O,O,O-Triethyl phosphorothioate	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260C
1,2-Dichlorobenzene	EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 8260C
1,4-Dichlorobenzene	EPA 8260C
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 8260C
Bromobenzene	EPA 8260C
Chlorobenzene	EPA 8260C
Ethyl benzene	EPA 8260C
Isopropylbenzene	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C
p-Isopropyltoluene (P-Cymene)	EPA 8260C
sec-Butylbenzene	EPA 8260C
Styrene	EPA 8260C
Volatile Aromatics	
tert-Butylbenzene	EPA 8260C
Toluene	EPA 8260C
Total Xylenes	EPA 8260C
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
1,1-Dichloroethane	EPA 8260C
1,1-Dichloroethene	EPA 8260C
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloroethane	EPA 8260C
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
3-Chloropropene (Allyl chloride)	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
Bromoform	EPA 8260C
Bromomethane	EPA 8260C
Carbon tetrachloride	EPA 8260C

Serial No.: 61194

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2021
Issued April 01, 2020

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. BERNARD KIRKLAND
EUROFINS TESTAMERICA INC. - SAVANNAH
5102 LAROCHE AVE
SAVANNAH, GA 31404

NY Lab Id No: 10842

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Volatile Halocarbons	
Chloroethane	EPA 8260C
Chloromethane	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
Dibromochloromethane	EPA 8260C
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methyl iodide	EPA 8260C
Methylene chloride	EPA 8260C
Tetrachloroethene	EPA 8260C
trans-1,2-Dichloroethene	EPA 8260C
trans-1,3-Dichloropropene	EPA 8260C
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
Trichlorofluoromethane	EPA 8260C
Vinyl chloride	EPA 8260C
Volatile Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260C
2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C

Volatile Organics	
Di-ethyl ether	EPA 8260C
Ethyl Acetate	EPA 8015C
Ethylene Glycol	EPA 8015C
Isobutyl alcohol	EPA 8260C
	EPA 8015C
Methyl tert-butyl ether	EPA 8260C
o-Toluidine	EPA 8270D
Propionitrile	EPA 8260C
tert-butyl alcohol	EPA 8260C
	EPA 8015C
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 5035A-L
	EPA 5035A-H
	EPA 9030B
	EPA 3010A
	EPA 3005A
	EPA 3550C
	EPA 3020A
	EPA 3546

Serial No.: 61194

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

THIS PAGE LEFT BLANK INTENTIONALLY

Eurofins Test America Certifications

Seattle

CERTIFICATE OF ACCREDITATION

ANSI National Accreditation Board

11617 Coldwater Road, Fort Wayne, IN 46845 USA

This is to certify that

Eurofins TestAmerica Laboratories, Inc. - Seattle

5755 8th Street East
Tacoma, WA 98424

has been assessed by ANAB and meets the requirements of international standard

ISO/IEC 17025:2005

and the

US Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V 5.1.1)

while demonstrating technical competence in the field of

TESTING

Refer to the accompanying Scope of Accreditation for information regarding the types of activities to which this accreditation applies

L2236
Certificate Number

ANAB Approval

Certificate Valid Through: 01/19/2022
Version No. 007 Issued: 07/03/2019

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005 AND DOD QUALITY SYSTEMS MAUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V 5.1.1)

Eurofins TestAmerica Laboratories, Inc. - Seattle

5755 8th Street East
Tacoma, WA 98424
Terri Torres
253-922-2310

TESTING

Valid to: **January 19, 2022** Certificate Number: **L2236**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D/200.7	Silver
ICP-AES	EPA 6010B/6010C/6010D/200.7	Aluminum
ICP-AES	EPA 6010B/6010C/6010D/200.7	Arsenic
ICP-AES	EPA 6010B/6010C/6010D/200.7	Boron
ICP-AES	EPA 6010B/6010C/6010D/200.7	Barium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Beryllium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Calcium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Cadmium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Cobalt
ICP-AES	EPA 6010B/6010C/6010D/200.7	Chromium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Copper
ICP-AES	EPA 6010B/6010C/6010D/200.7	Iron
ICP-AES	EPA 6010B/6010C/6010D/200.7	Potassium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Magnesium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Manganese
ICP-AES	EPA 6010B/6010C/6010D/200.7	Molybdenum
ICP-AES	EPA 6010B/6010C/6010D/200.7	Sodium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Nickel
ICP-AES	EPA 6010B/6010C/6010D/200.7	Lead
ICP-AES	EPA 6010B/6010C/6010D/200.7	Antimony

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D/200.7	Selenium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Silicon
ICP-AES	EPA 6010B/6010C/6010D/200.7	Tin
ICP-AES	EPA 6010B/6010C/6010D/200.7	Titanium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Strontium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Thallium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Vanadium
ICP-AES	EPA 6010B/6010C/6010D/200.7	Zinc
ICP-MS	EPA 6020/6020A/6020B/200.8	Silver
ICP-MS	EPA 6020/6020A/6020B/200.8	Aluminum
ICP-MS	EPA 6020/6020A/6020B/200.8	Arsenic
ICP-MS	EPA 6020/6020A/6020B/200.8	Barium
ICP-MS	EPA 6020/6020A/6020B/200.8	Beryllium
ICP-MS	EPA 6020/6020A/6020B/200.8	Cadmium
ICP-MS	EPA 6020/6020A/6020B/200.8	Cobalt
ICP-MS	EPA 6020/6020A/6020B/200.8	Chromium
ICP-MS	EPA 6020/6020A/6020B/200.8	Copper
ICP-MS	EPA 6020/6020A/6020B/200.8	Iron
ICP-MS	EPA 6020/6020A/6020B/200.8	Manganese
ICP-MS	EPA 6020/6020A/6020B/200.8	Molybdenum
ICP-MS	EPA 6020/6020A/6020B/200.8	Nickel
ICP-MS	EPA 6020/6020A/6020B/200.8	Lead
ICP-MS	EPA 6020/6020A/6020B/200.8	Antimony
ICP-MS	EPA 6020/6020A/6020B/200.8	Selenium
ICP-MS	EPA 6020/6020A/6020B/200.8	Thallium
ICP-MS	EPA 6020/6020A/6020B/200.8	Uranium
ICP-MS	EPA 6020/6020A/6020B/200.8	Vanadium
ICP-MS	EPA 6020/6020A/6020B/200.8	Zinc
CVAAS	EPA 7470A/245.1	Mercury
GC/MS	EPA 8260B/8260C/624/624.1	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C/624/624.1	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C/624/624.1	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C/624/624.1	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C/624/624.1	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C/624/624.1	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C/624/624.1	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C/624/624.1	1,2,3-Trichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/624/624.1	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C/624/624.1	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C/624/624.1	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	1,2-Dibromo-3-Chloropropane
GC/MS	EPA 8260B/8260C/624/624.1	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C/624/624.1	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C/624/624.1	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C/624/624.1	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C/624/624.1	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C/624/624.1	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C/624/624.1	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C/624/624.1	2-Chloroethylvinylether
GC/MS	EPA 8260B/8260C/624/624.1	2-Chlorotoluene
GC/MS	EPA 8260B/8260C/624/624.1	2-Hexanone
GC/MS	EPA 8260B/8260C/624/624.1	4-Chlorotoluene
GC/MS	EPA 8260B/8260C/624/624.1	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C/624/624.1	Acetone
GC/MS	EPA 8260B/8260C/624/624.1	Acetonitrile
GC/MS	EPA 8260B/8260C/624/624.1	Acrolein
GC/MS	EPA 8260B/8260C/624/624.1	Acrylonitrile
GC/MS	EPA 8260B/8260C/624/624.1	Benzene
GC/MS	EPA 8260B/8260C/624/624.1	Bromobenzene
GC/MS	EPA 8260B/8260C/624/624.1	Bromodichloromethane
GC/MS	EPA 8260B/8260C/624/624.1	Bromoform
GC/MS	EPA 8260B/8260C/624/624.1	Bromomethane
GC/MS	EPA 8260B/8260C/624/624.1	Carbon disulfide
GC/MS	EPA 8260B/8260C/624/624.1	Carbon tetrachloride
GC/MS	EPA 8260B/8260C/624/624.1	Chlorobenzene
GC/MS	EPA 8260B/8260C/624/624.1	Chlorobromomethane
GC/MS	EPA 8260B/8260C/624/624.1	Chlorodibromomethane
GC/MS	EPA 8260B/8260C/624/624.1	Chloroethane
GC/MS	EPA 8260B/8260C/624/624.1	Chloroform
GC/MS	EPA 8260B/8260C/624/624.1	Chloromethane
GC/MS	EPA 8260B/8260C/624/624.1	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C/624/624.1	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C/624/624.1	Dibromomethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/624/624.1	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C/624/624.1	Ethylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	Ethylene Dibromide
GC/MS	EPA 8260B/8260C/624/624.1	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C/624/624.1	Isopropylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	Methyl Ethyl Ketone
GC/MS	EPA 8260B/8260C/624/624.1	Methyl Isobutyl Ketone
GC/MS	EPA 8260B/8260C/624/624.1	Methyl tert-butyl ether
GC/MS	EPA 8260B/8260C/624/624.1	Methylene Chloride
GC/MS	EPA 8260B/8260C/624/624.1	m-Xylene & p-Xylene
GC/MS	EPA 8260B/8260C/624/624.1	Naphthalene
GC/MS	EPA 8260B/8260C/624/624.1	n-Butylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	N-Propylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	o-Xylene
GC/MS	EPA 8260B/8260C/624/624.1	sec-Butylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	Styrene
GC/MS	EPA 8260B/8260C/624/624.1	tert-Butylbenzene
GC/MS	EPA 8260B/8260C/624/624.1	Tetrachloroethene
GC/MS	EPA 8260B/8260C/624/624.1	Toluene
GC/MS	EPA 8260B/8260C/624/624.1	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C/624/624.1	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C/624/624.1	Trichloroethene
GC/MS	EPA 8260B/8260C/624/624.1	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C/624/624.1	Vinyl Acetate
GC/MS	EPA 8260B/8260C/624/624.1	Vinyl chloride
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1,1,2-Tetrachloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1-Dichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,2,4-Trimethylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,2-Dichloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,3,5-Trimethylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	2-Hexanone
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Benzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Bromoform
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Bromomethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Butadiene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Chlorodibromomethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Chloroform
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	cis-1,2-Dichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	cis-1,3-Dichloropropene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Ethylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Dibromomethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Bromodichloromethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Ethylene Dibromide
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Hexachlorobutadiene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Isopropylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Isopropyl alcohol
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	m&p-Xylene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Methyl tert-Butyl Ether
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Naphthalene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	n-Butylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	o-Xylene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Sec-Butylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Tert-Butylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Tetrachloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Toluene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	trans-1,3-Dichloropropene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Trichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Vinyl chloride
GC/MS	EPA 8270C/8270D/625/625.1	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D/625/625.1	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D/625/625.1	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D/625/625.1	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D/625/625.1	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D/625/625.1	bis(2-chloroisopropyl)ether
GC/MS	EPA 8270C/8270D/625/625.1	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D/625/625.1	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D/625/625.1	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D/625/625.1	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D/625/625.1	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D/625/625.1	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D/625/625.1	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D/625/625.1	2,6-Dinitrotoluene
GC/MS	EPA 8270C/8270D/625/625.1	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D/625/625.1	2-Chlorophenol
GC/MS	EPA 8270C/8270D/625/625.1	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D/625/625.1	2-Methylphenol
GC/MS	EPA 8270C/8270D/625/625.1	2-Nitroaniline
GC/MS	EPA 8270C/8270D/625/625.1	2-Nitrophenol
GC/MS	EPA 8270C/8270D/625/625.1	3 & 4 Methylphenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D/625/625.1	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D/625/625.1	3-Nitroaniline
GC/MS	EPA 8270C/8270D/625/625.1	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D/625/625.1	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D/625/625.1	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D/625/625.1	4-Chloroaniline
GC/MS	EPA 8270C/8270D/625/625.1	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D/625/625.1	4-Nitroaniline
GC/MS	EPA 8270C/8270D/625/625.1	4-Nitrophenol
GC/MS	EPA 8270C/8270D/625/625.1	Acenaphthene
GC/MS	EPA 8270C/8270D/625/625.1	Acenaphthylene
GC/MS	EPA 8270C/8270D/625/625.1	Aniline
GC/MS	EPA 8270C/8270D/625/625.1	Anthracene
GC/MS	EPA 8270C/8270D/625/625.1	1,2-Diphenylhydrazine as Azobenzene
GC/MS	EPA 8270C/8270D/625/625.1	Benzo[a]anthracene
GC/MS	EPA 8270C/8270D/625/625.1	Benzo[a]pyrene
GC/MS	EPA 8270C/8270D/625/625.1	Benzo[b]fluoranthene
GC/MS	EPA 8270C/8270D/625/625.1	Benzo[g,h,i]perylene
GC/MS	EPA 8270C/8270D/625/625.1	Benzo[k]fluoranthene
GC/MS	EPA 8270C/8270D/625/625.1	Benzoic acid
GC/MS	EPA 8270C/8270D/625/625.1	Benzyl alcohol
GC/MS	EPA 8270C/8270D/625/625.1	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/8270D/625/625.1	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/8270D/625/625.1	Bis(2-ethylhexyl) phthalate
GC/MS	EPA 8270C/8270D/625/625.1	Butyl benzyl phthalate
GC/MS	EPA 8270C/8270D/625/625.1	Carbazole
GC/MS	EPA 8270C/8270D/625/625.1	Chrysene
GC/MS	EPA 8270C/8270D/625/625.1	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D/625/625.1	Dibenzofuran
GC/MS	EPA 8270C/8270D/625/625.1	Diethyl phthalate
GC/MS	EPA 8270C/8270D/625/625.1	Dimethyl phthalate
GC/MS	EPA 8270C/8270D/625/625.1	Di-n-butyl phthalate
GC/MS	EPA 8270C/8270D/625/625.1	Di-n-octyl phthalate
GC/MS	EPA 8270C/8270D/625/625.1	Fluoranthene
GC/MS	EPA 8270C/8270D/625/625.1	Fluorene
GC/MS	EPA 8270C/8270D/625/625.1	Hexachlorobenzene
GC/MS	EPA 8270C/8270D/625/625.1	Hexachlorobutadiene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D/625/625.1	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D/625/625.1	Hexachloroethane
GC/MS	EPA 8270C/8270D/625/625.1	Indeno[1,2,3-cd]pyrene
GC/MS	EPA 8270C/8270D/625/625.1	Isophorone
GC/MS	EPA 8270C/8270D/625/625.1	Naphthalene
GC/MS	EPA 8270C/8270D/625/625.1	Nitrobenzene
GC/MS	EPA 8270C/8270D/625/625.1	N-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D/625/625.1	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D/625/625.1	N-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D/625/625.1	Pentachlorophenol
GC/MS	EPA 8270C/8270D/625/625.1	Phenanthrene
GC/MS	EPA 8270C/8270D/625/625.1	Phenol
GC/MS	EPA 8270C/8270D/625/625.1	Pyrene
GC/MS	EPA 8270C/8270D/625/625.1	Pyridine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	1,3-Dinitrobenzene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	1,4-Dioxane
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,4,6-Trichlorophenol
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,4-Dinitrotoluene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,6-Dinitrotoluene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Acenaphthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Acenaphthylene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Anthracene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[a]anthracene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[a]pyrene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[b]fluoranthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[g,h,i]perylene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[k]fluoranthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Bis(2-chloroethyl)ether
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Chrysene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Fluoranthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Fluorene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachlorobenzene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachlorobutadiene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachlorocyclopentadiene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachloroethane
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Indeno[1,2,3-cd]pyrene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Naphthalene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Nitrobenzene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	N-Nitrosodimethylamine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	N-Nitrosodi-n-propylamine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Pentachlorophenol
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Phenanthrene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Pyrene
GC-ECD	EPA 8011/504.1	1,2-Dibromoethane
GC-ECD	EPA 8011/504.1	1,2-Dibromo-3-Chloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8011/504.1	1,2,3-Trichloropropane
GC-ECD	EPA 8081A/8081B/608/608.3	2,4’-DDD
GC-ECD	EPA 8081A/8081B/608/608.3	2,4’-DDE
GC-ECD	EPA 8081A/8081B/608/608.3	2,4’-DDT
GC-ECD	EPA 8081A/8081B/608/608.3	4,4'-DDD
GC-ECD	EPA 8081A/8081B/608/608.3	4,4'-DDE
GC-ECD	EPA 8081A/8081B/608/608.3	4,4'-DDT
GC-ECD	EPA 8081A/8081B/608/608.3	Aldrin
GC-ECD	EPA 8081A/8081B/608/608.3	alpha-BHC
GC-ECD	EPA 8081A/8081B/608/608.3	alpha-Chlordane
GC-ECD	EPA 8081A/8081B/608/608.3	beta-BHC
GC-ECD	EPA 8081A/8081B/608/608.3	Cis-Nonachlor
GC-ECD	EPA 8081A/8081B/608/608.3	delta-BHC
GC-ECD	EPA 8081A/8081B/608/608.3	Dieldrin
GC-ECD	EPA 8081A/8081B/608/608.3	Endosulfan I
GC-ECD	EPA 8081A/8081B/608/608.3	Endosulfan II
GC-ECD	EPA 8081A/8081B/608/608.3	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B/608/608.3	Endrin
GC-ECD	EPA 8081A/8081B/608/608.3	Endrin aldehyde
GC-ECD	EPA 8081A/8081B/608/608.3	Endrin ketone
GC-ECD	EPA 8081A/8081B/608/608.3	gamma-BHC (Lindane)
GC-ECD	EPA 8081A/8081B/608/608.3	gamma-Chlordane
GC-ECD	EPA 8081A/8081B/608/608.3	Heptachlor
GC-ECD	EPA 8081A/8081B/608/608.3	Heptachlor epoxide
GC-ECD	EPA 8081A/8081B/608/608.3	Hexachlorobenzene
GC-ECD	EPA 8081A/8081B/608/608.3	Hexachlorobutadiene
GC-ECD	EPA 8081A/8081B/608/608.3	Methoxychlor
GC-ECD	EPA 8081A/8081B/608/608.3	Mirex
GC-ECD	EPA 8081A/8081B/608/608.3	Oxy-Chlordane
GC-ECD	EPA 8081A/8081B/608/608.3	Technical Chlordane
GC-ECD	EPA 8081A/8081B/608/608.3	Toxaphene
GC-ECD	EPA 8081A/8081B/608/608.3	Trans-Nonachlor
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1016
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1221
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1232
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1242
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1248

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1254
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1260
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1262
GC-ECD	EPA 8082/8082A/608/608.3	PCB-1268
GC-IT/MS	EPA 8151A MOD	2,4,5-T
GC-IT/MS	EPA 8151A MOD	2,4-D
GC-IT/MS	EPA 8151A MOD	2,4-DB
GC-IT/MS	EPA 8151A MOD	4-Nitrophenol
GC-IT/MS	EPA 8151A MOD	Dalapon
GC-IT/MS	EPA 8151A MOD	Dicamba
GC-IT/MS	EPA 8151A MOD	Dichlorprop
GC-IT/MS	EPA 8151A MOD	Dinoseb
GC-IT/MS	EPA 8151A MOD	MCPA
GC-IT/MS	EPA 8151A MOD	Mecoprop
GC-IT/MS	EPA 8151A MOD	Pentachlorophenol
GC-IT/MS	EPA 8151A MOD	Silvex (2,4,5-TP)
GC-FID	EPA 8015B	Gasoline
GC-FID	AK101	Gasoline
GC-FID	NWTPH-Gx	Gasoline
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C5-C6)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C8-C10)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C10-C12)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aromatic HCs >C8-C10)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aromatic HCs >C10-C12)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aromatic HCs >C12-C13)
GC-FID	EPA 8015B	Diesel
GC-FID	AK102	Diesel
GC-FID	NWTPH-Dx	Diesel
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C8->C10)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C10->C12)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C12->C16)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C16->C21)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C21->C34)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C8->C10)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C10->C12)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C12->C16)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C16->C21)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C21->C34)
GC-FID	EPA 8015B	Motor Oil
GC-FID	AK103	Motor Oil
GC-FID	NWTPH-Dx	Motor Oil
Titration	EPA 310.1 / SM 2320B	Alkalinity
Colorimetric / RFA	EPA 353.2	Nitrate
Colorimetric / RFA	EPA 353.2	Nitrite
Colorimetric / RFA	EPA 353.2	Nitrate + Nitrite
Probe	EPA 405.1 / SM 5210B	BOD
Titration	EPA 410.2 SM 5220C	COD
Colorimetric / RFA	SM 5220D 21st Ed	COD
Gravimetric	EPA 1664A	Oil & Grease
Colorimetric/RFA	EPA 9012A	Total Cyanides
Colorimetric	EPA 7196A	Hexavalent Chromium
Ion Chromatography	EPA 300.0/9056A	Bromide
Ion Chromatography	EPA 300.0/9056A	Chloride
Ion Chromatography	EPA 300.0/9056A	Fluoride
Ion Chromatography	EPA 300.0/9056A	Sulfate
Ion Chromatography	EPA 300.0/9056A	Nitrate
Ion Chromatography	EPA 300.0/9056A	Nitrite
TOC Analyzer (IR)	EPA 415.1/9060/9060A	TOC
Probe	EPA 9040B/9045C/150.1	pH

Non-Potable Water		
Technology	**Method**	**Analyte**
Conductivity meter	EPA 9050A/120.1 SM 2510B	Specific Conductance
Setaflash	EPA 1020A	Flashpoint
Preparation	**Method**	**Type**
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Nonvolatile Organics
Continuous Liquid-Liquid Extraction	EPA 3520C	Semivolatile and Nonvolatile Organics
Purge and Trap	EPA 5030B	Volatile Organic Compounds
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure
Florisil Cleanup	EPA 3620B	Cleanup of pesticide residues and other chlorinated hydrocarbons
Silica Gel Cleanup	EPA 3630C	Column Cleanup
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup Reagent
Sulfuric Acid Cleanup	EPA 3665A	Cleanup for Quantization of PCBs

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Silver
ICP-AES	EPA 6010B/6010C/6010D	Aluminum
ICP-AES	EPA 6010B/6010C/6010D	Arsenic
ICP-AES	EPA 6010B/6010C/6010D	Boron
ICP-AES	EPA 6010B/6010C/6010D	Barium
ICP-AES	EPA 6010B/6010C/6010D	Beryllium
ICP-AES	EPA 6010B/6010C/6010D	Calcium
ICP-AES	EPA 6010B/6010C/6010D	Cadmium
ICP-AES	EPA 6010B/6010C/6010D	Cobalt
ICP-AES	EPA 6010B/6010C/6010D	Chromium
ICP-AES	EPA 6010B/6010C/6010D	Copper
ICP-AES	EPA 6010B/6010C/6010D	Iron
ICP-AES	EPA 6010B/6010C/6010D	Potassium
ICP-AES	EPA 6010B/6010C/6010D	Magnesium
ICP-AES	EPA 6010B/6010C/6010D	Manganese
ICP-AES	EPA 6010B/6010C/6010D	Molybdenum

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Sodium
ICP-AES	EPA 6010B/6010C/6010D	Nickel
ICP-AES	EPA 6010B/6010C/6010D	Lead
ICP-AES	EPA 6010B/6010C/6010D	Antimony
ICP-AES	EPA 6010B/6010C/6010D	Selenium
ICP-AES	EPA 6010B/6010C/6010D	Silicon
ICP-AES	EPA 6010B/6010C/6010D	Tin
ICP-AES	EPA 6010B/6010C/6010D	Titanium
ICP-AES	EPA 6010B/6010C/6010D	Strontium
ICP-AES	EPA 6010B/6010C/6010D	Thallium
ICP-AES	EPA 6010B/6010C/6010D	Vanadium
ICP-AES	EPA 6010B/6010C/6010D	Zinc
ICP-MS	EPA 6020/6020A/6020B	Silver
ICP-MS	EPA 6020/6020A/6020B	Aluminum
ICP-MS	EPA 6020/6020A/6020B	Arsenic
ICP-MS	EPA 6020/6020A/6020B	Barium
ICP-MS	EPA 6020/6020A/6020B	Beryllium
ICP-MS	EPA 6020/6020A/6020B	Cadmium
ICP-MS	EPA 6020/6020A/6020B	Cobalt
ICP-MS	EPA 6020/6020A/6020B	Chromium
ICP-MS	EPA 6020/6020A/6020B	Copper
ICP-MS	EPA 6020/6020A/6020B	Iron
ICP-MS	EPA 6020/6020A/6020B	Manganese
ICP-MS	EPA 6020/6020A/6020B	Molybdenum
ICP-MS	EPA 6020/6020A/6020B	Nickel
ICP-MS	EPA 6020/6020A/6020B	Lead
ICP-MS	EPA 6020/6020A/6020B	Antimony
ICP-MS	EPA 6020/6020A/6020B	Selenium
ICP-MS	EPA 6020/6020A/6020B	Thallium
ICP-MS	EPA 6020/6020A/6020B	Uranium
ICP-MS	EPA 6020/6020A/6020B	Vanadium
ICP-MS	EPA 6020/6020A/6020B	Zinc
CVAAS	EPA 7471A	Mercury
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-Chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Chloroethylvinylether
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Acetonitrile
GC/MS	EPA 8260B/8260C	Acrolein
GC/MS	EPA 8260B/8260C	Acrylonitrile
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon disulfide
GC/MS	EPA 8260B/8260C	Carbon tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chlorobromomethane
GC/MS	EPA 8260B/8260C	Chlorodibromomethane
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylene Dibromide
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Isopropylbenzene
GC/MS	EPA 8260B/8260C	Methyl Ethyl Ketone
GC/MS	EPA 8260B/8260C	Methyl Isobutyl Ketone
GC/MS	EPA 8260B/8260C	Methyl tert-butyl ether
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	m-Xylene & p-Xylene
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	N-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl chloride
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1,1,2-Tetrachloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,2,4-Trimethylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1, 2-Dibromoethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,1-Dichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,2-Dichloroethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,3,5-Trimethylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	2-Hexanone
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Benzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Bromoform
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Bromomethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Butadiene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Chlorodibromomethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Chloroform
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	cis-1,2-Dichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	cis-1,3-Dichloropropene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Dibromomethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Ethylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Bromodichloromethane
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Ethylene Dibromide
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Hexachlorobutadiene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Isopropylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Isopropyl alcohol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	m&p-Xylene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Methyl tert-Butyl Ether
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Naphthalene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	n-Butylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	o-Xylene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Sec-Butylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Tert-Butylbenzene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Tetrachloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Toluene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Trans-1,2-Dichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	trans-1,3-Dichloropropene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Trichloroethene
GC/MS SIM	EPA 8260B SIM EPA 8260C SIM	Vinyl chloride
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	bis(2-chloroisopropyl)ether
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3 & 4 Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine as Azobenzene
GC/MS	EPA 8270C/8270D	Benzo[a]anthracene
GC/MS	EPA 8270C/8270D	Benzo[a]pyrene
GC/MS	EPA 8270C/8270D	Benzo[b]fluoranthene
GC/MS	EPA 8270C/8270D	Benzo[g,h,i]perylene
GC/MS	EPA 8270C/8270D	Benzo[k]fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic acid
GC/MS	EPA 8270C/8270D	Benzyl alcohol
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/8270D	Bis(2-ethylhexyl) phthalate
GC/MS	EPA 8270C/8270D	Butyl benzyl phthalate
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Diethyl phthalate
GC/MS	EPA 8270C/8270D	Dimethyl phthalate
GC/MS	EPA 8270C/8270D	Di-n-butyl phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno[1,2,3-cd]pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	1,3-Dinitrobenzene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	1,4-Dioxane
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,4,6-Trichlorophenol
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,4-Dinitrophenol
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,4-Dinitrotoluene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	2,6-Dinitrotoluene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	3,3'-Dichlorobenzidine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	4-Chloroaniline
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Acenaphthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Acenaphthylene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Anthracene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[a]anthracene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[a]pyrene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[b]fluoranthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[g,h,i]perylene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Benzo[k]fluoranthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Bis(2-chloroethyl)ether
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Chrysene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Fluoranthene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Fluorene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachlorobenzene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachlorobutadiene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachlorocyclopentadiene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Hexachloroethane
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Indeno[1,2,3-cd]pyrene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Naphthalene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Nitrobenzene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	N-Nitrosodimethylamine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	N-Nitrosodi-n-propylamine
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Pentachlorophenol
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Phenanthrene
GC/MS SIM	EPA 8270C SIM EPA 8270D SIM	Pyrene
GC-ECD	EPA 8011	1,2-Dibromoethane
GC-ECD	EPA 8011	1,2-Dibromo-3-Chloropropane
GC-ECD	EPA 8011	1,2,3-Trichloropropane
GC-ECD	EPA 8081A/8081B	2,4'-DDD
GC-ECD	EPA 8081A/8081B	2,4'-DDE
GC-ECD	EPA 8081A/8081B	2,4'-DDT
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	alpha-BHC
GC-ECD	EPA 8081A/8081B	alpha-Chlordane
GC-ECD	EPA 8081A/8081B	beta-BHC
GC-ECD	EPA 8081A/8081B	delta-BHC
GC-ECD	EPA 8081A/8081B	Cis-Nonchlor
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin aldehyde
GC-ECD	EPA 8081A/8081B	Endrin ketone
GC-ECD	EPA 8081A/8081B	gamma-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	gamma-Chlordane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor epoxide
GC-ECD	EPA 8081A/8081B	Hexachlorobenzene
GC-ECD	EPA 8081A/8081B	Hexachlorobutadiene
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Mirex
GC-ECD	EPA 8081A/8081B	Oxy-Chlordane
GC-ECD	EPA 8081A/8081B	Technical Chlordane
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Trans-Nonachlor
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC-IT/MS	EPA 8151A MOD	2,4,5-T
GC-IT/MS	EPA 8151A MOD	2,4-D
GC-IT/MS	EPA 8151A MOD	2,4-DB
GC-IT/MS	EPA 8151A MOD	4-Nitrophenol
GC-IT/MS	EPA 8151A MOD	Dalapon
GC-IT/MS	EPA 8151A MOD	Dicamba
GC-IT/MS	EPA 8151A MOD	Dichlorprop
GC-IT/MS	EPA 8151A MOD	Dinoseb
GC-IT/MS	EPA 8151A MOD	MCPA
GC-IT/MS	EPA 8151A MOD	Mecoprop MCPP
GC-IT/MS	EPA 8151A MOD	Pentachlorophenol
GC-IT/MS	EPA 8151A MOD	Silvex (2,4,5-TP)
GC-FID	EPA 8015B	Gasoline
GC-FID	AK101	Gasoline
GC-FID	NWTPH-Gx	Gasoline
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C5-C6)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C6-C8)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C8-C10)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aliphatic HCs >C10-C12)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aromatic HCs >C8-C10)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aromatic HCs >C10-C12)
GC-FID	NWVPH	Volatile Petroleum Hydrocarbons (aromatic HCs >C12-C13)
GC-FID	EPA 8015B	Diesel
GC-FID	AK102	Diesel
GC-FID	NWTPH-Dx	Diesel
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C8->C10)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C10->C12)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C12->C16)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C16->C21)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aliphatic C21->C34)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic -8->C10)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C10->C12)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C12->C16)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C16->C21)
GC-FID	NWEPH	Extractable Petroleum Hydrocarbons (aromatic C21->C34)
GC-FID	EPA 8015B	Motor Oil
GC-FID	AK103	Motor Oil
GC-FID	NWTPH-Dx	Motor Oil
Colorimetric/RFA	EPA 9012A	Total Cyanides
Ion Chromatography	EPA 300.0/9056A	Bromide

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Ion Chromatography	EPA 300.0/9056A	Chloride
Ion Chromatography	EPA 300.0/9056A	Fluoride
Ion Chromatography	EPA 300.0/9056A	Sulfate
Ion Chromatography	EPA 300.0/9056A	Nitrate
Ion Chromatography	EPA 300.0/9056A	Nitrite
TOC Analyzer (IR)	EPA 9060/9060A	TOC
Probe	EPA 9040B/9045C	pH/Corrosivity
Conductivity meter	EPA 9050A	Specific Conductance
Setaflash	EPA 1020A	Flashpoint
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Nonvolatile Organics in leachates
Continuous Liquid-Liquid Extraction	EPA 3520C	Semivolatile and Nonvolatile Organics in leachates
Microwave Extraction	EPA 3546	Semivolatile and Nonvolatile Organics
Ultrasonic Extraction	EPA 3550B	Semivolatile and Nonvolatile Organics
Solvent Dilution	EPA 3580A	Semivolatile and Nonvolatile Organics
Waste Dilution	EPA 3585	Volatile Organic Compounds
Purge and Trap	EPA 5030B	Volatile Organic Compounds
Purge and Trap	EPA 5035A	Volatile Organic Compounds
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics in leachates
Acid Digestion (Sediments, Sludges, Soils)	EPA 3050B	Inorganics
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure
Florisil Cleanup	EPA 3620B	Cleanup of pesticide residues and other chlorinated hydrocarbons
Silica Gel Cleanup	EPA 3630C	Column Cleanup
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup Reagent
Sulfuric Acid Cleanup	EPA 3665A	Cleanup for Quantitation of PCBs

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2236.

R.D.R
Vice President

MicroVision Laboratories, Inc

Laboratory Accreditation

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

MicroVision Labs, Inc.

187 Billerica Road, Chelmsford, MA 01824

(Hereinafter called the Organization) and hereby declares that Organization is accredited in accordance with the recognized International Standard:

ISO/IEC 17025:2017

This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system
(as outlined by the joint ISO-ILAC-IAF Communiqué dated April 2017):

Analytical Microscopy and Chemical Testing

(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Tracy Szerszen
President/Operations Manager

Perry Johnson Laboratory Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
June 29, 2019	June 29, 2019	August 31, 2021

Accreditation No.:	*Certificate No.:*
98218	L19-320

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Certificate of Accreditation: Supplement

MicroVision Labs, Inc.
187 Billerica Road, Chelmsford, MA 01824
Contact Name: John Knowles Phone: 978-250-9909

Accreditation is granted to the facility to perform the following testing:

FIELD OF TEST	ITEMS, MATERIALS OR PRODUCTS TESTED	SPECIFIC TESTS OR PROPERTIES MEASURED	SPECIFICATION, STANDARD METHOD OR TECHNIQUE USED	RANGE (WHERE APPROPRIATE) AND DETECTION LIMIT
Analytical Microscopy [F]	Particles, Nanoscale Particles, Bulk Materials, Biomedical Products, Semiconductors, MEMS Devices, Membranes	SEM, Morphology, and Dimensions	ASTM E766, MVL 01, MVL 05, MVL 07	15 X to 50 000 X
	Particles, Dust, Fibers, Soot, Coal, Coal Ash	LM, PLM, Morphology, and Optical Properties	ASTM PDF-STP47926S, MVL 02, MVL 05, MVL 07	40 X to 400 X
Chemical [F]	Particles, Bulk Materials, Coal, Coal Ash, Lead Paint, Soot, Slag	EDS, Semi-Quantitative Elemental Analysis, Compositional Analysis and Elemental Maps	ASTM E1508, MVL 05, MVL 07	0.1 % to 1 %
	Polymers, Oils, Grease, Organic Compounds, Membranes	Micro-FTIR	ASTM E1252, MVL 07, MVL 21	Qualitative

1. The presence of a superscript F means that the laboratory performs testing of the indicated parameter at its fixed location. Example: Outside Micrometer [F] would mean that the laboratory performs this testing at its fixed location.

APPENDIX D

Laboratory SOPs

Eurofins Test America SOPs

Corpus Christi

TITLE: TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES)

EPA SW-846 METHODS 6010B and 200.7

Approvals (Signature/Date):

John Mathewson — 8/1/18
John Mathewson — Date
Inorganic Department Manager

John Mathewson — 8/1/18
J — Date
Environmental Health & Safety Coordinator

Tina M. Godines — 7/31/18
Christina Godines — Date
Quality Assurance Manager

Chip Meador — 8/6/18
Chip Meador — Date
Laboratory Director

Facility Distribution No. Intranet **Distributed To:**

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) provides guidance for the analysis of trace metals by inductively coupled plasma atomic emission spectrometry (ICP-AES).

1.2 This SOP is based on EPA SW-846 Method 6010B and EPA Method 200.7.

1.3 Applicable matrices for Method 6010B include water, waste, soil, sludge, sediment, and TCLP extracts. Applicable matrices for Method 200.7 include water and waste.

1.4 The detection limits, sensitivity, and optimum operating ranges for each metal will vary with the sample matrix and ICP instrument model. The typical reporting limits for this method are listed in table 1.

2.0 SUMMARY

2.1 The laboratory uses simultaneous ICP-AES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.

2.2 Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photo-multiplier tubes or a charge injection device (CID). The photo-currents from the photo-multiplier tubes or a charge injection device (CID) are processed and controlled by a computer system.

2.3 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

3.0 DEFINITIONS

3.1 Dual View ICP – an ICP equipped with both radial and axial viewing capabilities.

3.2 Dissolved Metals - Those elements which pass through a 0.45-μm membrane. (The sample is acidified after filtration).

3.3 Suspended Metals - Those elements which are retained by a 0.45-μm membrane.

3.4 Total Metals - The concentration determined on an unfiltered sample following vigorous digestion.

3.5 Refer to EPA Methods 200.7 and 6010B of SW-846 for definitions of terms used in this SOP.

3.6 TALS – TestAmerica Laboratory System

3.7 NCM – NonConformance Memo, generated in the TALS

4.0 INTERFERENCES

4.1 Interferences: Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by the following:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1 A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result. Refer to Section 3.0 of EPA Method 6010B for additional information on interferences.

4.1.2 Spectral Interferences: Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte signal. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections must be applied to the analyte to compensate for the effects of these unwanted emissions.

4.1.2.1 When running a single-element interferent standard at the upper concentration range, the absolute value of any result for non-spiked elements that exceeds two times the reporting limit requires investigation and corrective action.

4.1.3 Physical Interference: An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation

and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If internal standard recoveries are not acceptable, then dilution of the sample may be necessary to overcome the interferences. Where the use of an internal standard might actually degrade the accuracy of the analytical result, sample results may be reported without IS correction.

4.1.4 Chemical Interferences: Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.1.5 Memory Interference: Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. Flushing the system with a rinse blank between samples can minimize these effects. The rinse times necessary for a particular element must be estimated prior to analysis. Until the required rinse time is established, the method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

4.1.6 High Salt Concentrations: High salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 80-120% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measureable analyte, over correction could go undetected if a negative value is reported as zero.

4.2 Comments and Helpful Hints: Routine instrument maintenance and good glassware cleaning procedures can prevent many operational problems and interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Method-Specific Safety

5.2.1 All standards and samples contain nitric acid. Lab coats, gloves and safety glasses should be worn while handling standards and samples.

5.2.2 The instrument uses an inductively coupled plasma atomic emission spectrometer. The plasma generates a very bright and intense light that is harmful to the eyes. The instrument is equipped with a protective glass to contain this light and should be in place during instrument operation.

5.2.3 The ICP plasma emits strong UV rays and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.3 Primary Material Used

The following is a list of the materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 The instrument used for analysis is an inductively coupled plasma atomic emission spectrometer that is equipped with background correction and operated by a dedicated workstation under the control of appropriate software. The software should provide for background compensation, system diagnostics, data storage, and calculations. Thermo Fischer ICP 6500E Trace Analyzer IS currently used. Instruments with demonstrated equivalent performance can also be used.

6.1.2 In addition to the ICP, a radio frequency generator and argon gas supply (welding grade or better) are required.

6.2 Laboratory Materials

6.2.1 Adjustable automatic pipettes

6.2.2 Volumetric flasks, 100mL

6.2.3 Bottles, for storing sample digestates, 150mL

7.0 REAGENTS AND STANDARDS

7.1 Shelf-Life: Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.1.1 The expiration date of intermediate concentration standards or working standards cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.

7.1.2 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be replaced immediately

7.1.3 Standards used for calibration and quality control purposes must be NIST traceable, where available.

7.1.4 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon, polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the TALS database.

7.1.5 Calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 9.16 of this procedure.

7.2 Reagents

7.2.1 DI water

7.2.2 Hydrochloric acid, concentrated

- Life of reagent: 1 year from receipt date
- Storage requirements: store at room temperature

7.2.3 Hydrochloric acid, 1:1 in reagent water

- Life of reagent: six months
- Storage requirements: store at room temperature

7.2.4 Nitric acid, concentrated

- Life of reagent: manufacturer's expiration date
- Storage requirements: store at room temperature

7.2.5 Nitric acid, 1:1 in reagent water

- Life of reagent: six months
- Storage requirements: store at room temperature

7.3 Standards

7.3.1 Stock Standard Solutions: Standard stock solutions are purchased from various vendors. Follow manufacturer's expiration date and storage requirements.

7.3.2 Mixed Calibration Standard Solutions: Prepare calibration standards at the same acid concentration as the calibration blank. Calibration standards are standard solutions containing mixtures of compatible elements that may be purchased and used for calibration. The calibration standard must be initially verified using a quality control sample and monitored weekly for stability. Prepare fresh calibration standards as needed.

7.3.3 Continuing Calibration Verification Standard: The continuing calibration verification (CCV) should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve.

7.3.4 Initial Calibration Verification Standard: The initial calibration verification (ICV) stock standards are purchased from a different vendor than is used to supply the initial calibration standards (ICAL). The ICV is analyzed immediately after the ICAL.

7.3.5 Interference Check Solution (ICSA/ICSAB): The interference check solution is obtained commercially from an outside source.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample Collection, Preservation, Storage and Holding Times: Refer to Chapter 3 and Table 3-2 of EPA SW-846 revision 4, February 2007 for additional information on sample collection preservation, storage and holding times. TestAmerica Corpus Christi supplies sample containers and chemical preservatives in accordance with the method. TestAmerica Corpus Christi does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures.

	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	500 mLs	HNO_3, pH < 2; Cool 4 ± 2°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

The exception is the analysis of dissolved silica by Method 200.7, which must be analyzed within 28 days from the date of collection.

Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.1.1 The samples are to be collected in 1-L plastic or glass containers that have been cleaned according to EPA specifications. A minimum volume of 500 mL should be collected for aqueous samples. Preserve aqueous samples with nitric acid to a **pH of <2** with the exception of samples that are to be extracted (i.e. EP Toxicity or TCLP). After extraction, the EP Toxicity or TCLP sample extracts are preserved with nitric to a pH of <2. The maximum holding time for preserved samples is 6 months from the date of sampling.

8.1.2 **Dissolved metals**: If samples are to be analyzed for dissolved metals, the samples must be field filtered within 15 minutes of collection and before adding preservatives. Rinse and condition the filter with 50 to 100mL of sample, discard this portion of filtrate, and then collect 500mL of sample. Acidify the collected sample filtrate with HNO_3 to a pH of ≤ 2.

8.1.3 If the samples are to be analyzed for dissolved metals and are not field filtered, the sample must be filtered through a 0.45um filter as soon as possible upon laboratory receipt. The filtered sample will then be preserved to a pH < 2 with HNO_3. The 24 hour wait period after addition of acid is not necessary in this case (for sample received unfiltered). However, an NCM must be filled out noting that the sample was not filtered at the field.

8.1.3.1 Filtration for dissolved metals may be done using a membrane filter of a different pore size as defined in project-specific requirements.

8.1.4 **Total metals**: An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, the acid must have been added immediately within 15 minutes of collection. If for some reason such as high alkalinity the sample pH is verified to be > 2, more acid must be added and the sample held for another 24 hours until verified to be pH < 2. The pH of all aqueous samples **must** be tested prior to digestion to ensure that the sample has been properly preserved. For samples received unpreserved, an NCM must be filled out if the samples are analyzed without the 24 hour hold after adding acid.

9.0 QUALITY CONTROL

All quality control measures described in EPA Method 200.7 Section 9.0, EPA Method 6010B Section 8.0, and Chapter One of SW-846 should be followed.

9.1 Demonstration of Proficiency

9.1.1 Initial Demonstration of Capability (IDOC)

Each laboratory analyst must demonstrate initial demonstration of capability (with each sample preparation and analytical method combination it utilizes) by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. Four replicate aliquots of the reference sample (also referred to as a spiked blank or a laboratory control sample, spiked at 1 to 4 times the reporting limit) containing each target analyte must be initially analyzed by the analyst in order to demonstrate the ability to generate acceptable accuracy and precision data.

9.1.2 Continuing Demonstration of Proficiency (DOC or CDOC)

Annual DOC is also required. Analysts must pass four LCSs, QC study, or proficiency (PT) study and document it yearly. LCS samples from actual test runs or results of a PT or QC sample(s) can be used for this purpose.

9.2 Method Detection Limits: Method detection limits (MDLs) shall be initially determined for all compounds of interest for each method using a clean matrix appropriate to the test method, such as laboratory pure water.

9.2.1 Prepare seven standards at three to five times the estimated MDL concentration.

9.2.2 The MDL standards are processed through the entire analytical process, including the digestion.

9.2.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte (99% confidence level). Then, calculate the MDL for each analyte.

9.3 Detectability Check Sample/ MDL Verification (DCS/MDLV): A Detectability Check Sample (DCS) is a reagent matrix spiked with a known amount of analytes at

approximately two to three times the calculated MDL. This sample used to check the MDL for reasonableness to verify the lab's ability to detect analytes at the MDL used for calculating the PQL/RL (practical quantitation limit/reporting limit). **This sample is prepared at client/agency/program request as needed**. [The DCS may also be known as the MDL verification Sample]. The DCS can be prepared from either Vendor/Source.

9.3.1 The DCS should be taken through all the preparatory and determinative steps to verify that a response is detected. This sample should be analyzed quarterly during the method use and randomly rotated through the instruments so that no instrument is excluded.

Acceptance Criteria: The calculated MDL is verified if the MDLV/DCS standard is detected and the result is significantly different than the blank.

Corrective Actions: If the first MDLV/DCS is not detected, the MDLV/DCS standard will be re-prepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

9.4 Linear Dynamic Range (LDR): The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges that may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file.

9.4.1 LDR Frequency: LDR should be determined every 6 months. By using the high level standard from each ICAL this frequency is met.

9.4.2 LDR Criteria: Recovery within 10% of the true concentration.

9.5 Batch

9.5.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.5.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.5.3 For this analysis, batch QC consists of a Method Blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.6 Initial and Continuing Calibration Blanks (ICB/CCB)

9.6.1 The calibration blank is an aliquot of reagent water prepared with the same type and concentration of acids as the calibration standard(s). Analyze an initial calibration blank (ICB) after the initial calibration verification standard. Analyze

the continuing calibration blanks (CCB) after every continuing calibration standard.

Acceptance Criteria: The absolute value of the ICB/CCB result must be < RL or less than 1/10 the concentration found in the associated samples.

Corrective Action: If the ICB/CCB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

9.6.2 Fill a 4-liter carboy with about 3.5 liters of reagent water. Slowly add the appropriate amount of concentrated HNO_3 and concentrated HCl. Mix carefully.

9.7 Initial Calibration Verification Standard (ICV): The initial calibration verification standard (ICV) is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. Check calibration with an ICV following the initial calibration.

9.7.1 For Method 200.7, the results of the ICV must be within 95-105% of the known value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

9.7.2 For Method 6010B, the results of the ICV must be within 90-110% of the known value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

9.8 Continuing Calibration Verification Standard (CCV): The CCV should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve. The CCV is analyzed immediately following daily calibration, after every 10 samples, and at the end of an analytical run. At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV should be at a concentration near the mid-point of the calibration curve.

9.8.1 The result obtained for each CCV must be within 90-110% of the known value to be in control. If the CCV exceeds these limits, take corrective action by terminating the analysis, correcting the problem, recalibrating the instrument, and reanalyzing the previous 10 analytical runs.

NOTE: The frequency of the CCV applies to all sample runs.

9.9 Method Blank (MB): The blank is de-ionized water taken through the procedure as if it were a sample. A method blank is required with every batch of 20 or less samples. In Method 200.7, this is referred to as a Laboratory Reagent Blank (LRB).

Acceptance Criteria: The method blank must not contain any analytes of interest above the reporting limit or above one-tenth of the concentration found in

the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

9.10 Laboratory Control Sample (LCS): A laboratory control sample (LCS) consists of either a control matrix spiked with analytes which is carried through the sample preparation and analysis procedure as if it were a sample. One LCS is required with each analytical batch. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. The LCS shall be used to determine batch acceptance. In Method 200.7, this is called a Laboratory Fortified Blank (LFB).

Acceptance Criteria: For Method 200.7, the recovery of the LCS must be within 85-115% or tighter.

For Method 6010B, the recovery of the LCS must be within 80-120% or tighter.

In the instance where the LCS recovery is greater than the upper limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the report narrative.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

9.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair is required at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/SDS. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. In Method 200.7, this is referred to as a Laboratory Fortified Matrix (LFM).

9.11.1 Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked.

9.11.2 Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

Acceptance Criteria: The recoveries for the MS and MSD should be within 75-125%. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated).

Corrective Action: If MSD/MSD recoveries fall outside of the established limits and the LCS is in control, the data will be flagged as outside of control

limits. Document the results, which are then used by the lab PM to prepare the case narrative to warn the client that the sample result is suspect.

Acceptance Criteria: The relative percent difference (RPD) between the MS and MSD must be within 20%.

Corrective Action: If the RPD fails to meet precision limit and the recoveries pass, the control limits should be checked as this would be a very rare occurrence if the limits are set properly. If the LCS is in control, it indicates long-term precision, and precision failures within the batch may be due to sample non-homogeneity. MS/MSD results which fall established control limits must be addressed in the narrative. Document the result, which is then used by the lab PM to prepare the case narrative.

9.12 Serial Dilution Test: A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. The test is performed by running a sample at a 5x dilution. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

Acceptance Criteria: If the analyte concentration is sufficiently high (minimally, a factor of 50 times the MDL), an analysis of a 1 to 5 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within ± 10% of the original determination.

Corrective Action: If the two results do not agree within ± 10%, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and an NCM prepared, which is then used by the lab PM to prepare the case narrative to warn the client the sample result is suspect.

9.13 Post Digestion Spike (PDS):

9.13.1 A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process. In Method 200.7, this is called an “analyte addition test.”

9.13.2 The post digestion spike (PDS) determines any potential matrix interferences. The same sample that was used for the serial dilution test should be used for the PDS.

9.13.3 At Corpus Christi laboratory, matrix interference is determined by evaluating data for the LCS and MS/MSD. Therefore, the PDS is not analyzed unless a client project or program requires it.

9.13.4 Recovery for the PDS should be within 75-125% of the expected results. There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with a NCM.

9.14 Method of Standard Additions (MSA): This technique involves constructing a calibration curve in the sample matrix itself to compensate for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.

9.14.1 Attachment 1 provides more guidance on performing MSA analyses.

9.15 Interference Check Analysis (ICSA/ICSAB): The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Analytes are spiked into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run or at the beginning and end of the run depend on project requirements. In Method 200.7, this is referred to as the Spectral Interference Check (SIC).

Acceptance Criteria: The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements with reporting limits $\leq$ 10 µg/L must be $\leq$ 2 x RL. The absolute value of ICSA results for the non-interfering elements with RLs > 10 µg/L must be $\leq$ RL.

Corrective action: If the ICSA results for the non-interfering elements do not meet these limits, the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required, then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in an absolute value > 2 x RL, then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal > 2.x RL, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data do not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

9.16 Monitoring Internal Standard Results: Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

Acceptance Criteria: For Method 200.7, if the internal standard counts fall within ±30% (70-130%) of the counts observed in the ICAL blank (STD1-Blank), then the data are acceptable.

For Method 6010B, if the internal standard counts fall within ±50% (50-150%) of the counts observed in the ICAL blank (STD1-Blank), then the data are acceptable.

Corrective Action: If the internal standard counts in the field samples are outside of the control limits, the following apply:

The field samples must be diluted and reanalyzed;

The IS concentrations must be raised; or

A different internal standard must be used.

9.17 Quality Control Sample (QCS)

The Method 200.7 requirement for a quarterly QCS is satisfied by the analysis of the second-source ICV, which is analyzed with each initial calibration.

9.18 Background Correction Point: To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength of interest and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations.

9.18.1 Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.19 Interelement Corrections (IECs): ICP interelement correction (IEC) factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

9.19.1 When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC

varies significantly from the previously determined IEC, then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs.

9.19.2 Refer to the facility-specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

9.19.3 Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace and CID detector instruments as reflected by the ICSA response.

9.20 Procedural Variations/ Nonconformance and Corrective Action

9.20.1 Any variation shall be completely documented using a NCM and approved by the Supervisor or QA Manager.

9.20.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor or QA Manager.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration Standards (ICAL): Instrument calibration is performed daily or every 24 hours using a minimum of three standards and a calibration blank. The concentrations are:

Calibration Standards					
Element	ICBLK mg/L	IC1 mg/L	IC2 / CCV mg/L	IC3 mg/L	IC4 mg/L
Ag	0	0.0025	1	2	
Al	0	0.025	10	20	100
As	0	0.005	1	2	
B	0	0.0025	1	2	
Ba	0	0.0025	1	2	
Be	0	0.0025	1	2	
Bi	0	0.005	1	2	
Ca	0	0.1	40	80	
Cd	0	0.0025	1	2	
Co	0	0.0025	1	2	
Cr	0	0.0025	1	2	
Cu	0	0.0025	1	2	
Fe	0	0.025	10	20	100
K	0	0.25	100	200	
Li	0	0.005	2	4	
Mg	0	0.1	40	80	
Mn	0	0.025	10	20	
Mo	0	0.0025	1	2	
Na	0	0.025	10	20	500
Ni	0	0.0025	1	2	
P	0	0.005	2	4	
Pb	0	0.005	1	2	
Sb	0	0.05	1	2	
Se	0	0.005	1	2	
Si	0		10	20	
Sn	0	0.005	2	4	
Sr	0	0.0025	1	2	
Ti	0	0.0025	1	2	
Tl	0	0.005	1	2	
V	0	0.0025	1	2	
Zn	0	0.0025	1	2	

10.2 The Low Cal Standard check Solution (CRI): The low level standard is analyzed as required by the programs or projects to demonstrate that the ICP is capable of detecting the target compounds at or below the reporting limit (RL). The determined concentration should be within ± 30% of the true concentration.

10.3 Initial Calibration Verification/Initial Calibration Blank (ICV/ICB): The initial calibration accuracy is verified by analyzing a second source standard (ICV).

10.3.1 ICV Frequency:

10.3.1.1 Perform with each initial calibration

10.3.2 ICV Criteria:

10.3.2.1 The ICV must fall within 5% (Method 200.7) or 10% (Method 6010) of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures.

Elements	ICV Concs mg/L
Ag	0.5
Al	5
As	0.5
B	1
Ba	0.5
Be	0.5
Bi	0.5
Ca	5
Cd	0.5
Co	0.5
Cr	0.5
Cu	0.5
Fe	50
K	50
Li	0.5
Mg	5
Mn	5
Mo	0.5
Na	50
Ni	0.5
P	5
Pb	0.5
Sb	0.5
Se	0.5
Si	10
Sn	0.5
Sr	0.5
Ti	0.5
Tl	0.25
V	0.5
Zn	0.5

10.3.3 ICB Frequency:

- An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.

10.3.4 ICB Criteria:

10.3.4.1 The ICB result must fall within +/- the RL from zero.

10.3.5 If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.

- Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4 Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB)

10.4.1 Calibration is monitored throughout the analytical run through the analysis of a known standard.

10.4.2 A CCV may be a second source or the same source as the calibration

10.4.3 CCV Frequency:

- Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.

10.4.4 CCV Criteria:

- The CCV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures.

- If a CCV has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (ie initial calibration). Samples after the last acceptable CCV require re-analysis.

10.4.5 CCB Frequency:

- A CCB is analyzed immediately following each CCV.

10.4.6 CCB Criteria:

- The CCB result must fall within +/- RL from zero.

10.5 Interference Check Analysis (ICSA/ICSAB)

10.5.1 The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions.

10.5.2 ICSA:

- The ICSA contains only interfering elements.
- Custom multi-element ICS solutions must be used.
- Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

10.5.3 ICSAB:

- The ICSAB contains analytes and interferents.
- Refer to Table II for the details of ICSAB composition.
- Custom multi-element ICS solutions must be used.

10.5.4 ICSA/ICSAB Frequency:

The ICSA and ICSAB must run with each initial calibration or every 12 hours whichever is shorter.

10.5.5 ICSA/ICSAB Criteria:

- The ICSAB results for interferents must fall within 80 – 120% of the true value.
- ICSA results for the non-interfering elements must fall within ± 2x RL from zero.

ICSA & ICSAB Standard	Elements	Concentration mg/L
ICSA Standard	Fe Al, Ca, Mg	100 250
ICSAB Standard	Ba, Be, Co, Cr, Cu, Mn, V Ag, Cd, Ni, Pb, Zn	50 100

- Life of standard: manufacturer's expiration date
- Storage requirements: store at room temperature

10.6 Linear Dynamic Range Verification (LDR): The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges that may be used for the analysis of samples should be judged by the analyst from the resulting data. The data,

calculations and rationale for the choice of range made should be documented and kept on file.

The linear range is determined for each element on the standard list.

10.6.1 LDR Frequency:

- Methods 200.7 and 6010B stipulate LDR check every 6 months. By using the high level standard from each ICAL this frequency is met.

10.6.2 LDR Criteria:

- Recovery within 10% of the true concentration.

10.7 Calibration Sequence

- Instrument warm up
- Profile standard
- Initial calibration (3 standards and a blank)
- ICV
- ICB
- CRI (analyzed as required by programs/projects)
- ICSA*
- ICSB*
- CCV
- CCB
- 10 samples
- CCV
- CCB
- 10 samples
- CCV
- CCB

*If sequence is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed.

11.0 ANALYTICAL PROCEDURE

11.1 Sample Preparation: Prepare the samples according to the appropriate sample preparation procedure (e.g., EPA SW-846 Methods 3010-3050, 200.7 digestion procedure). Groundwater samples that have been pre-filtered and acidified will not need acid digestion. Refer to sample preparation SOPs for the digestion procedures.

11.2 Instrument Operation, Daily Checks, and Calibration:

11.2.1 Instrument Operation

11.2.1.1 Set up the instrument with the proper operating parameters. Background correction and inter-element correction factors must be programmed into the ICP computer and applied to

all analyses. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

11.2.1.2 The analyst should follow the instructions provided by the instrument manufacturer.

11.2.2 Instrument Calibration: Instrument calibration must be performed daily or each time the instrument is set up. Profile and calibrate the ICP system according to the manufacturer's instructions using the mixed calibration standard solutions and the calibration blank. Flush the system between each standard in the sequence. Use the average intensity of multiple exposures for the standardization and sample analysis. The calibration curve for each metal consists of a blank and a minimum of three standards.

11.3 Sample Analysis

11.3.1 Flush the system with the calibration blank solution for at least 1 minute preceding sample analysis. Analyze the calibration verification standard and the calibration blank after each 10 samples.

11.3.2 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established. Dilute and reanalyze samples with yttrium recoveries outside the acceptance limits of 70-130%.

11.3.3 Dilutions of High Levels of Elements of Interest: For Method 200.7, measurements for all target elements must fall within 90% of the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed 90% of the linear range or use an alternate wavelength for which QC data are established. Dilutions must be prepared using the reagent blank solution to maintain the correct acid strength.

11.3.4 Dilutions for High Levels of Interfering Elements: Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted to a level at or below the working range.

12.0 DATA GENERATION AND CALCULATIONS

12.1 Data Generation: The data to be generated and recorded are: data analyzed, analyst, method/SOP number, standard IDs, known standard and spike concentration values, sample volume or weight, dilution factors, and the measured concentrations for samples and QC parameters.

12.2 Calibration Calculations: Calibration calculations are performed automatically by the computer workstation.

12.3 Concentration Calculations: Concentration values are calculated based on the sample volume or weight and the average of the multiple exposures for each sample. Dilution factors must be applied for samples that are diluted. All results should be reported with up to three significant figures.

12.4 Quality Control Calculations

12.4.1 Relative Percent Difference for Duplicates

$$RPD = \frac{|A - B|}{\frac{1}{2}(A + B)} \times 100\% = \frac{|A - B|}{(A + B)} \times 200\%$$

Where: A = concentration of analyte in original sample
B = concentration of analyte in duplicate sample

12.4.2 Percent Recovery of Standards

$$\text{Percent Recovery} = \frac{\text{Analyzed Value}}{\text{Known Value}} \times 100\%$$

12.4.3 Percent Recovery of Matrix Spike

$$\text{Percent Recovery} = \frac{SSC - SC}{SA} \times 100\%$$

Where: SSC = Spiked Sample Concentration
SC = Sample Concentration (previously determined)
SA = Spike Added Concentration

12.5 Sample Calculation

12.5.1 Aqueous Sample

The final concentration for a digested aqueous sample is calculated as follows:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
V1	=	Final volume in liters after sample preparation
V2	=	Initial volume of sample digested in liters

12.5.2 Soil Sample

The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
V	=	Final volume in liters after sample preparation
W	=	Weight in Kg of wet sample digested
S	=	Percent solids/100

NOTE: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

13.0 DATA ASSESSMENT AND CORRECTIVE ACTION

13.1 Data Assessment

13.1.1 The analyst does primary data review at the time of analysis.

13.1.2 The section supervisor, senior analyst, or designee does secondary data review

13.1.3 The section supervisor, project manager, QA manager, or laboratory director does completeness review and approval.

13.2 Corrective Action and Contingencies for Handling Out of Control Data: If any QC acceptance limit is exceeded, notify the section supervisor or QA manager, initiate a nonconformance memo (NCM) and take corrective action as advised.

14.0 DATA REDUCTION AND REPORTING

14.1 Data Reduction: The analytical value is directly transferred from the instrument into TALS. In TALS, the analytical value is converted to the reported value by the application of the appropriate calculation factors to account for the sample preparation, dilution and dry weight.

14.2 Raw Data Entry and Reporting:

14.2.1 All information pertaining to sample preparation and analysis must be entered as it is acquired in the designated bound logbook or computer generated run logs and instrument printout. This includes the test method; analyte(s); matrix; full ID for samples, standards, and QC; reagent IDs, sample volumes and weights (including units); dilution factors; analyst's initials; and date/time of preparation and analysis. All information and data pertinent to the method and analysis should be recorded to facilitate data validation. Any information which is important or may influence the test results should be noted, such as sample

appearance, problems encountered, unusual conditions, reasons for repeated tests or rejected data, etc.

14.2.2 Instrument printouts must be initialed by the instrument operator.

14.2.3 Instrument printouts must be uniquely identified and maintained according to the laboratory's procedure for data storage. The computer generated spreadsheets or graphs should be organized by date and instrument and kept on file.

14.2.4 Any data that does not meet the method/project requirements or QC specifications must be documented in a NCM. Document the nonconformance, the sample(s) and parameter(s) affected, and the corrective action taken. The NCM is used to generate a case narrative to include with the final data report.

15.0 METHOD PERFORMANCE

15.1 Refer to Tables 1-9 of EPA Method 200.7 for performance data.

15.2 Refer to Tables 4-6 of EPA Method 6010B for performance data.

16.0 POLLUTION CONTROL

16.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

17.0 WASTE MANAGEMENT

17.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

17.2 Waste Stream Produced by the Method: All samples and standards are placed into the laboratory acid waste stream for disposal.

18.0 REFERENCES / CROSS-REFERENCES

18.1 U.S. Environmental Protection Agency, Method 200.7, Revision 4.4.

18.2 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 6010B (Revision 2, December 1996).

18.3 CC-QAM, TestAmerica Quality Assurance Manual, current revision

18.4 Associated SOPs, current revisions.

18.4.1 CC-GLO-014, Maintenance and Calibration of Balances and Class I Weights, current revision.

18.4.2 CC-GLO-015, Thermometer Calibration, current revision.

18.4.3 CC-GLO-016, Calibration and Verification of Pipettes and Dispenser, current revision.

18.4.4 CC-GLO-017, Sample Composition, Homogenization and Subsampling, current revision.

19.0 DOCUMENTATION

19.1 Analysis: The preparation of samples and standards for metals is documented in TALS.

19.2 Traceability of standards: Upon receipt or preparation, each chemical salt, solvent, acid, standard, or other reagent is entered into TALS and issued a unique ID# based upon the type and sequential order in which the item was received. Further information entered into the database includes the manufacturer, lot # (if applicable), date received or prepared, expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., metals ICV solution, CCV, or LCS…). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

20.0 TABLE AND ASSOCIATED DOCUMENTS

Table 1: Analytes and Reporting Limits

Table 2: LCS and MS/MSD Concentrations

Table 3: Summary of quality control requirements

Attachment 1: MSA Guidance

20.1 Associated Documents

20.1.1 Method Detection Limit Study

21.0 REVISION HISTORY

Historical File:	Revision 00: 10/11/02	Revision 01: 09/09/04
	Revision 02: 09/12/06	Revision 03: 08/31/09
	Revision 04: 10/08/10	Revision 05: 01/20/11
	Revision 06: 03/31/15	Revision 07: 05/29/15
	Revision 08: 06/19/17	Revision 09: 08/15/18

21.1 Revision 09, dated xx July 2018, updated by Christina Godines:

21.1.1 Updated sections throughout to merge SOP No. CC-ATM-M016, Method 200.7, into this SOP:

- Added reference to 200.7 in Sections 1.2 and 3.5;
- Added silica exception to Section 8.1;
- Added filter pore size variance in project-specific requirements to Section 8.1.3.1;
- Updated wording in Section 5.1 for Safety and added 5.1.1.3 about UV rays;
- Added QC reference for Method 200.7 in Section 9.0;
- Added LRB, LFB, and LFM references in Sections 9.6, 9.7, and 9.8;
- Added %R and limits specific to 200.7 in Section 9.7;
- Added Sections 7.17, 7.18, and 7.19 from 200.7 SOP;
- Added dilution detail to Sections 11.3.3 and 11.3.4;
- Added 200.7 Tables 1-9 reference in Section 15.0 Method Performance;
- Updated Acceptance Criteria in Table 3;

21.1.2 Added Sections 16.0 Pollution Control and 17.0 Waste Management;

21.1.3 Updated formatting throughout sections to match current SOP format.

21.2 Reasons for changes, Revision 7:

21.2.1 Updated IDOC requirement in Section 7.1.1

21.2.2 Updated acceptance criteria in Section 7.9.8.

Table 1: Reporting Limits for Methods 6010B and 200.7

STANDARD ANALYTE LIST AND TYPICAL REPORTING LIMIT

ELEMENT	Symbol	CAS #	Reporting Limit (mg/L) Water	Reporting Limit (mg/Kg) Soil
Aluminum	Al	7429-90-5	0.100	5.0
Antimony	Sb	7440-36-0	0.005	2.0
Arsenic	As	7440-38-2	0.010	2.0
Barium	Ba	7440-39-3	0.010	1.0
Beryllium	Be	7440-41-7	0.004	0.5
Bismuth	Bi	7740-69-9	0.020	1.0
Boron	B	7440-42-8	0.050	10
Cadmium	Cd	7440-43-9	0.005	0.5
Calcium	Ca	7440-70-2	0.200	50
Chromium	Cr	7440-47-3	0.010	1.0
Cobalt	Co	7440-48-4	0.005	1.0
Copper	Cu	7440-50-8	0.010	2.0
Iron	Fe	7439-89-6	0.200	20
Lead	Pb	7439-92-1	0.010	0.5
Lithium	Li	7439-93-2	0.020	0.5
Magnesium	Mg	7439-95-4	0.200	20
Manganese	Mn	7439-96-5	0.010	2.5
Molybdenum	Mo	7439-98-7	0.010	2.0
Nickel	Ni	7440-02-0	0.010	2.0
Phosphorus	P	7723-14-0	0.500	50
Potassium	K	7440-09-7	0.500	100
Selenium	Se	7782-49-2	0.010	1.0
Silicon	Si	7631-86-9	0.200	20
Silica	SiO2		0.429	42.9
Silver	Ag	7440-22-4	0.005	0.5
Sodium	Na	7440-23-5	1.0	100
Strontium	Sr	7440-24-6	0.005	1.0
Thallium	Tl	7440-28-0	0.005	1.0
Tin	Sn	7440-31-5	0.040	10
Titanium	Ti	7440-32-6	0.005	1.0
Vanadium	V	7440-62-2	0.005	1.0
Zinc	Zn	7440-66-6	0.020	2.5

Note: The analytes' reporting limits are evaluated yearly and may be subject to change.

Table 2: Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (µg/L)	Matrix Spike Level (µg/L)
Aluminum	4,000	4,000
Antimony	400	400
Arsenic	400	400
Barium	400	400
Beryllium	400	400
Bismuth		
Boron	800	800
Cadmium	400	400
Calcium	40,000	40,000
Chromium	400	400
Cobalt	400	400
Copper	400	400
Iron	40,000	40,000
Lead	400	400
Lithium	400	400
Magnesium	40,000	40,000
Manganese	4,000	4,000
Molybdenum	400	400
Nickel	400	400
Phosphorous	4,000	4,000
Potassium	40,000	40,000
Selenium	400	400
Silicon	8,000	8,000
Si (as SiO_2)	17,144	17,144
Silver	400	400
Sodium	40,000	40,000
Strontium	400	400
Thallium	200	200
Tin	400	400
Titanium	400	400
Vanadium	400	400
Zinc	400	400

Table 3: Summary of quality control requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	**6010:** 90 - 110 % recovery. **200.7:** 95 - 105 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.15	See Section 9.15
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.15
Dilution Test	One per prep batch.	For samples > 50x MDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than RL; sample results greater than 10x the blank concentration are acceptable; samples for which the contaminant is < RL may not require redigestion or reanalysis	Rerun once in a new tube. If >RL, re-digest and reanalyze samples. Note exceptions under criteria section.

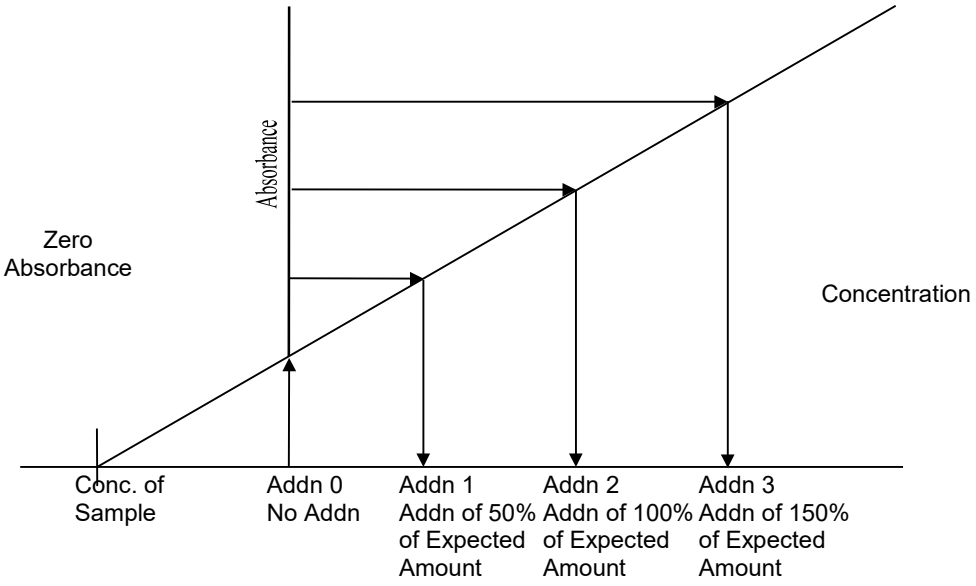
QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	**6010:** 80 – 120 % recovery. **200.7:** 85 – 115 % recovery. Samples for which the contaminant is < RL and the LCS results are > 120% for 6010, or > 115% for 200.7, may not require redigestion or reanalysis	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	Every 10%	75-125% recovery	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	Every 10%	75-125 % recovery; RPD $\leq$ 20%	See Corrective Action for Matrix Spike.

Attachment 1: MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the absolute value of the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear (r=0.995 or greater) over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

TITLE: CATION-EXCHANGE CAPACITY (CEC) OF SOILS

SW-846 Method 9081 and LDNR Method 29-B

Approvals (Signature/Date):

John Mathewson — 5/17/19 John Mathewson — Date Inorganics Department Manager	John Mathewson — 5/17/19 John Mathewson — Date Environmental Health & Safety Coordinator
Tina M. Godines — 5/17/19 Christina Godines — Date Quality Assurance Manager	Chip Meador — 5/20/19 Chip Meador — Date Laboratory Director

Facility Distribution No. Intranet **Distributed To:** _____

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) provides procedure used by Eurofins TestAmerica Corpus Christi for determining the Cation Exchange Capacity of soil samples.

1.2 This SOP is based on SW-846 Method 9081 and LDNR Method 29-B.

1.3 The procedure involves the extraction of the solid sample with first a sodium acetate solution, then with an ammonium acetate solution. The displaced sodium is then measured in the ammonium acetate extract using ICP/ICPMS analysis.

1.4 The typical Reporting limit for this method is 0.05meq/100gm.

2.0 SUMMARY OF METHOD

2.1 The soil is mixed with an excess of sodium acetate solution. This results in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaced the adsorbed sodium with ammonium. The concentration of displaced sodium is then measured in the ammonium acetate extract using ICP/ICPMS analysis.

3.0 DEFINITIONS

3.1 Refer to SW-846 Method 9081 for definitions of terms used in this SOP.

3.2 LDNR – Louisiana Department of Natural Resources.

3.3 TALS - TestAmerica Laboratory System.

3.4 NCM – Non-Conformance Memo. generated in the TALS.

4.0 INTERFERENCES

4.1 See SOP CC-ATM-M005/CC-ATM-M025, current revision, for ICP/ICPMS Analysis by SW-846 Method 6010/6020 or equivalent SOP.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1.1 Method Specific Safety: None

5.1.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Isopropanol alcohol 99%	Flammable	400 ppm-TWA	Flammable liquid and vapor. Harmful if swallowed or inhaled. Causes irritation to eyes and respiratory tract. Affects central nervous system. May be harmful if absorbed through skin. May cause irritation to skin.
Sodium hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or sever burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium acetate	Irritant	None	Do not ingest. Avoid contact with eyes, skin and clothing. Use only with adequate ventilation. Wear appropriate respirator when ventilation is inadequate. Keep in the original container or an approved alternative made from a compatible material, kept tightly closed when not in use. Empty containers retain product residue and can be hazardous. Do not reuse container.
Ammonium acetate	Irritant	None	May cause irritation to skin, eyes, and respiratory tract. May be harmful if swallowed.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Top Loading Balance

6.2 Laboratory Centrifuge

6.3 Mechanical shaker

6.4 Centrifuge tubes: 50 mL

6.5 Digestion tubes with mL markings

6.6 Graduated cylinders, Class A

6.7 Volumetric flasks, Class A

6.8 pH test strips

7.0 REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests.

Shelf-Life: Reagents received from the vendor expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.1 Ammonium Hydroxide (NH_4OH), concentrated

7.2 Glacial acetic acid

7.3 Ammonium Acetate (NH_4OAc), 1N:

7.3.1 Dilute 57 mL of glacial Acetic Acid (99.5%) with reagent water to a volume of approximately 500 mL. Then add 69 mL concentrated Ammonium Hydroxide, mix, and add reagent water to obtain a volume of about 980 mL. Check the pH of the resulting solution; add more NH_4OH, as needed, to obtain a pH of 7 ± 0.05, and dilute the solution to a volume of 1 liter with reagent water. Record the pH.

7.3.2 Bring to the 1 liter mark with DI water. Shake well.

7.4 Sodium Acetate, 1N:

7.4.1 Dissolve 136 g of $NaC_2H_2O_2 \cdot 3H_2O$ (sodium acetate) in about 500 mL reagent water and dilute to 1 liter final volume. The pH of this solution should be 8.2 ± 0.05. If needed, add a few drops of Acetic acid or Sodium Hydroxide (10N) solution to bring the solution to pH 8.2. Record the pH.Check the pH; adjust to 8.2 with acetic acid or NaOH, if needed.

7.4.2 Bring to the 1 liter mark with DI water. Shake well.

7.5 Isopropyl alcohol: 2-propanol 99%

7.6 DI water: Deionized, type I reagent water

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample Collection, Preservation, Storage and Holding Times: Soil samples are collected in plastic or glass 1000 mL sample containers, and stored at 4°C. The holding time for extraction and analysis is 6 months from date of collection.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	HDPE or Glass	20 grams	No requirement	6 months	SW-846 Chapter 2

Temperature preservation is not used.

9.0 QUALITY CONTROL

9.1 Refer to the individual analytical method used for determining sodium for quality controls to be followed.

9.2 Demonstration of Proficiency: Each laboratory analyst must complete initial and ongoing demonstration of capability for each method used. Refer to employee training records.

9.3 Method Detection Limits: Not applicable to this method; refer to method used for determining sodium.

9.4 Batch: A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.5 Instrument QC: Refer to SOPs CC-ATM-M005 for ICP and CC-ATM-M025 for ICP-MS, current revisions.

9.6 Sample QC: The following quality control samples are prepared in each batch:

9.6.1 Method Blank (MB): The blank is de-ionized water taken through the procedure as if it were a sample. A method blank is required with every batch of 20 or less samples. The MB must be less than the Reporting Limit (RL).

9.6.2 Sample Duplicate: One sample duplicate is required at a 10% frequency (one per 10 samples or less).

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Method Blank	One per batch	< RL	Correct problem, then re-prep and reanalyze all samples and QC in the affected analytical batch.
Sample Duplicate	One per batch	<20% RPD	Report.

10.0 PROCEDURE

10.1 Weigh out and label samples

10.1.1 For RCRA Cation Exchange Capacity, weigh 4 to 4.1 g of medium- or fine-textured soil or 6 to 6.1 g of coarse-textured soil and transfer the sample to a 50-mL centrifuge tube. (A fine soil has > 50% of the particles ≤ 0.074 mm, medium soil has >50% ≥ 0.425 mm, while a coarse soil has more than 50% of its particles ≥ 2 mm.) Record the soil weight used.

10.1.2 For LDNR 29-B Cation Exchange Capacity, prepare 5.0 ± 1 grams as described in SOP CC-ATM-WC063, current revision, for LDNR 29-B Soil Preparation.

10.1.3 Label one centrifuge as “prep blank” or “method blank”. This procedure requires one blank per 20 samples and one sample duplicate per 10 samples.

10.2 Sample preparation:

10.2.1 Sodium Acetate

10.2.1.1 Add 33mL of 1N sodium acetate to each centrifuge tube. Cap the tubes. Place on a shaker and shake for 5 minutes. Place the tubes in the centrifuge and spin for about 15 minutes until the supernatant liquid is clear. Decant the liquid. (Do not save).

10.2.1.2 Repeat step 10.2.1.1 three more times.

10.2.2 Isopropyl alcohol (2-propanol)

10.2.2.1 Add 33 mL of 70-90% isopropyl alcohol to each centrifuge tube. Cap the tubes. Place on shaker and shake for 5 minutes. Place the tubes in the centrifuge and spin for about 15 minutes until the supernatant liquid is clear. Decant the liquid. (Do not save).

10.2.2.2 Repeat step 10.2.2.1 two more times.

10.2.3 Ammonium acetate

10.2.3.1 Add 33 mL ammonium acetate to each centrifuge tube. Cap the tubes. Place on shaker and shake for 5 minutes. Place the tubes in the centrifuge and spin for about 15 minutes until the supernatant liquid is clear. **Decant the 1 N ammonium acetate extract into a 100-mL volumetric flask.**

10.2.3.2 Repeat step 10.2.3.1 two more times.

10.2.4 Dilute the combined washing (1N ammonium acetate) to the 100-mL mark with 1N ammonium acetate solution.

10.3 Sample Analysis:

10.3.1 Analyze the batch for sodium (Na) using ICP Method 6010, ICP-MS Method 6020, or equivalent method.

11.0 DATA GENERATION AND CALCULATIONS

11.1 Data Generation

The data to be generated and recorded are the measured concentration values of sodium in units of mg/L.

11.2 Calculation of CEC

$$\frac{\text{Na mg/L}}{23} = \text{meq/L (in the extract)}$$

$$\text{CEC} = \frac{\text{Meq Na x 10}}{\text{Sample wt in gm (4 g)}} = \text{meq / 100g soil}$$

11.3 Calculation of Relative Percent Difference for Duplicates

$$\text{RPD} = \frac{|\,A - B\,|}{\frac{1}{2}(A + B)} \times 100\% = \frac{|\,A - B\,|}{(A + B)} \times 200\%$$

Where: A = concentration of analyte in original sample
B = concentration of analyte in duplicate sample

12.0 DATA ASSESSMENT AND CORRECTIVE ACTION

12.1 Data Assessment

12.1.1 The analyst does primary data review at the time of analysis.

12.1.2 The section supervisor or senior analyst does secondary data review.

12.1.3 The section supervisor, project manager, QA Manager, or Laboratory Director does completeness review and approval.

12.2 Handling Out-of-Control or Unacceptable Data and Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must not be released without approval of the supervisor or QA personnel.

13.0 DATA REDUCTION AND REPORTING

13.1 Data Reduction

The analytical value is entered into TALS manually and the analytical value is converted to the reported value by the application of the calculation in Section 11.2.

13.2 Raw Data Entry and Reporting

13.2.1 Report the results of the calculation as "CEC" in units of meq/ 100gm (milliequivalents per 100 grams). All information pertaining to sample preparation and analysis must be entered as it is acquired on the computer generated run logs, or instrument printout, or designated bound logbook. This includes the test method: analyte(s); matrix; full ID for samples, analyst's initials; and date/time of calculations. All information and data pertinent to the method and/or analysis should be recorded to facilitate data validation. Any information that is important or may influence the test results should be noted, such as sample appearance, problems encountered, unusual conditions, reasons for repeated tests or rejected, etc.

13.2.2 Instrument printouts must be initialed and dated by the instrument operator.

13.2.3 Instrument generated data must be uniquely identified and maintained according to the laboratory's procedure for data storage.

13.2.4 Any data that does not meet the method/project requirements or QC specifications must be documented on a NCM. Document the out of control event or nonconformance, the sample(s) and parameter(s) affected, and the corrective action taken. The NCM is used to generate a case narrative to include with the final data report.

14.0 METHOD PERFORMANCE:

14.1 Refer to method used for performance data.

14.2 Section supervisor/group leader has the responsibility to ensure that this procedure is performed by an associate who has been trained in its use and has the required experience.

15.0 POLLUTION CONTROL

15.1 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

16.0 WASTE MANAGEMENT

16.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

16.2 Waste Stream Produced by the Method: All samples and standards are placed into the laboratory acid waste stream for disposal.

17.0 REFERENCES / CROSS-REFERENCES

17.1 Method 9081, Test Method for Evaluating Solid Waste, USEPA SW-846, 1986.

17.2 Methods 29-B, Laboratory Procedures for Analysis of Exploration & Production Waste, Louisiana Department of Natural Resources, Revision 4, May 2005.

17.3 Corporate Environmental Health and Safety Manual (CW-E-M-001), current revision.

17.4 Eurofins TestAmerica Quality Assurance Manual (CC-QAM), current revision.

17.5 Associated SOPs:

17.5.1 CC-ATM-M005, Trace Metals by ICP-AES (6010B and 200.7), current revision.

17.5.2 CC-ATM-M025, Trace Metals by ICP-MS (6020 and 200.8), current revision.

17.5.3 CC-ATM-WC034, Soil and Waste pH, current revision.

17.5.4 CC-ATM-WC063, Soil Preparation (LDNR 29-B), current revision.

17.5.5 CC-GLO-001, Documentation Procedures, current revision.

17.5.6 CC-GLO-002, General Glassware Cleaning, current revision.

17.5.7 CC-GLO-014, Maintenance and Calibration of Balances and Class I Weights, current revision.

18.0 METHOD MODIFICATIONS:

18.1 To minimize waste generated, the Eurofins TestAmerica Corpus Christi laboratory prepares samples at half the weights/volumes specified in Section 10.0.

19.0 ATTACHMENTS, TABLES, ASSOCIATED DOCUMENTS AND FORMS

19.1 Table 1: Equivalent weights table for major Cations and Anions

19.2 Table 2: Conversion factors for mg/L to meq/L

20.0 REVISION HISTORY

Historical File:

Revision 00: 05/25/04	Revision 01: 08/07/06
Revision 02: 02/26/08	Revision 03: 04/26/09
Revision 04: 06/10/10	Revision 05: 05/27/15

Revision 06: 06/07/17 Revision 07: 01/31/19
Revision 08: 05/20/19

20.1 Revision 08, dated 20 May 2019, updated by Christina Godines:

20.1.1 Updated with rebranding throughout;

20.1.2 Added reference to LDNR 29-B in Sections 1.2, 7.0, 17.0;

20.1.3 Added LDNR to Definitions in Section 3.2;

20.1.4 Updated Sections 7.3 and 7.4 with additional detail;

20.1.5 Added LDNR detail and reformatted Section 10.0;

20.1.6 Updated References to include LDNR and Cross-references to include new soil prep SOP in Section 18.0;

20.1.7 Added method modification in Section 19.0 to use half volumes/weights during sample preparation.

20.2 Revision 07, dated 31 January 2019, updated by Christina Godines:

20.2.1 Removed 'Sample Disposal' from Section 5.0;

20.2.2 Updated wording in Section 6.0 and removed 'Waste Management and Pollution Prevention' (added as Sections 15.0 and 16.0);

20.2.3 Renamed Section 8.0 to 'Equipment and Supplies;'

20.2.4 Added 'Cross-references' to Section 14.0 name and added related SOPs;

20.2.5 Moved Section 15.0 to Section 14.0; renamed15.0 to Method Modifications;

20.2.6 Moved Section 5.0 to Section 8.0;

20.2.7 Moved Section 7.0 to Section 9.0, added QC parameters and detail including Batch, Method Blanks, and Sample Duplicate;

20.2.8 Added Calculation of RPD for duplicates in Section 11.0;

20.2.9 Biennial review and update.

20.3 Reason for changes, Revision 05:

20.3.1 Updated Section 10.4.1 to reflect current use of 70-90% isopropyl alcohol.

Table 1: Equivalent weights table for major Cations and Anions

Equivalent Weights Table for the Major Cations and Anions			
Cations		**Anions**	
Element	Equivalent Weight	Element	Equivalent Weight
Calcium (Ca)	20.04	Chloride (Cl)	35.453
Magnesium (Mg)	12.1525	Carbonate (CO3)	30.004
Sodium (Na)	22.9898	Bicarbonate (HCO3)	61.016
Potassium (K)	39.098	Sulfate (SO4)	48.03
Lithium (Li)	6.94	Nitrate (NO3)	62.004
Iron (Fe)	27.97	Bromide (Br)	79.904
Manganese (Mn)	27.469	Nitrogen (N)	14.007
Strontium (Sr)	43.81	Ammonium (NH4)	18.039
Barium (Ba)	68.665	Phosphate (PO4)	31.66

Table 2: Conversion factors for mg/L to meq/L

Contituent	To convert mg/L to meq/L multiply by	To convert meq/L to mg/L multiply by
Calcium (Ca)	0.0499	20.04
Magnesium (Mg)	0.0823	12.15
Sodium (Na)	0.0435	22.99
Potassium (K)	0.0256	39.098
Aluminum(Al)	0.1112	8.99
Manganese (Mn)	0.0364	27.47
Barium (Ba)	0.0146	68.665
Chromium (Cr)	0.0577	17.33
Copper (Cu)	0.0315	31.75
Hydrogen (H)	0.9921	1.008
NH4	0.0554	18.04
Br	0.0125	79.9
Cl	0.0282	35.45
F	0.0526	18.99
Bicarbonate (HCO3)	0.016	61.016
Carbonate (CO3)	0.033	30.004

TITLE: RECEIPT, LOG-IN, AND STORAGE OF ENVIRONMENTAL SAMPLES

Approvals (Signature/Date):

John Mathewson 1/17/18 John Mathewson Date Inorganics Manager/EH&S Coordinator	Gayland Fisher 1/17/18 Gayland Fisher Date Organics Manager
Tina M. Godines 1/17/18 Christina Godines Date Quality Assurance Manager	Chip Meador 1/17/18 Chip Meador Date Laboratory Director

This SOP was previously identified as SOP No. CC-GLO-008 Rev 10

Facility Distribution No. Intranet **Distributed To:** _____

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) provides procedures used by Corpus Christi for receiving samples into the laboratory for analysis, checking the integrity of the samples, log-in, storage, and disposition of all environmental samples received at Corpus Christi.

2.0 SUMMARY

2.1 Receipt of environmental samples at Corpus Christi must be performed in adherence with this SOP to ensure proper chain-of-custody, sample receipt conditions, preservation, and assignment of appropriate analytical methods.

3.0 DEFINITIONS

3.1 Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records.

~~3.2~~ Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results.

3.3 Custody Seal: a printed adhesive seal affixed to a shipping and/or sample container. Custody seals are used to verify that shipping and sample containers have not been opened or tampered with prior to receipt at the laboratory.

3.4 Equipment Blank**:** a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

3.5 Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

3.6 Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

3.7 Matrix: the substrate of a test sample.

3.8 Non-Conformance Memo (NCM): an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

3.9 Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

3.10 Trip Blank - A sample of analyte-free media that accompanies samples during shipping. A trip blank is used to document contamination attributable to shipping and field handling procedures.

3.11 Temperature Blank Bottle: This bottle is included in each out-going cooler to verify the temperature of samples upon receipt.

3.12 TALS: TestAmerica Laboratory Systems

4.0 INTERFERENCES

4.1 Not applicable to this SOP.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1.1 Specific Safety Concerns or Requirements

5.1.1.1 If it is suspected that a sample (bottle/vial/jar) has broken or is leaking into the cooler, open the cooler inside the fume hood and use cut resistant gloves when appropriate.

5.1.1.2 Proper lifting techniques must be used when lifting coolers. A fully loaded cooler may require more than one employee to safely lift onto a counter.

5.1.2 Primary Material Used: The following table lists materials used in this process that have serious or significant hazard rating. **NOTE: This list does not include all materials used in the process.** The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the process can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Sodium Hydroxide	Corrosive	2mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation and severe burns and scarring with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and sever burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Equipment

6.1.1 Computer with TALS

6.1.2 Copier/Scanner

6.1.3 SatoCL412e Label Maker

6.1.4 Thermometer, IR gun

6.2 Supplies

6.2.1 Chain of Custody

6.2.2 Labels

6.2.3 pH paper strips

7.0 REAGENTS AND STANDARDS

7.1 Not applicable to this SOP.

8.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIMES, STORAGE AND DISPOSAL

8.1 Sample Collection, Preservation, Storage and Holding Times: Sample collection, preservation, storage and holding times will be in conformance with the specific method and Sample Handling Guide.

8.2 Sample Disposal: Dispose of samples according to approved procedures for sample disposal. The standard sample disposal time is 6 weeks from login date.

9.0 QUALITY CONTROL

9.1 Sample Containers and Preservatives

9.1.1 Types of Sample Containers: Appropriate sample containers shall be used for each parameter. Plastic containers are generally used for metals analysis and the majority of inorganic parameters. Glass containers with Teflon-lined lids or septa are used for all organic parameters. The type of container and level of cleanliness must meet EPA specifications.

9.1.2 Certified Pre-Cleaned Sample Containers: Certified pre-cleaned sample containers should be used where appropriate, such as for volatile samples. Bottle manufacturers should provide information regarding the level of cleanliness for each type of container used by the laboratory. The certificates of cleanliness provided by the bottle manufacturer for each lot of bottles received by the can be retrieved on the ESS website as needed.

9.1.3 Preservatives: Preserve containers are ordered directly from ESS.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Not applicable to this SOP.

11.0 PROCEDURE

11.1 Sample Receipt Hours: Generally, Sample Control personnel will be available for courier deliveries and to receive samples during normal business hours, Monday through Friday and Saturday mornings for sample receipt only (no courier services on weekends).

11.1.1 If samples are delivered when Sample Control personnel are not present, laboratory personnel shall sign, receive samples, note receipt temperature on COC and store samples in the walk-in refrigeration unit(s). The COC will be placed on the Sample Control desk.

11.1.2 Requests for courier services outside of the regular route **MUST** be made 24 hours in advance **AND** must receive approval by the Laboratory Director.

11.2 Sample Acceptance Policy: Corpus Christi's Sample Acceptance Policy consists of a set of documents that clearly outline the requirements under which samples are to be accepted. These documents specify the requirements for sample containers, preservation, including proper pH and temperature, volume, holding times, bottle labels, and chain of custody documentation. The Sample Acceptance Policy includes verifying the following areas of concern:

11.2.1 Documentation

11.2.1.1 Proper, full, and complete documentation, which shall include client identification, sample identification, location, date and time of collection, sample collector's name, preservation, sample type, requested methods for analysis, and any special remarks concerning the sample

11.2.2 Sample Labeling

11.2.2.1 Proper sample labeling shall include a unique identification and labeling system for the samples. The samples should be labeled using durable and water resistant labels and/or indelible ink

11.2.3 Appropriate Sample Containers and Preservatives

11.2.3.1 Check for the use of appropriate sample containers and preservatives.

11.2.4 Holding Times

11.2.4.1 Adhere to method specified holding times

11.2.5 Sample Volume

11.2.5.1 Adequate sample volume for performing the necessary tests

11.2.6 Sample Temperature

11.2.6.1 All samples which require thermal preservation shall be considered acceptable if the arrival temperature complies with either of the following criteria (based on "2009 NELAP Standards"):

11.2.6.2 Within 2°C of the required temperature of the method specified range.

- For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable.

- Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

- The client is contacted to inform them of the unacceptable condition if the conditions in Sections 11.2.1 – 11.2.6 are not met. This contact will be noted on the Sample Receipt Checklist or in TALS. A Non Conformance Memo (NCM) is generated for the unacceptable sample conditions.

11.2.6.3 Sample Courier's provide sample pick up for certain clients located within a 50 mile radius of the laboratory. The courier only provides sample pick up services and is not performing test in the field.

11.2.6.3.1 The courier drives to the designated client site to retrieve coolers and leaves bottle kits for the next day.

11.2.6.3.2 The courier returns the coolers and relinquishes them to the sample control technicians in the laboratory.

11.2.6.3.3 The courier has a mobile device in the company vehicle at all times. The courier is instructed not to use the mobile device while driving and must abide by all vehicle safety regulations.

11.3 Sample Receipt Procedures

11.3.1 All associated shipping documents; Chain of Custody Records, etc. shall be placed in the job files.

11.3.2 The sample receipt procedure is as follows:

11.3.3 Shipping Cooler, Container Inspection and Receipt

11.3.3.1 Inspect the custody seals on the shipping container. Indicate on the TALS Sample Receipt Checklist whether the custody seals are intact, broken, or missing & on the Chain of Custody (COC).

11.3.3.2 Remove all air bills, freight invoices, and shipping documents from the exterior of the shipping container. Verify that the number of packages

or coolers received agrees with the number declared on the shipping document.

11.3.3.3 Record the date and time of sample receipt on the chain of custody. The person receiving the sample shipment must sign the (COC).

11.3.3.4 Open the shipping cooler/container and remove all paperwork included in or attached to the sample shipment and place the paperwork in the job file.

11.3.3.5 All coolers/containers in the sample receiving area are inspected to ensure that samples are not broken or leaking in the cooler. If a cooler or container exhibits damage or unusual characteristics, the coolers/containers will be opened under a fume hood. Samples will be treated with extreme care until they are determined to be safe to handle.

11.3.3.6 Include all shipping documents in the job file.

11.3.3.7 Record damaged or missing packages/containers on the Sample Receipt Checklist and notify the PM of the problems & NCM the sample in TALs.

11.3.4 Sample Condition Inspection

11.3.4.1 Check the temperature upon receipt of all samples that require thermal preservation. The sample temperature should be checked using any one of these procedures:

11.3.4.1.1 IR Temperature Gun: Use the IR Gun and scan the sample temperature through the container. The IR Gun must be within 1 to 3 inches (or within the length of your pointer finger) from the sample when reading is taken. Record the sample temperature reading and the IR Gun ID on the Chain of Custody form and/or the TALS Sample Receipt Checklist Custody Record. The IR Temperature Gun is checked at the beginning of each day to ensure the accuracy of the temperature reading.

11.3.4.1.2 Digital Thermometer: Place the probe of the digital thermometer in the temperature blank container (if available) or in the shipping container and close the lid. Take a temperature reading after 2-3 minutes. Record the temperature reading on the TALS Sample Receipt Checklist and Chain of Custody Record.

11.3.4.1.3 Standard Thermometer: The sample temperature is determined by inserting a thermometer between one sample

container and the bubble-pack. If the sample is not wrapped in bubble-pack, wrap the sample with bubble–pack and insert the thermometer between the bubble-pack and the container.

Note: The acceptance temperature for coolers is ≤ 6°C without freezing. This criterion replaces the previous criteria of 4°C ± 2°C. This criterion is used as default criteria. Some states, programs, and clients may specify other acceptance criteria.

Note: Evidence of Cooling for Local Sampling Event: For samples that are received at the lab immediately or soon after sampling, the cooler and sample temperature will most likely not be ≤ 6°C. In these cases, notification is not required if the temperature is above 6°C and there is ice in the container, as long as the temperature of the container is less than ambient (room) temperature. This situation should be noted on the Sample Receipt Checklist in TALS by receipt personnel (e.g.: just sampled, on ice, etc.)

Note: To avoid compromising sample integrity, sample contents and/or opened containers must not be used for temperature check unless written instructions specify otherwise.

11.3.4.2 If temperature excursions are noted (i.e. samples not received at ≤ 6°C), document this temperature excursion on the TALS Sample Receipt Checklist and notify the appropriate PM. The PM will contact the client for further instructions.

11.3.4.3 Remove samples from the shipping container in a well-ventilated area using appropriate protective equipment (safety glasses and gloves, at a minimum). If broken samples are received, place them inside the fume hood. When sample hazards are known or suspected, samples must be removed and inspected in a fume hood.

11.3.4.4 Document any unusual sample conditions (strong odors, leaking or broken sample containers, missing sample labels, etc.) on the TALS Sample Receipt Checklist. Inform the Project Manager concerning sample conditions which may affect sample analysis (inverted septum, incorrect preservative, air bubbles greater than ¼ inch or 6 mm in diameter in VOA vials, etc.) NCM of sample conditions should be generated in TALs.

11.3.4.5 Clean and dry the exterior surfaces of sample containers, where necessary, prior to sample storage.

11.3.4.6 Volatiles samples are to be removed from coolers checked for headspace and container count. Stack VOA's in Racks and places the samples back in the original cooler until login is complete.

11.3.4.6.1 Volatile samples are to be logged, labeled and placed in the designated fridge within an approximate time frame of 20 minutes.

11.3.4.6.2 The laboratories plan of action if samples cannot be logged and labeled within the approximate 20 minute time frame: sample control personnel will place the samples in coolers that contain ice packs or bags of ice to keep the samples cool until the samples are ready to be logged in.

11.3.5 Chain of Custody Record: Chain of custody is the documented evidence of the entire sample transfer from one party to another, from sample collection to the final destination for analysis or return to the client. Chain of custody shall be followed for all samples received. The procedure applies to either the TestAmerica Chain of Custody Record or a client form. Laboratory personnel receiving samples shall document and verify the following items on the Chain of Custody Record.

11.3.5.1 Document method of shipment (FedEx, UPS, DHL….) and airbill number if applicable.

11.3.5.2 Document the **date** and **time** of sample receipt in the next available section marked “RECEIVED BY:”

11.3.5.3 The sample custodian or other laboratory personnel receiving samples shall **sign**, **date**, and **record the time** on the Chain of Custody Record.

11.3.5.4 Verify that the number and type of containers received agrees with the number and type of containers specified on the Chain of Custody Record.

11.3.5.5 Verify that the sample information recorded on the sample label agrees with the sample information on the Chain of Custody Record.

11.3.5.6 Discrepancies or unusual sample conditions noted during sample receipt must be documented on the Sample Receipt Checklist for client notification.

11.3.5.7 If samples are delivered directly to the laboratory by a TestAmerica representative that is not a TestAmerica sample custodian, the TestAmerica representative who initially received the samples must sign over custody of the samples upon arrival at the lab.

11.3.6 Sample Verification

11.3.6.1 If removable sample ID tags are provided (EPA sample tags, etc.), remove these tags from the sample container, date/initial the tags, and place the sample tags in the job file.

11.3.6.2 Document all discrepancies between actual and declared conditions of the sample transmittal on the TALS Sample Receipt Checklist. Notify PM(s) for any unacceptable sample receipt condition. Generate NCM in TALs.

11.3.7 Sample Preservation Verification

11.3.7.1 The pH of liquid samples shall be verified during sample login for all samples that are marked as being preserved except for VOA and Oil and Grease samples. The pH of volatile organics and Oil and Grease samples are taken during the analysis process and recorded in the appropriate logbook or instrument printout.

11.3.7.1.1 For plastic or glass containers, transfer a small amount of sample (less than 0.5mL) to pH paper using a disposable plastic transfer pipette. Verify that the proper pH level is present for each sample container submitted. The pH of the container will be recorded in the TALS system. The lost number of the pH paper will be entered into TALS.

11.3.7.1.2 If preservation discrepancies are noted, document the discrepancy for each affected sample container on the NCM. Write the pH value on the bottle and notify the PM of any unacceptable sample preservation condition. The client shall be contacted by the PM to determine whether the sample should be preserved in the laboratory or whether the client prefers to re-sample.

11.3.7.2 The pH of chemically preserved aqueous samples shall also be verified prior to analysis and recorded in the appropriate logbook.

11.3.8 Sample Receipt Checklist: A TALS Sample Receipt Checklist shall be completed for samples received. At a minimum, the following information shall be documented on the Checklist:

11.3.8.1 Client ID and laboratory job number

11.3.8.2 Chain of Custody Record provided with samples

11.3.8.3 Condition of custody seals (if provided)

11.3.8.4 Sample condition at receipt

11.3.8.5 Temperature of cooler

11.3.8.6 Any discrepancies in the type of sample containers used

11.3.8.7 Any discrepancies in the type or volume of preservatives used

11.3.8.8 Headspace or presence of air bubbles in volatile sample vials

11.3.8.9 Agreement between Chain of Custody Record and sample labels

11.3.9 Sample Condition Notification Procedure

11.3.9.1 The project manager or designee must inform the client in a timely manner of any discrepancies which may affect sample integrity or sample analysis. Client contact by telephone regarding sample receipt and chain of custody issues shall be recorded in the job file or in an NCM. In addition, the client's authorization to proceed with analyses on samples or sample containers that did not meet the required specifications shall also be documented in the job file or in an NCM.

11.4 Inter-Laboratory Sample Transfer: All of the chain of custody and sample receipt requirements detailed in this SOP applies to samples transferred between TestAmerica locations and other subcontract laboratory facilities. Guidelines for inter-laboratory sample transfers are as follows:

11.4.1 Shipping Laboratory: Shipping laboratories shall contact a project manager at the receiving laboratory prior to sample transfer with the details on all analytical, quality control, requested job turnaround time, and final reportable requirements.

11.4.2 Client Requirements: If client requirements cannot be met by the receiving laboratory, the shipping laboratory shall be contacted immediately.

11.4.3 Chain of Custody Record: A Chain of Custody Record shall be provided by the

originating laboratory for all samples transferred between TestAmerica laboratory locations or subcontract laboratory facilities. A copy of the Chain of Custody Record is kept electronically.

11.4.4 Packaging Requirements: Glass containers should be wrapped in bubble bag or equivalent prior to being packed in the cooler. Coolers should be lined with plastic bags and containers should be packed upright in those bags. A temperature blank bottle is included in each cooler. The coolers should be filled with ice to ensure proper temperature upon arrival at the subcontracted laboratory. Coolers will be shipped for next morning delivery via Federal Express or another shipper that can guarantee next day delivery.

11.5 Sample Log-In: All samples received by the laboratory shall be logged into TALS. The system assigns a unique identification number. Samples received after normal working hours shall be placed in the proper storage location and logged into TALS on the next working day. Samples are logged in according to the following procedure.

11.5.1 Job and Project Information

11.5.1.1 The system automatically assigns a unique job number and sequential sample numbers for each job during log-in.

11.5.1.2 When possible, log-in the job using a client-specific project that has been setup, reviewed, and approved by the assigned project manager. If a project has not been setup for a sample delivery group, contact the PM and proceed with log-in of as much information as possible.

11.5.1.3 Verify that the client reporting contact and invoicing information matches the information submitted on the Chain of Custody Record. Contact the project manager if additional client contacts must be entered in TALS. Where necessary, enter job questions for clients that require customized electronic data deliverables (SAIC, AFCEE, etc.). Contact the project manager to determine which projects require this additional log-in information.

11.5.2 Sample and Container Information

11.5.2.1 It is important that each log-in sample contains the correct sample date/time, date/time received, and the client's sample identification. Each sample container is assigned a unique ID. Complete the sample log-in information section for each sample received by entering the container types, preservatives (and bottle lot number), pH (and pH paper lot number) and sample storage location. Print computer-generated sample label.

11.5.2.2 Label each container. Verify the laboratory assigned sample identification versus the client sample identification for each container prior to affixing the sample label.

11.5.2.3 Distribute the samples to the appropriate storage areas.

11.5.2.4 **NOTE:** Volatile/TX1005 soil samples are segregated in the soil refrigerators and/or freezers. See section 11.6.2 for more information.

11.5.3 Methods and Tests

11.5.3.1 Log-in the requested methods and tests for each sample. Utilize the systems methods and tests group function for logging in multiple samples with the same parameter requests. Where possible, use the methods and tests in a client-specific project, which has been setup, reviewed and approved by the assigned project manager. If a project has not been defined for a sample delivery group, contact the PM for further instruction.

11.5.3.2 Assign the appropriate sample matrix type to each parameter. Verify that all required sample preparation methods (extractions, digestions, moisture, etc.) are also logged in. Subcontract work must also be logged in to produce the final data deliverable package.

11.5.4 Log-In Documentation

11.5.4.1 Place the login sample summary report, the COC, and all the associated shipping documents in the job file.

11.5.4.2 Distribute job files to the appropriate PM for log-in review, approval and corrections electronically.

11.6 Sample Storage: Samples, including all sample fractions, extracts, leachates, and other sample preparation products shall be stored and segregated according to the preservation requirements for each test method. Most environmental samples require refrigeration at ≤ 6°C. In addition, the following sample storage requirements shall be followed for environmental samples:

11.6.1 Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.

11.6.2 Volatile Organics Samples

- VOA vial water samples submitted for volatile organics analysis shall be segregated from all other sample types in a separate refrigerator.
- Bulk soil samples submitted for 5030B/8260 analysis shall be stored in a separate refrigerator.
- VOA vial soil samples submitted for 5035/8260 analysis shall be stored in a separate freezer.
- TX1005 soil samples submitted for TX1005 analysis shall be stored in a separate freezer.

11.6.3 Organic Analysis Extracts: Organic analysis extracts prepared in the laboratory shall be segregated from all other sample types in a separate refrigerator.

11.6.4 Storage of Samples and Sample Extracts: Samples and sample extracts shall not be stored in the same refrigeration unit with laboratory reagents or standards.

11.6.5 Holding Blanks: Holding blanks shall be utilized for volatile sample storage units.

11.6.6 Responsibilities: All sample-handling staff are responsible for maintaining the sample segregation and storage requirements for environmental parameters. Samples shall be returned to the appropriate storage location immediately following analysis. All samples requiring refrigeration shall be returned to the appropriate refrigeration unit at the conclusion of each working day.

11.7 Sample Handling Procedures: Samples are to be handled according to the requirements for the applicable parameters. Sample handling requirements that include holding times and preservatives for environmental parameters are provided in TestAmerica's Sample Handling Guide.

11.7.1 EPA Specified Holding Times: All sample analyses must be completed within the maximum allowable holding times specified by the EPA. Holding times are based on the date and time sampled, not on the date and time of laboratory receipt. TALS holding time status/backlog reports should be used to monitor all parameters.

11.7.2 **Samples received by the laboratory that have expired holding times must be immediately notified to the PM**. The laboratory **may** proceed with analysis unless directed otherwise by the client. Client contact regarding expired holding time must be documented and maintained in the job file along with NCM in TALs. All samples analyzed outside the maximum allowable holding time must be documented in a case narrative and provided to the client with the final report.

11.7.3 Sample Aliquoting: The laboratory shall use documented procedures (CC-GLO-017: Sample homogenization and subsampling) and appropriate techniques to obtain representative samples whenever sample aliquots are required (i.e. subcontracted analysis).

11.8 Priority Sample Analysis: Clients are responsible for providing the laboratory with samples in a timely manner so that the laboratory can complete the analytical work within the EPA recommended holding times and client specified turnaround. The laboratory is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half of the holding time has passed. If these conditions are not met, the Project Manager (PM) will contact the client regarding the course of action. The laboratory will attempt to expedite sample analysis.

Samples designated for rush turnaround and parameters with short holding times must be given priority for sample analysis. Rush samples should be indicated as such to enable the laboratory to meet the client's turnaround time requirements. The following procedures should be followed for samples with short holding times or priority/rush jobs:

11.8.1 Inspection of Cooler Contents: Open all coolers immediately upon receipt and examine the Chain of Custody Record and all associated paperwork to determine which jobs must be logged in as priority jobs.

11.8.2 Rush Turnaround Requests: Rush turnaround time requests shall be brought to the attention of the laboratory section and the project manager to ensure turnaround time requirements will be met. If rush turnaround time requirements cannot be met due to workload, the project manager shall contact the client to inform them of the inability to meet their turnaround time requirement.

11.8.3 Short Holding Time or Rush Turnaround Samples: Samples with limited holding time remaining and/or rush turnaround time client requests should be treated as priority jobs and must be logged in immediately upon receipt in order to meet regulatory and client requirements. See Table I for short hold time analyses.

11.9 Sample Tracking: TALS is used to monitor and track sample status from sample receipt to final reporting and invoicing.

11.9.1 Activities monitored by the systems include sample preparation, sample analysis, data review, report preparation, and electronic data deliverables. Based on client or project requirements, additional sample tracking procedures may be implemented.

11.10 Sample Security: Samples shall be maintained in the secured facility in designated sample storage areas. Based on project or client requirements, additional sample security and tracking procedures will be implemented.

11.11 Sample Disposal: Samples, digestates, leachates and extracts or other sample preparation products shall be disposed of in an environmentally approved manner. Samples shall be retained for a minimum of 6 weeks from the date of sample receipt unless otherwise specified by a client/contract. After this period, samples will either be returned to the client or disposed of in accordance with Federal, State and Local regulations. TestAmerica reserves the right to charge the client for shipping and/or disposal costs of unused samples.

12.0 DATA GENERATION AND CALCULATIONS

12.1 Not applicable to this SOP.

13.0 METHOD PERFORMANCE

13.1 Not applicable to this SOP.

14.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 WASTE MANAGEMENT

15.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

16.0 REFERENCES / CROSS-REFERENCES

16.1 Reference Current QAM CC-QAM-001 (current revision)

16.2 CC-LOG-WI001 Method 5035 Sampling Instructions (current revision)

16.3 CC-LOG-WI002 Sample Acceptance Policy (current revision)

16.4 CC-LOG-WI003 Sample Handling Guide (current revision)

16.5 CC-LOG-WI004 Sampling, Shipping, & COC Instructions (current revision)

17.0 METHOD MODIFICATIONS

17.1 Not applicable to this SOP.

18.0 REVISION HISTORY

Historical File:		Revision 04: 07/09/09		Revision 09: 06/03/15
Revision 00: 04/15/04		Revision 05: 07/30/10		Revision 10: 07/13/16
Revision 01: 09/01/06		Revision 06: 04/05/11		Revision 11: 01/17/18
Revision 02: 09/15/06		Revision 07: 01/15/15		
Revision 03: 05/08/09		Revision 08: 02/19/15		

Revision Date	Change from	Change to	Reason
07/13/16	Revision 9	Revision 10	Typographical Errors Sec 17. Minor formatting change Biennial Review Sec 11.4.4, Removed Trip Blank requirement.

18.1 Revision 11, dated 17 January 2018, updated by Christina Godines:

18.1.1 Section 11.3.7.1.1 updated to include recording the lot number of the pH paper in TALS;

18.1.2 Section 11.5.2.1 updated to include entering preservative bottle lot number and pH paper lot number in TALS;

18.1.3 Section 6.0 updated to include thermometer, IR gun, labels, and pH paper strips;

18.1.4 Updated formatting throughout to conform to current corporate guidelines on writing SOPs in SOP No. CW-Q-S-002

19.0 TABLE, FORM, ATTACHMENT

19.1 Attachment 1: Short Holding Time Analyses

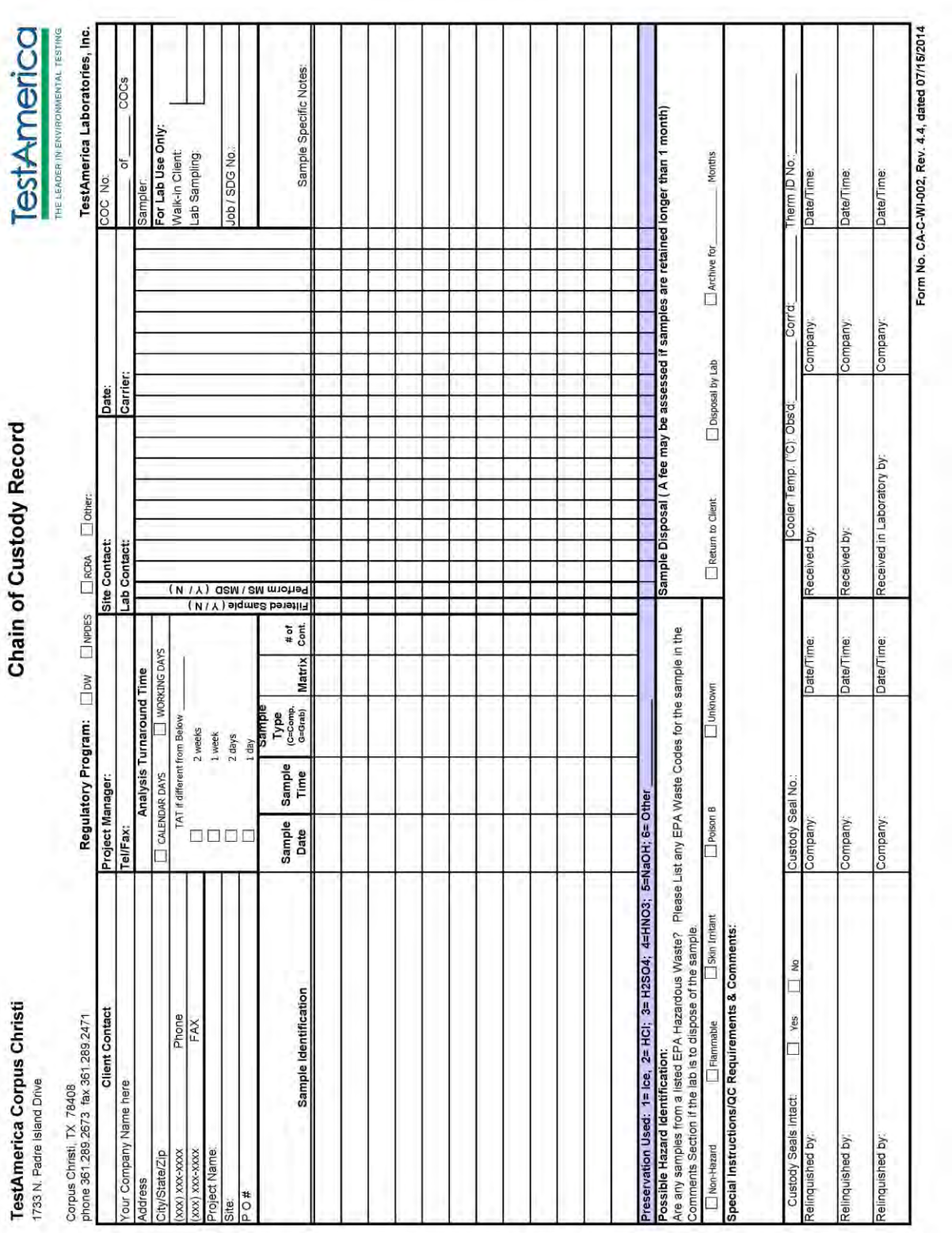
19.2 Attachment 2: Example Chain of Custody Record

Attachment 1 – Short Holding Time Analyses

Holding Time	Analysis	Method No.
Analyze Immediately On Collection (testing for these parameters should be done in the field)	**Dissolved Oxygen (DO)**	**SM4500-O G / 360.1**
	Ferrous Iron	**SM3500-FE D**
	Free Carbon Dioxide (CO2)	**SM4500-CO_2 C, D (Subcontract)**
	Iodide	**(Subcontract)**
	pH (water)	**SM4500-H^+ B / 9040C**
	pH (soil)	**9045D**
	Residual Chlorine	**SM4500-Cl F / 330.4**
	Sulfite (SO_3^{2-})	**SM4500-SO_3 B / 377.1**
6 hrs from Collection	**Enterococci**	**ASTM D6503-99 / Enterolert(IDEXX) (Subcontract)**
	Fecal Coliform (membrane filter)	**SM9222D (Subcontract)**
	Total Coliform (membrane filter)	**SM9222B (Subcontract)**
Analyze within 24 hrs from Collection.	**Hexavalent Chrom (Chromium (VI))**	**SM3111C / 218.4 / 7197**
Filter within 24 hrs prior to acidification (Should be Field filtered)	**Dissolved Metals**	**200.8 / 6020 (ICPMS)** **200.7 / 6010 (ICP/Salt only)**
Analyze within 48 hrs from Collection.	**Biological Oxygen Demand (BOD)**	**SM5210B / 405.1**
	Carbonaceous BOD (CBOD)	**SM5210B**
	Color	**SM2120B / 110.2**
	MBAS/Surfactants	**SM5540C / 425.1**
	Nitrate by IC **Nitrite by IC**	**300.0 / 9056**
	Nitrite	**SM4500-NO2 B / 354.1**
	Odor	**SM2150B / 140.1 (Subcontract)**
	Orthophosphate	**365.3**
	Settleable Solids	**SM2540F (SubContract)**
	Turbidity	**SM2130B / 180.1**
Freeze within 48 hrs	**Encores*/5035VOA**	**EPA 8260B**
	TPH TX1005 Soil	**TX 1005 (Soil)**
Analyze within 72 hrs from Collection.	**Formaldehyde / Crotonaldehyde (H2O)**	**8315?** **SubContract**
	BTEX / GRO – Air Tedlar bags	**8021B**

*Encore samples: Sample custodian must notify VOC analysts upon laboratory receipt so that Encores samples can be extruded into VOA vials within 48 hours.

Attachment 2: Example of Chain of Custody



TestAmerica Corpus Christi
1733 N. Padre Island Drive
Corpus Christi, TX 78408
phone 361 289 2673 fax 361 289 2471

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

Chain of Custody Record

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

Client Contact
Your Company Name here
Address
City/State/Zip
(xxx) xxx-xxxx Phone
(xxx) xxx-xxxx FAX
Project Name:
Site:
P O #

Project Manager:
Tel/Fax:

Site Contact:
Lab Contact:

Date:
Carrier:

COC No:
___ of ___ COCs

Analysis Turnaround Time
☐ CALENDAR DAYS ☐ WORKING DAYS
TAT if different from Below
☐ 2 weeks
☐ 1 week
☐ 2 days
☐ 1 day

Sampler:

For Lab Use Only:
Walk-in Client:
Lab Sampling:

Job / SDG No.:

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ Return to Client ☐ Disposal by Lab ☐ Archive for _____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd: _____ Corr'd: _____ | Therm ID No.: _____

	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:					
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.4, dated 07/15/2014

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Corpus Christi
Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica Corpus Christi will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When samples are to be shipped by commercial carrier, please do not forget to sign, date and time the "relinquished by" box on the chain-of-custody.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes client identification, sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination, inadequate preservation or fail to meet any of the above requirements.
- Non-standard analytical requirements and/or expedited turnaround times should be discussed with the Project Manager prior to sample acceptance.
- Known safety hazards should be communicated on the chain-of-custody form.
- Due to the health and safety hazards, samples containing hydrofluoric acid (HF) will not be accepted.
- Our laboratory is not licensed to accept radioactive or mixed waste samples.
- If an analysis not performed by TestAmerica Corpus Christi is requested, we will notify you for your decision to have the analysis performed at another TestAmerica facility, to have TestAmerica subcontract with another laboratory, or to have the samples returned to you.

THIS PAGE LEFT BLANK INTENTIONALLY

Eurofins Test America SOPs

Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Polychlorinated Biphenyls (PCBs) by GC/ECD [SW846 Methods 8082 and 8082A]

Approvals (Signature/Date):

Tegan Moore — 1/4/19
Technical Specialist — Date

Doug Gomer — 1/4/19
Health & Safety Manager / Coordinator — Date

Roxanne Sullivan — 1/4/19
Quality Assurance Manager — Date

Richard Clinkscales — 1/4/19
Laboratory Director — Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This SOP describes the procedure for the determination of concentrations of polychlorinated biphenyls (PCB) as Aroclors using the methodology prescribed in EPA SW-846 Method 8082 and 8082A.

1.2 This procedure is applicable to the gas chromatography analysis of extracts of aqueous, solid, and oil samples. When utilized for the analysis of oils, additional cleanup procedures may be required. This procedure also defines the conditions required when using a large volume injection.

1.3 This SOP does not include the procedures for extracting environmental samples. Refer to TestAmerica SOPs DV-OP-0006, DV-OP-0007, DV-OP-0015 and DV-OP-0016 for sample preparation procedures. Refer to SOP DV-OP-0012 for waste dilutions.

1.4 Additional information is provided in this SOP for the inclusion of the analysis of polychlorinated terphenyls (PCT) by the same protocols used for the determination of Aroclors.

1.5 This SOP does not include the determination of the concentration of PCB congeners.

1.6 Analytes, Matrix(s), and Reporting Limits

Tables 1 and LVI-1 list the specific Aroclors that are determined using this procedure and their associated reporting limits (RLs).

2.0 Summary of Method

2.1 Preparation

2.1.1 Aqueous Samples

PCBs are extracted from a one-liter aqueous sample with methylene chloride using a separatory funnel (SW-846 Method 3510). The extract is evaporated to approximately 25 mL and exchanged to hexane. The final extract volume is 10 mL, however depending on special client requirements the final extract volume can also be 1 mL or 5 mL. The extraction procedure is detailed in SOP DV-OP-0006.

2.1.2 LVI Aqueous Samples

PCBs are extracted from a 250 mL aqueous sample to a final volume of 5 mL with hexane (SW-846 Method 3510). The extraction procedure is detailed in SOP DV-OP-0006.

2.1.3 Solid Samples

PCBs are extracted from solid materials using either sonication or microwave extraction. If sonication extraction is selected the samples are

extracted with a 50:50 Acetone:Methlyene Chloride mixture, concentrated down to approximately 25 mL, exchanged with hexane, and brought to a 10 mL final volume. See DV-OP-0016 and DV-OP-0007 for details. If microwave extraction is selected the samples are extracted with a 50:50 Acetone:Hexane mixture, and concentrated down to a 10 mL final volume. See DV-OP-0015 and DV-OP-0007 for details.

2.1.4 Oil Samples

Oil samples are typically prepared by diluting 1 gram of sample to a final volume of 10 mL with hexane. The extraction procedure is detailed in SOP DV-OP-0012.

2.1.5 Wipe Samples

Wipes are typically collected using either filter paper or gauze. These samples can then be extracted using the procedure outlined in SOP DV-OP-0016.

2.1.6 Cleanup Procedures

Cleanup options are discussed in Section 4 below. Instructions for performing various cleanup procedures are detailed in SOP DV-OP-0007. All cleanups that are performed must be documented in an NCM.

2.2 Analysis

Samples are analyzed using a gas chromatograph with dual electron capture detectors (ECDs). Specific Aroclor mixtures are identified by the pattern of peaks compared to chromatograms of reference standards. The concentrations of Aroclors in the sample extract are determined using an internal standard calibration. Second column confirmation is only performed when requested by the client or as a program requirement. Work under the DoD/DOE QSM program and method 8082A require second column confirmation. The presence of multiple peaks that are characteristic of an aroclor in the sample serves as confirmation of analyte presence.

3.0 Definitions

3.1 Polychlorinated biphenyls (PCBs): PCBs are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl, with a general chemical formula of $C_{12}H_{10-x}Cl_x$. There are 209 possible congeners.

3.2 Aroclor: PCBs were produced as technical mixtures by the chlorination of biphenyl. Production processes were designed to produce mixtures with characteristic chlorine contents. In the United States, most of the PCBs in the environment are in the form of Aroclors, which were produced by Monsanto from the 1930s through 1977. Each Aroclor mixture is identified by a four-digit number, the first two digits of which indicate the number of carbons in the biphenyl ring, i.e., 12, and the second two of which indicate the weight percent of chlorine. For example, Aroclor 1254 has

12 carbons and 54% by weight chlorine. The exception is Aroclor 1016, which has 12 carbons and 42% by weight chlorine.

NOTE: Each specific Aroclor produces a characteristic gas chromatographic pattern that represents the relative amounts of PCB congeners in the formulation. The formulation of the mixtures from batch to batch was fairly consistent, but never exactly the same. In almost all cases, the gas chromatogram can be used as a fingerprint to identify the specific Aroclor. Exceptions occurred for Aroclors 1254 and 1221. In each case, at least one batch was produced under different conditions, which resulted in an Aroclor mixture with the same approximate chlorine content, but with a significantly different distribution of congeners. These odd batches of 1254 and 1221 produce chromatographic patterns that are very different from the typical formulations. Standards for these odd batch Aroclors can be used to aid in the qualitative identification of Aroclors in environmental samples.

3.3 AR1660: Laboratory designation for the mixture of Aroclors 1016 and 1260.

3.4 AR2154: Laboratory designation for the mixture of Aroclors 1221 and 1254.

3.5 AR3262: Laboratory designation for the mixture of Aroclors 1232 and 1262.

3.6 AR4268: Laboratory designation for the mixture of Aroclors 1242 and 1268.

3.7 Polychlorinated Terphenyls: Polychlorinated terphenyls (PCTs) are chemically related to PCBs with the exception that PCTs have an additional phenyl group. The PCTs included in this analysis are AR 5432, AR 5442, and AR 5460. The preparation and analysis is treated the same as for the PCB Aroclor analysis.

3.8 Lower Limit of Quantitation (LLOQ): The lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be $\geq$ the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit. The required LLOQ verification is performed at a concentration of 1-2 times the LLOQ (or RL).

4.0 Interferences

4.1 Hydrocarbons can co-elute and thereby mask the Aroclor pattern. The laboratory uses acid cleanup with concentrated sulfuric acid to remove hydrocarbons from solid and oil sample extracts, and for water samples when extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Acid cleanup removes low-to-medium molecular weight polar organic interferences from sample extracts. Detailed instructions for performing acid cleanup are provided in SOP DV-OP-0007.

All QC is brought through the cleanup process and reported with the samples. An aliquot of all samples and QC is set aside and not brought through the cleanup process. If the QC is out of criteria due to the cleanup process then the QC that

wasn't brought through the cleanup process will be analyzed and used to verify the batch for the samples not brought through clean-up.

4.2 Sulfur will interfere and can be removed using procedures described in SOP DV-OP-0007.

4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Generally any sample that has a concentration greater than the highest calibration point will warrant consideration for carryover. Any affected samples are re-analyzed.

4.4 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector.

4.4.1 Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.4.2 Single-component chlorinated pesticides, if present, may co-elute with individual PCB congeners and interfere with the identification and/or quantitation of the aroclors. This can be addressed by analyzing a chlorinated pesticide mixed standard prior to an initial calibration to identify where potential interferences might occur.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (M-E-001 DV), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2.4 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the TestAmerica Denver Radiation Safety Officer and the TestAmerica Corporate EH&S Director shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment. Always use proper procedures for the safe handling of radioactive materials when working on the GC parts that come into direct contact with the ECD.

5.3 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Serious or Significant Hazard Rating

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hydrogen gas	Explosive	None	The main hazard is flammability. Exposure to moderate concentrations may cause dizziness, headache, nausea, and unconsciousness. Exposures to atmospheres less than 8 to 10% oxygen will bring about sudden unconsciousness, leaving individuals unable to protect themselves. Lack of sufficient oxygen may cause serious injury or death.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain of the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

A gas chromatographic system with dual columns and dual ECD (^{63}Ni) detectors, and a data system capable of measuring peak area and/or height. The current instruments that are in use for this analysis are HP6890N-instrument P3 and HP5890 Series II- instrument W. Instrument R- HP5890 Series II may be used on occasion.

6.2 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Columns

6.3.1 Primary Column: Rtx CLP Pest, 30 m x 0.32 mm id, 0.5 µm coating.

6.3.2 Secondary Column: Rtx CLP Pest II, 30 m x 0.32 mm id, 0.25 µm coating.

6.3.3 Additional columns that can be used for confirmation include 30m x 0.32mm id HP-5 or HP-1701.

6.4 Supplies

6.4.1 Autosampler vials, crimp caps with PTFE-faced septa.

6.4.2 Y-splitter, septa, guard columns, ferrules, Agilent deactivated injection port liners, Siltek glass wool.

6.4.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4.4 Various class A volumetric flasks from 5 mL to 250 mL.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.2 Hexane, pesticide grade. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen

7.2 Stock Standards

7.2.1 All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.2.2 All standards must be refrigerated at 0-6 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before use.

7.2.3 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier if the vendor indicates an earlier date.

7.2.4 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner if comparison with check standards indicates a problem.

7.3 PCB and Surrogate Stock Calibration Standards

7.3.1 Stock A (AR_(*Aroclor #*)_RES

For each of the Aroclors listed in Tables 1 and LVI-1, a commercially prepared stock standard solution is obtained. Each stock standard contains the specific Aroclor in pesticide-grade hexane (or in some cases, isooctane) at a concentration of 1,000 µg/mL. The current vendor is RESTEK (PCB/Catalog#) AR1016/32006, AR1221/32007, AR1232/32008, AR1242/32009, AR1248/32010, AR1254/32011, AR1260/32012, AR1262/32409, and AR1268/32410, other vendors may be used.

Surrogate Stock B (AR_SURR_RES)

A commercially prepared stock standard solution is obtained that contains the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in acetone, at a concentration of 200 µg/mL. The current vendor is RESTEK catalog #32000, other vendors may be used.

Internal Standard Stock

A commercially prepared stock standard solution is obtained that contains the internal standard 1-bromo-2-nitrobenzene in acetone, at a concentration of 1000 µg/mL. The current vendor is RESTEK catalog #32279, other vendors may be used.

7.3.2 PCT Stock

A commercially prepared stock standard solution is obtained that contains the individual PCT compounds at a concentration of 35 ug/mL in hexane. The current vendor is Accustandard and the catalog numbers are AR 5432 T432S, AR 5442 T442S, and AR 5460 T460S. Equivalent standards from other vendors and at other concentrations may be used.

7.4 Intermediate and Working Level Calibration Standard Solutions

7.4.1 Stock C (Level 7 Calibration) Standard Solutions

(AR_*(aroclor #)***_L***(level)*

A Stock C standard solution is prepared for the various Aroclors or combination of Aroclors as summarized in the following table. In each case, the Stock C standard solution is also the highest concentration (i.e., Level 7) calibration standard.

Stock C (Level 7)	Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_1660	0.1 mL of Aroclor 1016 Stock A	1000	100	Aroclor 1016	1.0
	0.1 mL of Aroclor 1260 Stock A	1000		Aroclor 1260	1.0
	0.025 mL of surrogate Stock B	200		TCMX	0.05
				DCB	0.05
AR_2154	0.1 mL of Aroclor 1221 Stock A	1000	100	Aroclor 1221	1.0
	0.1 mL of Aroclor 1254 Stock A	1000		Aroclor 1254	1.0
AR_3262	0.1 mL of Aroclor 1232 Stock A	1000	100	Aroclor 1232	1.0
	0.1 mL of Aroclor 1262 Stock A	1000		Aroclor 1262	1.0
AR_4268	0.1 mL of Aroclor 1242 Stock A	1000	100	Aroclor 1242	1.0
	0.1 mL of Aroclor 1268 Stock A	1000		Aroclor 1268	1.0
AR_1248	0.1 mL of Aroclor 1248 Stock A	1000	100	Aroclor 1248	1.0

ARHVI_*(aroclor #)***_L***(level)*

Stock C (Level 7)	LVI Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_1660	0.05 mL of Aroclor 1016 Stock A	1000	100	Aroclor 1016	0.5

Stock C (Level 7)	LVI Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
	0.05 mL of Aroclor 1260 Stock A	1000		Aroclor 1260	0.5
	0.0125 mL of surrogate Stock B	200		TCMX	0.025
				DCB	0.025
AR_2154	0.05 mL of Aroclor 1221 Stock A	1000	100	Aroclor 1221	0.5
	0.05 mL of Aroclor 1254 Stock A	1000		Aroclor 1254	0.5
AR_3262	0.05 mL of Aroclor 1232 Stock A	1000	100	Aroclor 1232	0.5
	0.05 mL of Aroclor 1262 Stock A	1000		Aroclor 1262	0.5
AR_4268	0.05 mL of Aroclor 1242 Stock A	1000	100	Aroclor 1242	0.5
	0.05 mL of Aroclor 1268 Stock A	1000		Aroclor 1268	0.5
AR_1248	0.05 mL of Aroclor 1248 Stock A	1000	100	Aroclor 1248	0.5

7.4.2 AR_1660 Calibration Levels

A total of 7 calibration standards are prepared for AR_1660 as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the Aroclors, but only the AR_1660 calibration standards include the surrogates. In all cases, measured volumes of the Stock C standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Stock C Used (mL)	Final Volume (mL)	Final PCB Conc (µg/mL)	Final Surrogate Conc (µg/mL)*
1	0.25	10	0.025	0.00125
2	0.5	10	0.050	0.0025
3	1.0	10	0.10	0.005
4	2.5	10	0.25	0.0125
5 (CCV)	5.0	10	0.50	0.025
5 (CCV) 1660 mix only	12.5	25	0.5	0.025
6	7.5	10	0.75	0.0375
7 (Stock C)	--	--	1.0	0.0500
* Surrogates are in the AR_1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				
LVI Level	**Vol of LVI Stock C Used (mL)**	**Final Volume (mL)**	**Final PCB Conc (µg/mL)**	**Final Surrogate Conc (µg/mL)***
1	0.25	10	0.0125	0.00063
2	0.5	10	0.025	0.00125
3	1.0	10	0.05	0.0025
4	2.5	10	0.125	0.0625

5 (CCV)	5.0	10	0.25	0.0125
5 (CCV) 1660 mix only	12.5	25	0.25	0.0125
6	7.5	10	0.375	0.0188
7 (Stock C)	--	--	0.50	0.0250
* Surrogates are in the AR_1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				

7.4.3 Working Single-Point PCB Calibration Standards

The Level 5 standard in the table above is used for single-point calibrations of the individual Aroclors. These standards are also used as pattern recognition standards.

7.4.4 Polychlorinated Terphenyl Calibration Levels

A total of 7 calibration standards are prepared for PCTs as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the PCTs. The level 7 standard is prepared from the stocks described in section 7.3.3 by diluting 1 mL of the stock to 35 mL final volume with hexane. The final concentration of the level 7 standard is 1.0 ug/mL. In all cases, measured volumes of the Level 7 standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Level 7 Used (mL)	Final Volume (mL)	Final PCT Conc (µg/mL)
1	0.25	10	0.025
2	0.5	10	0.05
3	1	10	0.10
4	2.5	10	0.25
5 (CCV)	5	10	0.50
6	7.5	10	0.75

7.5 Second-Source Standards for Initial Calibration Verification (ICV)

These standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.5.1 Second-Source Stock A' Aroclor Standard Solutions (AR_*(aroclor#)*_RESss

Commercially prepared solutions in pesticide-grade hexane (or isooctane) are routinely obtained for Aroclors 1016 and 1260. The Aroclor concentration in each solution is 1000 µg/mL. A second source may be obtained for the other Aroclors, if necessary. The current second source is RESTEK (PCB/Catalog#) AR1221/32007.sec; AR1016/1260/32039.sec; AR1232/32008.sec; AR1242/32009.sec; AR1248/32010.sec;

AR1254/32011.sec; AR1262/32409.sec; AR1268/32410.sec, other vendors may be used.

Second source surrogate

A commercially prepared solution is obtained containing TCMX and DCB each at a concentration of 200 ug/mL. The current second source surrogate is Restek 32000.

Working Level ICV AR_*(aroclor#)*_ICV

The working level second source ICV standard is prepared by combining 0.025 mL of Aroclor 1016 Stock A', 0.025 mL of Aroclor 1260 stock A', and 0.00625 mL of the surrogate second source stock and diluting to a final volume of 100mL with pesticide grade hexane. This results in a concentration of 0.25 ug/mL for each of the Aroclors and 0.0125 ug/mL for each surrogate. If a second source verification standard is prepared for any of the Aroclors other than the AR_1660 mixture, the surrogates are not added to that mix.

7.5.2 PCT Second Source Stock and working level.

7.5.2.1 A commercially prepared solution of each of the PCT mixes is obtained from a different vendor and typically prepared at a concentration of 100 ug/mL in hexane. The current second source vendors and catalog numbers are AR 5432 Chem Services F290RPS, AR 5442 Chem Services F860RPS, and AR 5460 Chem Services F292RPS. Other vendors may be used.

7.5.2.2 The working level PCT standard is prepared at a concentration of 0.25 ug/mL by diluting 0.025 mL of each stock to a final volume of 10 mL.

7.6 Continuing Calibration Verification Standard (CCV), 0.5 µg/mL

The working CCV solution is the same as the Level 5 initial calibration standard, as shown in the table in Section 7.4.2.

7.7 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL Standard.

7.8 Laboratory Control Standard (LCS) Spiking Solution (AR1660)

NOTE: The LCS/MS spiking solution (8082LCS) is prepared and used as part of the scope of the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015, and DV-OP-0016. The following information is provided for reference only.

In general the LCS is made up at a concentration of 2 µg/mL in a water-soluble solvent such as acetone. For oil samples extracted by waste-dilution, the standard

is made in hexane. The standard contains Aroclors 1016 and 1260 only. Typically 1 mL of this standard is added to 1 liter of water samples, 30 g of soil samples, or 1 g of oil samples. The current LCS vendor is Restek at a concentration of 1000ug/ml. The solution is prepared by diluting 0.5 ml of this stock into 250 ml with acetone solvent.

7.9 Matrix Spike (MS) Spiking Solution:

The working matrix spike solution is the same as the LCS spike solution. Matrix spike samples are prepared by adding 1.0 mL of the working solution to a second one-liter aliquot of the selected aqueous sample, or to a 30-gram subsample of the selected soil sample. The MS duplicate (MSD) is prepared in the same way using a third aliquot of the selected sample.

7.10 Surrogate Spike Solution

7.10.1 Stock Surrogate Spike Solution:

A commercially prepared solution containing 200 µg/mL each of decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) in acetone is purchased. The current source is Restek 32000.

7.10.2 Working Surrogate Spike Solution (8081/82 Surr)

NOTE: Samples are spiked with the surrogate compounds during sample preparation, which is described in the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015 and DV-OP-0016. The following information is provided for reference only.

The working level surrogate solution is made up to contain DCB and TCMX at a concentration of 0.2 µg/mL. For water and soil samples the solution is made in a water-soluble solvent like acetone. For all oil samples extracted by waste dilution the solution is made in hexane.

7.11 Internal Standard Spiking solution 1 µg/mL (BNB IS)

An intermediate stock (BNB stock) at 10 µg/mL is prepared by diluting 1 mL of the commercial Internal Standard Stock from Section 7.3 to a final volume of 100 mL in hexane. The Internal standard spiking solution is then prepared by diluting 10 mL of this intermediate to a final volume of 100 mL. Every standard, QC sample, and client sample is spiked with 0.1 mL of the internal standard spiking solution into 1 mL. For the LVI preparation 0.05 mL of internal standard is spiked into 1 mL of sample or standard.

7.12 Primer Mix

The primer mix typically consists of a mixture of CCV standards and/or old calibration standards. The concentrations of the components of the primer mix are not critical. The primer mix is injected one or more times prior to analyzing standards and samples to ensure that the chromatographic system is stable, i.e., that retention times are reproducible.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[2]	Reference
Water[1]	Amber glass	1 Liter	Cool, ≤ 6°C	1 Year to extraction 40 days to analysis	SW-846
Water[3]	Amber glass	250 mL	Cool, ≤ 6°C	1 Year to extraction 40 days to analysis	SW-846
Solid	Glass	8 oz	Cool, ≤ 6°C	1 Year to extraction 40 days to analysis	SW-846

[1]To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

[2]California, Connecticut, Pennsylvania and South Carolina do not allow the 1 year holding time. For work performed in these states, the extraction holding time is 7 days for water and 14 days for solid.

[3]Samples collected in 250 mL bottles will be extracted by SW846 method 3510 followed by analysis using the LVI procedure described in this SOP.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD/DOE QSM 5.0or 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as

appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). The current MDL value is maintained in the TestAmerica Denver LIMS. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on an instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. A batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The minimum batch QC in each run is an acceptable method blank or instrument/calibration blank. See QA Policy DV-QA-003P for further details. For DoD QSM 4.2 or DOD/DOE QSM 5.0 or 5.1, the MS/MSD must be from the project site and if insufficient sample to analyze the MS/MSD pair is available, this is documented in an NCM and an LCSD is performed.

9.4 Method blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water (for aqueous sample batches) or Ottawa sand (for solid sample batches) to which the surrogate compounds are added. The method blank is subject to the entire extraction and analysis process. The method blank must be analyzed on every instrument that is used to analyze the samples from the batch.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (RL) or above one-tenth of the concentration found in the associated samples. See section 9.7 for further evaluation of the surrogate in the MB.

Corrective Action: If the method blank exceeds allowable levels, the source of the contamination must be investigated and all associated samples that produced detections for the contaminant must be re-extracted and reanalyzed. Any samples that produce concentrations more than 10 times the concentration of the same compound as the blank contaminant may be reported with proper flagging and narration. Method Blanks for South Carolina compliance work MUST be below the RL.

9.5 Laboratory Control Sample (LCS)

One LCS is prepared and analyzed with each batch of samples. The LCS is prepared as described in Section 7.8. The LCS is subject to the entire extraction and analysis process.

Acceptance Criteria: The LCS recovery must be within the established control limits. The laboratory's standard control limits are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Current control limits are maintained in LIMS. South Carolina requires the LCS to recover within limits of 70-130%.

Corrective Action: If recoveries are not within the established limits, the analytical system is out of control and corrective action must occur. All associated samples must be re-extracted and reanalyzed. If the LCS exceeds the upper control limit then all samples that do not contain detections for the affected compound may be reportable with client consent and proper flagging and narration. Note that exceptions for an out of control LCS are not allowed for DoD/DOE QSM 5.0 or 5.1 projects unless prior approval is indicated in the projects instructions.

9.6 Matrix Spike (MS) and Matrix Spike Duplicate Samples (MSD)

One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require preparation and analysis of an MS/MSD pair at a 10% frequency. Preparation of the MS is described in Section 7.9. The MSD is another aliquot of the sample selected for the MS that is spiked in the same manner as the MS. For DoD QSM 4.2 or DOD/DOE QSM 5.0 or 5.1, the MS/MSD must be from the project site and if insufficient sample to analyze the MS/MSD pair is available, this is documented in an NCM but no LCSD is performed.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD/DOE QSM 5 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in

order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client’s needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.7 Surrogates

Each field sample, QC sample, and each calibration standard that is used for the AR_1660 initial calibration, is spiked with surrogate compounds decachlorobiphenyl (DCB) and trichloro-m-xylene (TCMX). The surrogate spike solution is prepared as described in Section 7.10.

Acceptance Criteria: The surrogate recoveries must be within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise.

Corrective Action: If recoveries of the surrogates in blanks are outside of the control limits, check for calculation or instrument problems. High recoveries might be acceptable if the surrogate recoveries for the samples and other QC in the batch are acceptable. Low surrogate recoveries in the blank require re-extraction and reanalysis of the associated samples especially those that have detections for the targeted compounds that are found in the blank. Samples that are ND and have the surrogates in control may be reportable with proper flagging and NCM.

For field samples, surrogate recovery is calculated and reported for DCB only. TCMX may also be added. However, if both surrogate compounds are added, and recoveries calculated, and either surrogate fails to fall within the control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). Samples with surrogate recoveries that are above the upper control limit may be reportable with flagging and narration if they do not have reportable detections. See SOP DV-QA-003P for further evaluation.

If matrix interference is not obvious from the initial analysis, it is only necessary to re-extract and reanalyze a sample once to demonstrate that poor surrogate recovery

is due to matrix effects, as long as the extraction/instrument system is proven to be working properly.

9.8 Internal standard

Acceptance Criteria: The internal standard recoveries must be within -50% to 200% for standard solutions of the recovery established by the midpoint of the ICAL or the opening CCV for the run on days when an ICAL is not analyzed.

DOD/DOE QSM 5.0 does not have criteria for internal standards. The above criteria is used. DOD/DOE QSM 5.1 uses the above criteria.

Corrective Action: If the internal standard response is outside of this range then the samples must be diluted until the recoveries are in control. Failure to meet this criteria in a CCV requires reanalysis of the standard and all affected samples analyzed in the bracket previous to the standard and after the standard. Recalibration is necessary if control cannot be established. For DOD/DOE QSM 5.1, if corrective action fails in field samples, the data must be qualified and explained in the case narrative. Flagging is not appropriate for failed standards.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Tables 2 and LVI-2 for typical operating conditions. The conditions described in Table LVI-2 are to be used when performing the large volume injection approach.

10.1.2 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the Chrom worklist to set up the analytical sequence.

10.1.3 Transfer unprocessed calibration data to the Chrom database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration checklist is scanned and stored in the documents section of each analytical batch.

10.2 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns.

10.3 Initial Calibration (ICAL)

10.3.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003 *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

10.3.2 An internal standard calibration using seven concentration levels of the AR_1660 mixture is routinely performed. (At least five calibration levels are required.) This provides concentration levels for Aroclor 1016, Aroclor 1260, and the surrogate compounds DCB and TCMX.

10.3.2.1 Prior to the analysis of the ICAL the resolution for the triplet towards the end of the Aroclor 1260 chromatogram for peaks 1&2 and peaks 2&3 must be <75% on one of the two columns used for the analysis.

10.3.2.2 See Tables 3 and LVI-3 for Calibration Levels. Calibration levels defined in Table LVI-3 are appropriate when the large volume injection approach is used.

10.3.2.3 The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes. This run is not part of the actual calibration.

10.3.2.4 Prior to analysis of the initial calibration standards it is recommended that a chlorinated pesticide standard (Method 8081) be analyzed as a locater standard to identify potential interferences in samples due to the presence of chlorinated pesticides.

10.3.3 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.3.4 The calibration curves for Aroclors 1016 and 1260 and the surrogate compounds are modeled either as average response factors or as calibration curves using a systematic approach to selecting the optimum calibration function.

10.3.5 The calibration for each of the other Aroclors (see Table 1 or LVI-1) is initially determined using a single, mid-level calibration standard. As needed, the laboratory may generate a multi-point calibration for other commonly detected Aroclors, such as 1221, 1254, and 1248. When additional multi-point calibrations are developed for the other Aroclors, a second-source ICV standard is also analyzed. When Aroclors are detected, they must be re-analyzed after an acceptable calibration curve (minimum five points) and its verification (ICV) are obtained.

NOTE: Samples from sites known to be contaminated with specific Aroclors should be analyzed using a multi-point calibration curve

for the identified Aroclors. This information is provided to the analyst through special instructions in LIMS.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of the preparation of the calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed

10.3.6 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.3.6.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.3.6.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.3.6.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.3.6.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

10.3.6.5 If a data point is rejected, it must be documented on the calibration checklist, in the sequence log and on an NCM which is filed with each project that is reported from the calibration.

10.3.7 The high and low standard for the initial calibration of the AR_1660 mixture defines the acceptable quantitation range for all of the Aroclors. The low calibration standard must be at or below the RL. If a sample extract contains any Aroclor at a concentration that exceeds the upper range of the calibration, then the extract must be diluted with hexane and reanalyzed.

10.3.8 Select 5 major peaks in the analyte pattern (only 3 peaks are usable for Aroclor 1221). The peaks that are chosen should have responses that are at least 25% of the response for the largest peak in the Aroclor pattern, with the exception of Aroclor 1268 where the requirement is 10%. Try to include one peak that is unique (differs in size or location relative to the other common Aroclors) to the Aroclor being quantitated. Calculate the response of each of the major peaks for each Aroclor, and use these responses independently, averaging the resultant concentrations found in

samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e., peaks that are significantly larger than would be expected from the rest of the pattern).

NOTE: A minimum of three accurate peaks must be used to quantify an Aroclor (two for Aroclor 1221).

10.4 Internal Standard Calibration

Internal standard calibration involves the comparison of an instrument response (e.g., peak area or peak height) from the target compound in the sample to the response of the internal standard compound, which is added to the sample or sample extract prior to injection. The same concentration of internal standard is added to each initial calibration standard. For each calibration level, the response factor, RF, is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$ *Equation 1*

Where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

10.5 Establishing the Calibration Function

Calibrations are modeled either as average response factors or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until calibration acceptance criteria are met. For dual column analysis the initial calibration criteria must be met on both columns.

10.5.1 Linear Calibration Using Average Response Factor

The response factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

NOTE: For samples requiring adherence to SW 846 Methods 8000B and 8000D (including South Carolina compliance work), forcing through zero is NOT allowed.

10.5.1.1 The average response factor is calculated as follows:

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$ Equation 2

Where:

RF_i = The response factor for the i^{th} calibration level.
n = The number of calibration levels.

10.5.1.2 The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \text{Equation 3}$$

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}} \qquad \text{Equation 4}$$

10.5.2 Evaluation of the Average Response Factor

Plot the calibration curve using the average RF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD must be ≤ 20%. SW-846 Method 8000B allows evaluation of the grand average across all compounds, but some programs (e.g., DoD/DOE QSM, Arizona and South Carolina require evaluation of each compound individually). Check project requirements.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.5.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated is the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

Equation 5

$$y = ax + b$$

Where:

y = Instrument response (peak area or height).
x = Concentration of the target analyte in the calibration standard.
a = Slope of the line.
b = The y-intercept of the line.

For an internal standard calibration, the above equation takes the following form:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b \qquad \textit{Equation 6}$$

To calculate the concentration in an unknown sample extract, the regression equations 5 and 6 are solved for concentration, resulting in the following equations, where x and C_s are now the concentration of the target analyte in the unknown sample extract:

$$x = \frac{y - b}{a} \qquad \textit{Equation 7}$$

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a} \qquad \textit{Equation 8}$$

10.5.4 Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit (RL), and preferably less than the MDL.

Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.

The linear regression must have a correlation coefficient (r) ≥ 0.99. Some programs (e.g., DoD QSM 4.2) require a correlation coefficient ≥ 0.995. DoD/DOE QSM 5.0 and 5.1 require r^2 >0.99.

Corrective Action: If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.

10.5.5 Second-order regressions and polynomial regression fits of third order or higher are not allowed for this method.

10.5.6 Acceptance criteria independent of calibration model

10.10.1.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.

$$\%\ Error = \frac{xi - x\prime i}{xi} x\ 100$$

Where: $x'i$ = measured amount of the analyte at calibration level *i* in mass or concentration units and xi = the true amount of the analyte at calibration level *i* in mass or concentration units.
% Error should be ≤ 30%. For some data uses ≤ 50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error:

$$RSE = 100\,x\sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where: *Xi* = True amount of the calibration level I, in mass or concentration units
X'i = measured amount of analyte in calibration level I, in mass or concentration units
p = number of terms in the fitting equation (average = 1), linear – 2, quaratic – 3)
n = number of calibration points
RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. IF not defined in the method, use ± 20%.

10.6 Second-Source Initial Calibration Verification (ICV)

An ICV is prepared for each multipoint calibration. The stock standards are obtained from a source different than that of the standards used for the calibration. The preparation of the ICV standard is described in Section 7.5. The concentration of each Aroclor in the ICV is 0.25 µg/mL; the concentration of each surrogate is 0.125 µg/mL. The ICV standard is analyzed immediately following the completion of the initial calibration. If any changes are made to the calibration curve types then the ICV must be recalculated to the final form of the ICAL.

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 15% of the expected value. Method 8082A and DOD/DOE QSM 5.0 and 5.1 allow a control of ± 20%.

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.7 Continuing Calibration Verification (CCV), 0.50 ug/mL, LVl 0.25 ug/mL.

10.7.1 12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration sequence.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.7.2 The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7). At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCSs, and method blanks. It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. Some programs, specifically DoD/DOE QSM 5.0 and 5.1, require a CCV after every 10 field samples and at the end of the run. If 12 hours elapse, analyze the 12-hour standard sequence instead. Unless the program specifically requires a closing CCV (as above for DOD and DOE), a closing CCV is not required when using an internal standard. Check the projects requirements regarding the closing CCV.

If an aroclor other than 1016 or 1260 is identified in any sample then a CCV for the identified aroclor must be analyzed within 12 hours of the samples analysis. This CCV must meet proper CCV criteria.

For DOD/DOE QSM 5.0 or 5.1, the CCVs for aroclors other than 1016 and 1260 are only required before sample analysis.

10.7.3 RL Standard

It may be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method between every 10 sample injections (see Section 7.7). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit and the samples contain no analytes above the reporting limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria or in the case of matrix effect to confirm the ability to see at the reporting limit.

NOTE: This procedure is not used when Method 8000C/D or DOD/DOE QSM 5.0 or 5.1 are required.

10.7.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.7.4.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 15%. Method 8082A and DOD/DOE QSM 5.0 and 5.1 require control of ± 20%. If a confirmation column is required (see Section 12.5), the CCV criteria must be met on both columns in order to confirm a detection. Both surrogates must pass in the CCV.

In some cases, the nature of the samples being analyzed may be the cause of a failing %D. When the %D for an analyte falls outside of acceptance criteria in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect; if so desired by the client. Dilution will typically be required to meet DOD 4.2 or DoD/DOE QSM 5.0 or 5.1 criteria.

Refer to Section 12 for which result to report.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \text{Equation 8}$$

10.7.4.2 Analytes Not Detected (< RL)

For any analyte not detected (ND) in client samples, the %D for that analyte in the bracketing CCVs should also be within acceptance criteria.

However, if the CCV %D exceeds the upper control of the acceptance criteria and the sample results are ND, it still may be possible to report sample results. In this case, the client should be consulted and an NCM written. Note that exceptions to an out of control CCV are not allowed for DoD/DOE QSM 5.0 or 5.1 unless prior approval is noted in the project instructions.

If the CCV %D falls below acceptance criteria and sample results are ND, but the target analytes are detected in the RL Standard, it may still be possible to report sample results, since the detection of the analyte(s) in the RL Standard indicate that there was sufficient sensitivity to detect the analyte(s) in the samples. In this case, the client should be consulted and an NCM written. This would only be used in cases where the matrix is affecting CCV recovery and dilution of the affected sample(s) is not an alternative.

DoD/DOE QSM 5.0 or 5.1 requires recalibration and reanalysis of all affected samples since the last acceptable CCV. As an alternative, the laboratory may analyze two additional consecutive CCVs within one hour of the failed CCV. If both pass, then the samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate: then reanalyze all affected samples since the last acceptable CCV.

If a DoD or DOE client accepts TestAmerica's Technical Specifications for DoD/DOE QSM work, samples that have no detections when a CCV has recoveries above the project acceptance limits would be reported with a case narrative comment, in addition to applying any data qualifier flags required by the project.

10.8 Retention Time (RT) Windows

10.8.1 Determine the retention time (RT) windows for the 5 major peaks selected for each Aroclor (3 peaks for Aroclor 1221). The AR1016 windows will be used to establish retention time windows for AR1221, AR1016, AR1232, AR1242, and AR1248. The AR1260 windows will be used to establish retention time windows for AR1254, AR1260, AR1262, and AR1268.

10.8.2 Determine new RT windows each time a new column is installed or annually.

10.8.3 Evaluate the deviation from expected retention time for each analyte in at least three CCV and/or LCS samples spread over at least 72 hours.

10.8.4 If three days of analytical data are not available, use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

10.8.5 Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at 0.01 min. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.

NOTE: For the Aroclors, the maximum deviation must be evaluated for each of the 3-5 major peaks used for sample calculations.

10.8.6 Retention time windows for analytes of interest must not overlap.

10.9 Ongoing Evaluation of Retention Time Windows

10.9.1 Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

10.9.1.1 Matrix spike analytes may fall outside the retention time window if there is a large non-target pak coeluting with the analyte in the matrix spike.

10.9.2 If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

10.9.3 Retention time windows should be reliably narrower than ± 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained. Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. 8000C and 8000D also require a new study when the column is clipped during maintenance.

10.9.4 Sample Retention Time Criteria

The surrogate must fall within the established RT window. Target analyte peaks must be within the established RT window to be reported as such. If the surrogate RT indicates a RT shift, it may be possible to accept a target analyte peak if it has shifted in the same direction as the surrogate peak. The presence of a definitive aroclor pattern will be positive evidence

of a hit and may supersede RT window criteria. An NCM should be written to explain this case.

10.9.5 Daily Retention Time Windows

The centers of the retention time windows are adjusted at the beginning of each analytical sequence based on the daily initial CCV or the RT marker mix.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006.

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007. Note that it is highly recommended that all samples be checked for sulfur and cleaned up if necessary before the samples are analyzed on the instrument. Sulfur can contaminate the column and hinder the quantification of certain compounds.

11.3.4 For water samples, a 1 L extraction is brought to a final extract volume in hexane of 10 mL. For water samples extracted from a 250 mL volume and analyzed by the LVI method the final volume is 5 mL.

11.3.5 For solid samples, a 30 g sample is brought to a final volume of 10 mL of hexane.

11.3.6 Wipe samples are processed as a modified soil and the final volume of these extracts is 10 mL of hexane.

11.3.7 Oil samples are processed as a waste. Typically 1 g of oil is brought to 10 mL with hexane.

11.3.8 Use hexane to dilute sample extracts, if necessary.

11.4 Instrument Troubleshooting and Maintenance

Before the start of any daily sequence the instrument system should be evaluated for possible maintenance.

11.4.1 If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control.

11.4.2 The injector septum should be changed after about 200 injections have been completed.

11.4.3 If the last CCV that was analyzed indicated a high response then a simple liner change is typically sufficient to bring the system back into control.

11.4.4 Analysis of a few solvent blanks or a system bake out may be necessary to drive out any residual contamination on the column.

11.4.5 A reduced response may indicate that the system needs to be evaluated for leaks.

11.4.6 Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated. The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed.

11.4.7 Re-calibration should not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

11.5 Gas Chromatography

Chromatographic conditions for this method are presented in Tables 2 and LVI-2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the Chrom DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the Chrom DB software.

11.6 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. For LVI analysis 2 µL of sample extract is introduced into the chromatographic system. Samples, standards, and QC samples must be introduced using the same procedure. Use the Chrom worklist to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.7 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Tables 3 and LVI-3 for the calibration levels

used.

11.7.1 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.7.1) and updating the retention time windows (Section 10.8.7)

11.7.2 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification. Any samples that were not bracketed by a closing CCV (when required) must be reanalyzed in the new 12 hour sequence.

11.7.3 The following is a typical analytical sequence for routine sample analysis:

- Primer (Injection of any standard that contains any of the analytes to establish the stability of the chromatographic system.)
- RT marker mix.
- Hexane instrument blank.
- Daily initial CCV (Unless an ICAL is performed, which is immediately followed by the second-source initial calibration verification.)
- 10 sample injections (The first set of samples analyzed usually includes the method blank and the LCS, and may include matrix spikes.)
- CCV
- Followed by cycles of 10 sample injections and a CCV, as needed
- Closing CCV, instrument blank, and RL Standard

11.8 Retention Times

The centers of the RT windows determined in section 10.8 are adjusted to the RT of each individual peak as determined in the 12-hour calibration verification. The RT window must be updated at the beginning of each analytical sequence.

11.9 When a sample result exceeds the upper calibration range, then that sample extract is diluted to obtain a result in the upper half of the calibration range and reanalyzed. Any samples that were analyzed immediately following the high sample are evaluated for carryover. Typically carryover is not observed until the sample concentration is well above the upper calibration level. If the samples had target analyte detections at or above the RL, the samples must be reanalyzed to rule out carryover.

11.10 Upon completion of the analytical sequence, transfer the raw chromatography data to the Chrom database for further processing. Review chromatograms online and determine whether manual data manipulations are necessary. All manual integrations must be justified and documented. See DV-QA-011P for requirements for manual integration. Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. Alternatively, the manual integration may be processed manually. In the latter case, print both the both the

before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration. These chromatograms must be scanned into the documents section of the LIMS system.

11.11 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.11.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork.

11.11.2 Perform a level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.11.3 Submit the data package and review checklist to the Data Review Group for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the level 2 review checklist. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Calculations / Data Reduction

12.1 Detailed equations can be found in the Corporate SOP CA-Q-P-003 *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

12.2 Qualitative Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram. A NCM must be written to explain the deviation from the expected RT criteria. Project specific allowance for DoD/DOE QSM 5.0 or 5.1 is required for the TestAmerica approach to the RT shift for aroclors such that the retention time shift occurs at a later point in the sequence. Samples with detections must be reanalyzed after the analysis of a retention time marker for any aroclors that are detected and show a RT shift unless project specific requirements allow for reporting data with a NCM.

12.3 Quantitation of Aroclors

Quantitation of Aroclors is accomplished using 5 major peaks (3 peaks for Aroclor 1221). If there is an interference that affects the accuracy of results, the analyst

may use as few as 3 major peaks (2 peaks for Aroclor 1221). The same peaks that are used for sample quantitation must be used for standards and QC quantitation.

12.4 Second column confirmation of Aroclors is performed only when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: DoD/DOE QSM 5.0 and 5.1 projects require the use of second-column confirmation of Aroclors unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

NOTE: South Carolina requires second column confirmation.

NOTE: Method 8082A indicates that second column confirmation is necessary when the sample composition is not well characterized.

12.5 Dual Column Quantitation

NOTE: This section is included for those clients/projects that require dual column confirmation.

12.5.1 A primary column is designated when dual column analysis is performed. If the continuing calibration fails for one of the columns then the appropriate corrective action must be taken. The result from the primary column is normally reported. The result from the secondary (confirmatory) column is reported if any of the following is true:

12.5.1.1 There is obvious chromatographic interference on the primary column.

12.5.1.2 The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident on the primary column.

12.5.1.3 For DoD QSM 4.2 or DOD/DOE QSM 5.0 work, calibration and QC criteria for the second column are the same as for the initial or primary column analysis.

12.5.2 Dual Column Results With > 40% RPD

12.5.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram. Method 8000C/D requires that the lower result be reported. For DOD/DOE QSM 5.0 or 5.1, use project-specific or method requirements to determine reporting convention. If none specified, report from the primary column.

12.5.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then

report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.5.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.5.2.4 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.5.3 Reporting Total Aroclors for dual column reporting or from multiple runs.If dual column quantitation is requested or if the results from the individual Aroclors are in multiple runs, then total aroclors will be calculated from the primary results only using the TALS method code Total_PCB.

12.6 Surrogate Recovery

12.6.1 Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB).

12.6.2 In cases where the addition of the surrogate tetrachloro-*m*-xylene (TCMX) is required, its recovery is calculated and reported. In cases where both surrogates are added and recoveries calculated, the recovery of each surrogate is evaluated and corrective action must be taken if either surrogate recovers outside of the established control limits and matrix interference is not evident. Depending on project requirements, corrective action may be necessary only if DCB and TCMX are both outside of acceptance limits.

12.7 Calibration Range and Sample Dilutions

12.7.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for any analyte, they must be reanalyzed to rule out carryover unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when blanks or other samples were analyzed after a sample with similar high concentration or when the detection in the sample with suspected carryover is much higher than the expected amount of the

carryover (i.e., the suspect sample's concentration is similar to or higher than the sample run previous to it). It may also be necessary to dilute samples because of matrix interferences.

12.7.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.7.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.7.4 Reporting Dilutions

Some programs (e.g., South Carolina, DoD and DOE) and some projects require reporting of multiple dilutions (check Method Comments in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.8 Interferences are Observed in Samples

12.8.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.8.2 Suspected Negative Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.8.3 Suspected Positive Interferences

If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option. At a minimum, the Data Review Template prepared by the analyst should include the following comment for inclusion in the case narrative:

"Based on review of the chromatograms for samples _____, it is my opinion that the evident interferences may be causing false results.

Date _____ Analyst _____"

12.9 Identifying and Reporting PCBs

12.9.1 In samples where the PCB pattern matches an individual Aroclor reasonably well, the samples should be quantified and reported as usual. When there are numerous PCB peaks present but there are no good matches to any individual Aroclor, choose the Aroclor (or Aroclors) that most closely match the sample and quantify the peaks as that Aroclor. The sample should not be reported as "not detect" based solely on the absence of a good match to a single Aroclor mixture. Multiple Aroclors should only be reported if their patterns are reasonably well separated. For example, 1232 and 1254 could be reported together, but not 1242 and 1248. See Attachment 1 for additional information on identifying Aroclors.

NOTE: When reporting and quantifying PCBs that do not closely match an Aroclor standard, it is absolutely essential and mandatory that this is explained in the report narrative. The analyst must write a NCM to explain their judgment for reporting PCBs in these instances.

12.9.2 Some example text that can be used in the report narrative is presented below:

Sample XXXX appears to contain PCBs based on the presence of numerous PCB peaks. However, due to weathering or other environmental processes, the PCBs in the sample do not closely match any of the Aroclor standards we use to calibrate our instruments. We quantified and reported the sample as Aroclor ZZZZ (or as a mixture of Aroclors ZZZZ and YYYY). Due to the poor match with the Aroclor standard(s), there is increased qualitative and quantitative uncertainty associated with this result. This approach is consistent with the guidance in section 7.9.3 of SW846 method 8082A. If these results do not meet the needs of your project then we would suggest a further analysis of the sample. Depending on the objectives, this may include congener-specific analysis by 8082A; or analysis by a more specific method (e.g., method 1668 or an adaptation of method 8270) for PCB congeners or PCB homolog totals.

12.9.3 Sample Matrix Issues

12.9.3.1 In some cases when analyzing for multi-component analytes, the sample matrix is so complex that it would obliterate any possible pattern that would allow us to identify the analyte. When this happens, it is true that the analyte is not detected at the normal detection limit. However, it is true that we could not have detected the analyte at the normal detection limit. Even if the analyte was present, we would not be able to recognize it.

12.9.3.1 When this occurs, the sample must be analyzed at a dilution that would allow us to detect the analyte, and the reporting limit should be the one appropriate for that dilution. Reporting a non-detect at the normal reporting limit is not an acceptable practice.

12.9.3.2 Some clients may insist on ND reporting if the patterns are not clear. In that event, add information to the project file to indicate that the information in this guidance have been communicated to the client, together with the client's instructions. In addition, in the event of a poor match to patterns, a narrative comment as suggested in the previous paragraph is still required.

12.9.4 Background on PCBs

12.9.4.1 PCBs were widely used in a variety of products prior to being banned in the 1970's. The most common usages were in electric motors and transformers. They were manufactured by gas phase chlorination of a biphenyl molecule. The nomenclature, in general, describes the weight percent of chlorine in the final product. Thus, Aroclor 1254 was produced by chlorinating a quantity of biphenyl until the resulting product was 54% chlorine by weight. Aroclor 1242 was 42% chlorine by weight.

12.9.4.2 PCBs were manufactured in batch processes, so there were slight variations between batches, but in general each Aroclor had a very reproducible pattern of chlorinated biphenyl isomers (congeners). With few exceptions, when we detect PCBs in the environment the initial contaminant was one of the Aroclors.

12.9.4.3 The one exception to the nomenclature of the Aroclors is Aroclor 1016. In the 1960's researchers started to find PCBs in fish tissue in the Great Lakes. The primary congeners appearing in the fish were pentachlorobiphenyls. The manufactures of PCBs devised a synthetic process that created an Aroclor with very similar properties to Aroclor 1242, but minimized the formation of pentachlorobiphenyl molecule. Aroclor 1016 41% chlorine by weight and as result it can be difficult to distinguish from 1242.

12.9.4.4 While the pattern of congeners was quite reproducible in the pure products once in the environment the pattern changes. The lesser chlorinated PCBs are more water soluble and are more volatile, while the more highly chlorinated PCBs bind to solids and sediments more strongly. As examples, landfill gas condensates tend to have a bias toward the lesser chlorinated congeners because they are more volatile. River sediments near source of PCBs tend to have a bias toward the more highly chlorinated congeners because the accompanying lesser chlorinated congeners were more water soluble. Downstream from the source of contamination, however, there will be a bias toward the less chlorinated congeners because the more heavily chlorinated congeners were trapped in the sediments near the

outfall. Anaerobic and aerobic microbial degradation reduce the concentrations of some congeners and an increase in concentrations of others. Although they are rarely the primary mechanisms, oxidative and photolytic processes are also selective, impacting some congeners more than others.

12.9.4.5 As a result, PCBs in the environment rarely have an exact match to the Aroclor standards that we use to calibrate our instruments. There is inevitably some level of judgment required to choose the Aroclor that has the best match to the sample in questions. Sometimes this is straightforward, but other times the judgment is difficult and can be controversial. In the worst cases, we can have situations where there are clearly PCB peaks throughout a chromatogram, but there is no good match with any of the Aroclors. It is recommended that at a minimum there must be some peak groupings present that are characteristic of an aroclor pattern in order to indicate a positive detection.

12.10 Calculations

12.10.1 Concentration of Analyte in Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.5 for details on establishing the calibration function):

12.10.1.1 Average Response Factor:

$$C_e = \frac{A_e}{RF}$$ Equation 12

12.10.1.2 Linear Regression:

$$C_e = \frac{[A_e - b]}{a}$$ Equation 13

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_e = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

12.10.2 Concentration of Analyte in Original Sample

The concentration of the analyte in the original sample is calculated as follows:

$$C_{sample} = \frac{C_e}{1000\ \frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF$$ Equation 14

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\ \frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).

DF = Dilution Factor (post extraction dilutions)

12.10.3 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\%$$ *Equation 15*

12.10.4 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD}\ \%\text{Recovery} = \left(\frac{SSR - SR}{SA}\right) \times 100\%$$ *Equation 16*

Where:

SSR = Measured concentration in spiked sample.

SR = Measured concentration in unspiked sample.

SA = Concentration of spike added to sample.

12.10.5 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\%$$ *Equation 17*

Where *R1* is the result for the MS and *R2* is the result for the MSD.

12.11 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DV-QA-0020.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy CA-Q-S-006. Each of the other aroclors have an MDLV performed annually to verify the MDL. For DoD, DOE and Texas TRRP projects, AR_1660 MDLVs and LOQVs are performed quarterly. MDLs and LOQs are stored in LIMS.

13.2 MDLV and LOQV

13.2.1 MDLVs and LOQVs must be performed on blank matrix as defined for the method blank (Section 9.3), spiked with the appropriate analytes at the programmatically required spike amounts.

13.2.2 MDLVs are analyzed for compounds on the DOD certificate and those required for Texas TRPP on each instrument each quarter. For DOD the spike requirement is 2-4X the MDL and for Texas it is 1-4X the MDL. There are no recovery requirements for MDLV. The MDLs for all other compounds are verified at least annually

13.2.3 LOQVs (aka LLOQV) are analyzed for compounds on the DOD certificate on each instrument each quarter. The spiking requirement is 1-2X the RL and must pass percent recovery limits. For all compounds analyzed by Method 8260, LOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test

need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (M-E-001 DV) for "Waste Management and Pollution Prevention.

14.2 Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Waste hexane solvent: Flammable Solvent – Waste Stream C

15.2.2 Vials containing extracts in hexane: Expired Extract Vials – Waste Stream A

15.2.3 Concentrated sulfuric acid and hexane from sample cleanup: Concentrated Acids with Organics - Waste Stream V

15.2.4 Expired reagents and standards – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.2.5 Samples containing polychlorinated biphenyls (PCB's) at concentrations ≥50 ppm are regulated under the Toxic Substance Control Act (TSCA) and must be segregated from all other waste streams. Analysts are responsible for contacting the Group Leader, Sample Control, and the Waste Coordinator immediately if a sample falls into the TSCA category.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.2 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.1.3 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.1.4 Method 3546, Microwave Extraction, Revision 0, February 2006.

16.1.5 Method 3580A, Waste Dilution, Revision 1, July 1992.

16.1.6 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.1.7 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.1.8 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, December, 1996.

16.1.9 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 1, February 2007

16.1.10 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.1.11 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.1.12 Method 8000D, Determinative Chromatographic Separations, Revision 4, July 2014.

16.1.13 Method 8000D, Determinative Chromatographic Separations, Revision 5, March 2018.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Final Version 5.0, July 2013.

16.4 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.1, July 2017

17.0 Method Modifications

Item	Method	Modification
1	8000B	Method 8000 allows for use of a second order or third order calibration curve. TestAmerica Denver does not allow for any curvilinear calibrations for the analysis of Aroclors.
2	8000C/D	Method 8000C and D require that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the samples the laboratory receives, and the number of times the GC column may require clipping, TestAmerica Denver does not perform an RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

18.0 Attachments

Table 1: Analyte List and Standard Reporting Limits
Table 2: Typical Instrument Conditions
Table 3: Calibration Levels (µg/mL)
Table 1-LVI: Analyte List and Standard Reporting Limits using Large Volume Injection
Table 2-LVI: Typical Instrument Conditions using Large Volume Injection
Table 3-LVI: Calibration Levels (µg/mL) using Large Volume Injection
Attachment 1: Aroclor Identification 101
Attachment 2: Example of Minimum Resolution Requirement for Aroclor 1260 Triplet

19.0 Revision History

Revision 15, January 4, 2019

- Added sections 13.1 and 3.8 in regards to LLOQ checks and standards.

Revision 14, October 31, 2018

- Added references to SW 846 Method 8000D
- Added explanations and calculations for % Error and Relative Standard Error
- Added method modification applicable to RT study requirements after column clipping
- Added references to South Carolina requirements

- Correct last sentence in the copyright information
- Updated the MDL SP number

Revision 13, July 31, 2018

- Annual Review

Revision 12, June 30, 2017

- Annual Review
- Updated references to DOD/DOE QSM 5.1 throughout in addition to existing QSM 5.0.
- Revised section 9.3 to reflect current requirements under DOD/DOE QSM that if no MS/MSD available, LCSD is performed in addition to LCS.
- Revised section 10.7.2 to reference both 1016 and 1260 rather than the combined nomenclature used internally by the laboratory.
- Updated Section 11.11.13 to reflect current practice for documenting review of manual integrations.
- Added references to section 16 for Method 8000D and DOD/DOE QSM 5.1.

Revision 11, June 3, 2016

- Annual Review
- Adjusted source references and concentrations in Section 7 as required to reflect current practice
- Updated Section 9.1 to reflect current practice
- Updated Section 9.6 to reflect current policy for over-range MS/MSD.
- Added “midpoint” to IS acceptance criteria in Section 9.8 to clarify standard used for evaluation of IS and updated acceptance window for IS recovery.
- Corrected resolution requirement criteria in Section 10.3.2.1
- Updated reference to corporate SOP on calibration curves in Section 10.3.1 and 12.1. Document was revised and number was changed.
- Added sentence to section 10.3.5 to reflect that all quantitations of identified aroclors are performed using a calibration curve (minimum five points).
- Added clarification to Section 10.3.8 for minimum height of peaks selected for quantitation relative to the largest peak in the aroclor (except 1268 at 10%)
- Added clarifications to paragraphs in Section 10.7.4.1
- Revised Section 10.8 and 10.9 to comply with Policy CA-T-P-005 for determining RT windows. This is intended to assist in minimizing false positive results.
- Revised language in Section 12.2 to explain when analyst interpretation of PCBs with a RT shift can be used
- Revised Section 13 to reflect current practice
- Replaced calibration factor with response factor throughout as this is now an internal standard method
- Editorial and formatting changes throughout

Revision 10, April 30, 2015

- Annual Review
- Added Internal Standard quantitation method throughout including
    - Section 7.3.1 – added description of internal standard stock
    - Section 7.11: IS solution,
    - Section 9.8: IS Acceptance criteria and corrective action

  - Section 10.3.2: Note regarding resolution check for Aroclor 1260
  - Section 10.4: Internal Standard Calibration
  - Incorporated equations for IS throughout Section 10.5
  - Added to section 10.7.2 that closing CCV may not be required with use of IS
  - Added requirement to run CCV for any identified aroclor if other than 1660
  - Section 10.8.7: Added use of RT marker for all representative aroclor peaks
- Corrected statement in Section 10.8.9 regarding shift noted by surrogate
- Added in Section 10.8.10 that RT windows can be set by RT marker
- Made additions to Section 11.7 for Analytical Sequence based on IS method
- Removed original Item 1 in Method Modifications table; renumbered.
- Added new Attachment 2

Revision 9, June 30, 2014

- Annual Review
- Updated the following sections to add include more detail and/or reflect correct practices. Sections: 1.2, 1.3, 2.1.2, 4.1, 6.1, 7.3.1, 7.4.1 (table), 7.4.2 (table), 7.5.1, 7.8, 9.1.2, 9.3, 9.5 (corrective action), 9.6 (corrective action), 10.1.2, 10.1.3, 10.3.6.4, 10.4, 10.5.4 (acceptance criteria), 10.6, 10.7.2, 10.7.4.1, 10.7.4.2, 11.5, 11.6, 11.9, 11.10.3, 12.2 and 12.3
- Corrected formatting and grammatical errors
- Added section 11.4 – Troubleshooting
- Additional references added to section 16
- Added total Aroclor calculation when summing individual Aroclors from dual column or different runs to section 12.5.3

Revision 8, October 30, 2013

- Updated sections 7.3,and 7.5 for the use of standard/standards
- Updated section 7.4.2 to correct table for CCV preparation
- Updated section 10.7 to add LVI CCV concentration
- Updated Table 2 and 2-LVI for injection volumes
- Added LVI tables to section 7.4

Revision 7, June 30, 2013

- Added section 2.1.2
- Added statement to Section 2.2 regarding use of 2nd column confirmation for DoD and method 80082A while presences of multiple peaks characteristic of an aroclor otherwise serve as confirmation when 2nd column is not required.
- Added sentence to Section 5.1.4 regarding procedures for handling radioactive materials
- Updated columns specified in Section 6.3
- Added injection port liners to section 6.4.2
- Added requirements for sample containers and sample volume for LVI to section 8.0
- Added clarifications to Corrective Action Section in Section 9.7 for evaluation and reporting of samples based on surrogate recoveries.
- Replaced references to Target software with Chrom software to reflect current practice
- Added dilution solvent (hexane) and extract final volumes for each matrix analyzed to section 10.3

- Removed note regarding restriction of use of Grand Mean as Method 8082 does not use the grand mean approach to CCV acceptance
- Added requirements for initial and final volume for LVI (Section 11.3.4)
- Added requirements for LVI injection volume (4 µL) to Section 11.5
- Added requirement for reporting lower result by Method 8000C (Section 12.5.2.1)
- Removed statement in section 12.9.2 duplicated in section 12.9.3.3
- Revised Table 2 to reflect current practice
- Revised Tables 1-LVI, 2-LVI, and 3-LVI to reflect current practice

Revision 6, June 15, 2012

- Added Tables 1-LVI, 2-LVI, and 3-LVI for large volume injection

Revision 5.1, January 16, 2012

- Changed extraction holding time for water and solid to 1 year with exclusion for California, Connecticut, Pennsylvania and South Carolina (Section 8).
- Reformatted paragraphs throughout

Revision 5, December 2011

- Combined SOP DV-GC-0021 and DV-GC-0030 Rev. 0.2. Upon implementation of this revision of SOP DV-GC-0021, SOP DV-GC-0030 will be deactivated.
- Added details for analysis of polychlorinated terphenyls by this procedure (sections 1, 3, 7 and Table 1).
- Updated Section 6 to include reference to master list of documents, software and hardware and volumetric flasks.
- Updated refrigerator temperature references from 4 $\pm$ 2°C to 0-6°C throughout.
- Updated vendors and catalog numbers for standards (Section 7)
- Updated Section 9 for consistency with SOP DV-QA-003P.
- Added calibration section to describe calibration models.
- Revised Procedure (new section 11) to be consistent with other SOPs revised in the last year.
- Added detail about review process (Section 11.8)
- Revised Calculations section (new section 12) to address dual column quantitation, sample dilution, and recovery calculations.
- Revised section numbers for previous sections 12-18.
- Updated Method Modifications section
- Revised Table 1 and Table 2

Earlier revision histories have been archived and are available upon request.

Table 1. Analyte List and Standard Reporting Limits

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aroclor 1016	1.0	33
Aroclor 1221	1.0	47
Aroclor 1232	1.0	33
Aroclor 1242	1.0	33
Aroclor 1248	1.0	33
Aroclor 1254	1.0	33
Aroclor 1260	1.0	33
Aroclor 1262	1.0	33
Aroclor 1268	1.0	33
PCT 5432	0.5	50
PCT 5442	0.5	75
PCT 5460	0.5	50

Table 2. Typical Instrument Conditions

Parameter	Recommended Conditions
Injection Port Temperature:	250 °C
Detector Temperature:	325 °C
Temperature Program:	Instrument W 125 °C for 1 minute 25 °C/min to 175 °C 10 °C/min to 275 °C 30 °C/min to 320 °C for 5 minutes Instrument P3 125 °C for 1.25 minutes 30 °C/min to 180 °C 12 °C/min to 280 °C 15 °C/min to 320 °C for 2.6 minutes
Column 1:	CLPI, 30 m x 0.32 mm id, 0.5 µm
Column 2:	CLPII, 30 m x 0.32 mm id, 0.25 µm
Injection:	2 µL 8082, 4 µL 8082 LVI
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek or J&W or Supelco glass tee, single gooseneck liner

Table 3. Calibration Levels (µg/mL)

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1221	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1232	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1242	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1248	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1254	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1260	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1262	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1268	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05
Decachlorobiphenyl	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05

Table 1-LVI. Analyte List and Standard Reporting Limits for Large Volume Injection

Compound	Water Reporting Limit (µg/L)
Aroclor 1016	1.0
Aroclor 1221	1.0
Aroclor 1232	1.0
Aroclor 1242	1.0
Aroclor 1248	1.0
Aroclor 1254	1.0
Aroclor 1260	1.0
Aroclor 1262	1.0
Aroclor 1268	1.0
PCT 5432	NA
PCT 5442	NA
PCT 5460	NA

Table 2-LVI. Typical Instrument Conditions for Large Volume Injection

Parameter	Recommended Conditions
Injection Port Temperature:	250 °C
Detector Temperature:	325 °C
Temperature Program:	Instrument P3 125 °C for 1.25 minutes 30 °C/min to 180 °C 12 °C/min to 280 °C 15 °C/min to 320 °C for 2.6 minutes
Column 1:	Restek Rtx-CLPesticides 30m X 0.32 mm id, 0.5 µm (Cat# 11139 or equivalent)
Column 2:	Restek Rtx-CLPesticides2 30m X 0.32 mm id, 0.5 µm(Cat# 11324 or equivalent)
Injection Volume:	1 µL 8082, 2 µL 8082 LVI
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek Universal Presstight Connector (Cat# 20400 or equivalent)
Injection Port Liner:	Agilent 5190-2293 90011 or equivalent

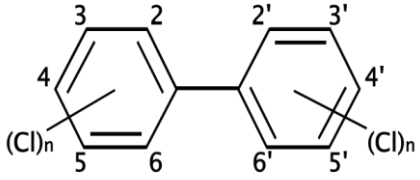
Table 3-LVI. Calibration Levels (µg/mL) for Large Volume Injection

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1221	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1232	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1242	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1248	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1254	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1260	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1262	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1268	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.00063	0.00125	0.0025	0.00625	0.0125	0.0188	0.025
Decachlorobiphenyl	0.00063	0.00125	0.0025	0.00625	0.0125	0.0188	0.025

Attachment 1.

Aroclor identification 101

It can be difficult to correctly identify which Aroclor is present in a sample. This document provides a few guidelines. We are calling this document Aroclor identification 101 not because it is simple, but because Aroclor identification 201 and 301 (mixed, weathered Aroclors) are much more difficult still (sort of like P-Chem!)



First, we should consider what Aroclors actually are: They are mixtures of polychlorinated biphenyls.

Each phenyl ring can accommodate between zero and 5 chlorines. There are 209 possible isomers with 1-10 chlorines (the surrogate decachlorobiphenyl is the fully chlorinated molecule). Of these, about 130 are present in various Aroclor mixes, accounting for the complexity of the chromatograms. The first two digits of the Aroclor number refers to the number of carbon atoms, the last two refer to the degree of chlorination. Thus Aroclor 1248 has 12 carbon atoms in each molecule, and 48% chlorine by mass. So, as the last two digits increase, the overall degree of chlorination increases, the volatility decreases, and the pattern of peaks moves later in the chromatogram. Arochlor 1248 consists of approximately 1% monochlorobiphenyl, 13% dichlorobiphenyl, 45% trichlorobiphenyl, 31% tetrachlorobiphenyl and 10% pentachlorobiphenyl.

It is estimated that 1.25 billion pounds of PCBs were produced until Monsanto ceased production in 1977. PCBs are very persistent, so much of this material is still present in the environment.

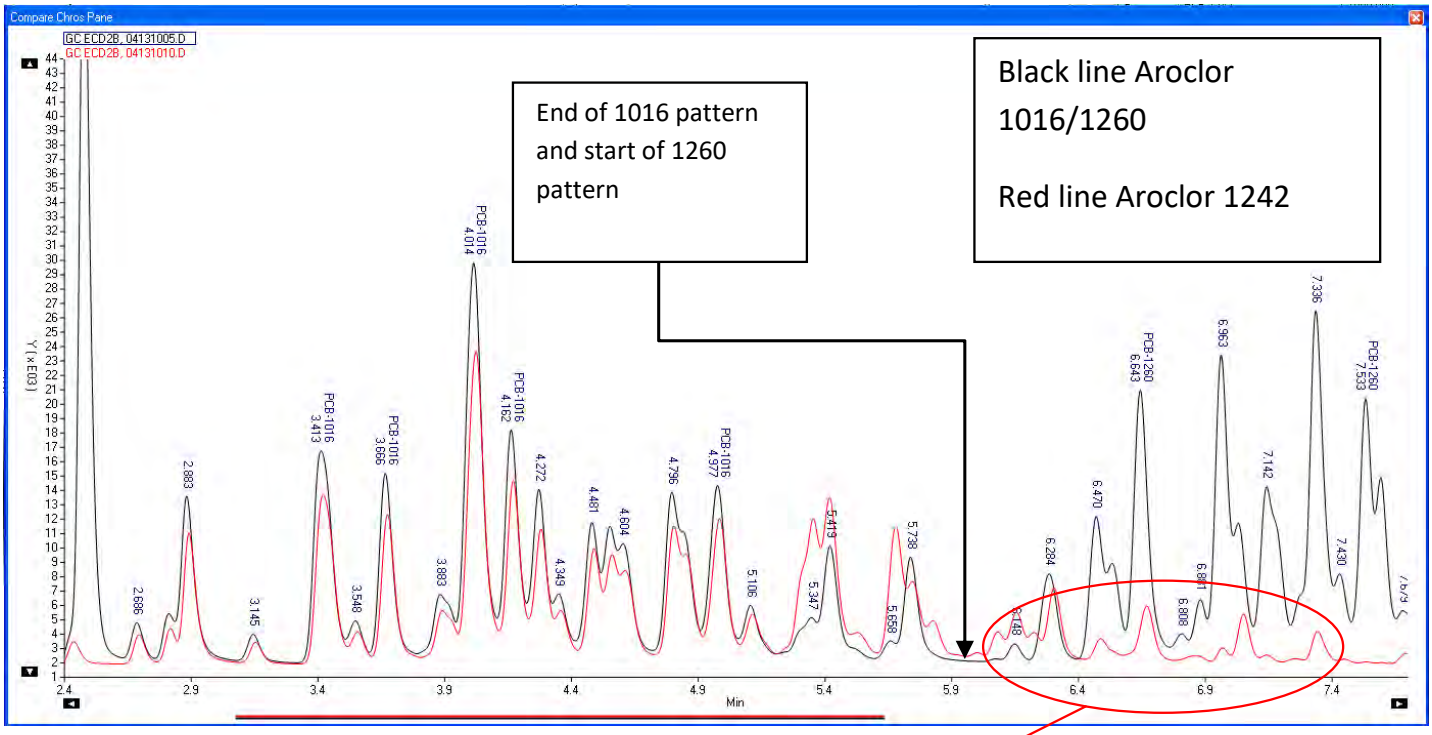
Example Chromatograms

In the following examples I'll refer to retention times frequently – your retention times will of course be different because of different chromatographic conditions but the same principles apply.

Attachment 1. Aroclor Identification 101 (Continued)

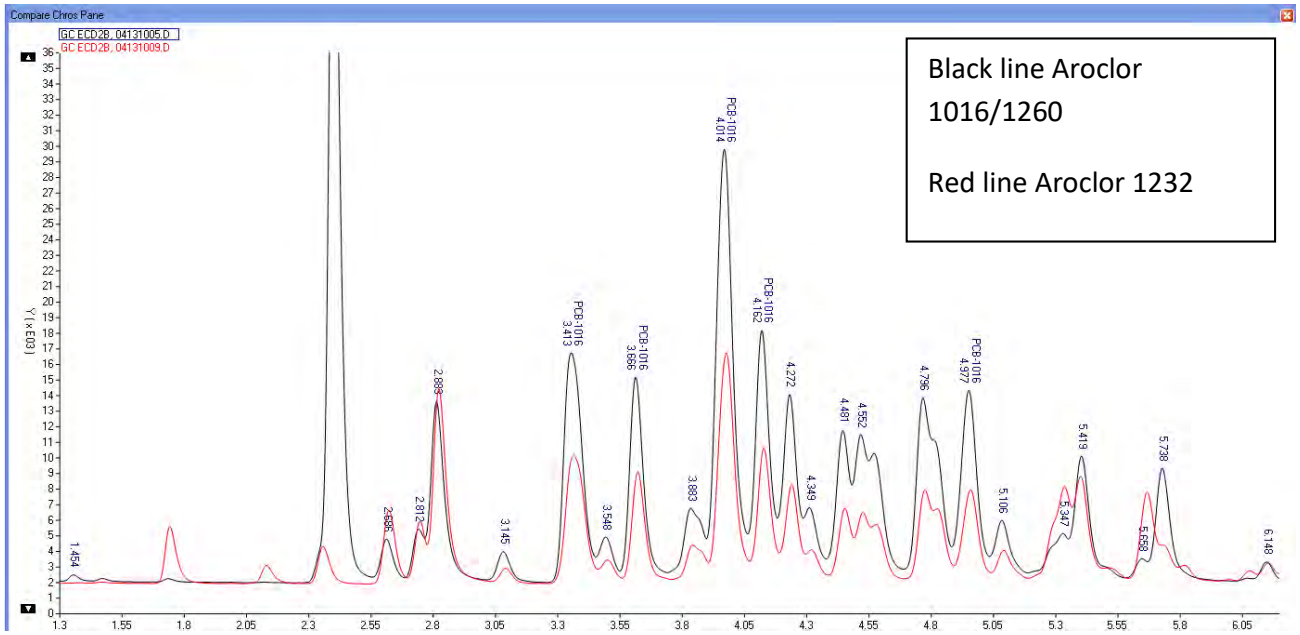
The first example considers Aroclor 1016 vs. 1242. Note that the early part of the chromatograms (2.5 – 5.3 minutes) are virtually identical. The key difference is the presence of some later eluting peaks (6.2-7.3 minutes in this chromatogram) in 1242 that are not present in 1016. This difference is masked by the fact that Aroclor 1260 is also present in this standard. Most labs analyze standards of 1016 and 1260 together – there is nothing wrong with this but it is a good idea to periodically (one run with each initial calibration?) analyze them separately so that you have a good idea of the two separate patterns.

The story of 1016 is interesting – in the early 1970's PCBs were starting to be found in fish in the Great Lakes. The more heavily chlorinated biphenyls were bioaccumulating more and were of greatest concern. So, Monsanto attempted to modify the manufacturing process to reduce the amount of pentachlorobiphenyls in Aroclor 1242, while still keeping the overall degree of chlorination similar. They were successful in this regard – Aroclor 1242 has about 10% pentachlorobiphenyls which show up between 6 and 7.4 minutes in the chromatogram below. Aroclor 1016 has 42% by weight chlorine (it does not follow the standard naming convention) but has no pentachlorobiphenyls.

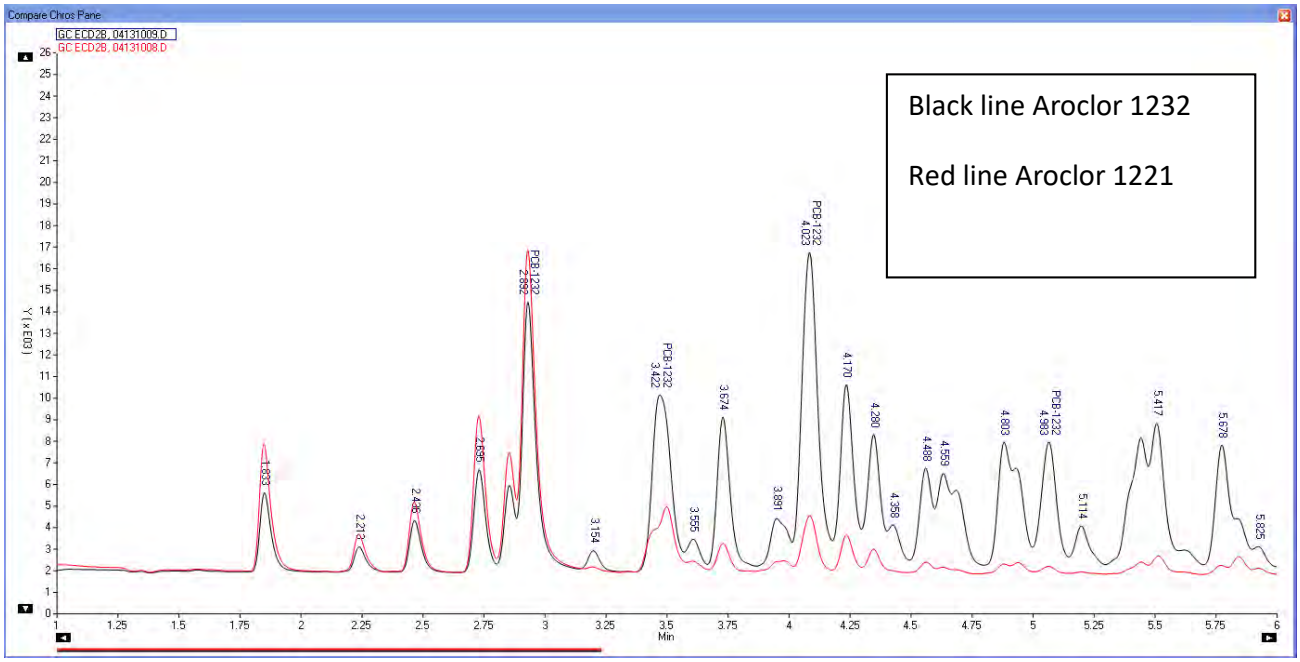


Attachment 1. Aroclor Identification 101 (Continued)

Here is 1016 vs. 1232. These are even more similar (the large peak at around 2.35 min is TCMX) but note the very early peaks present in 1232 and not in 1016, and also note that the front end is stronger in 1232 for example in 1232 the peak at 2.88 min is about the same size as those at 4.79 and 4.97, whereas in 1016 the later peaks are twice as large.

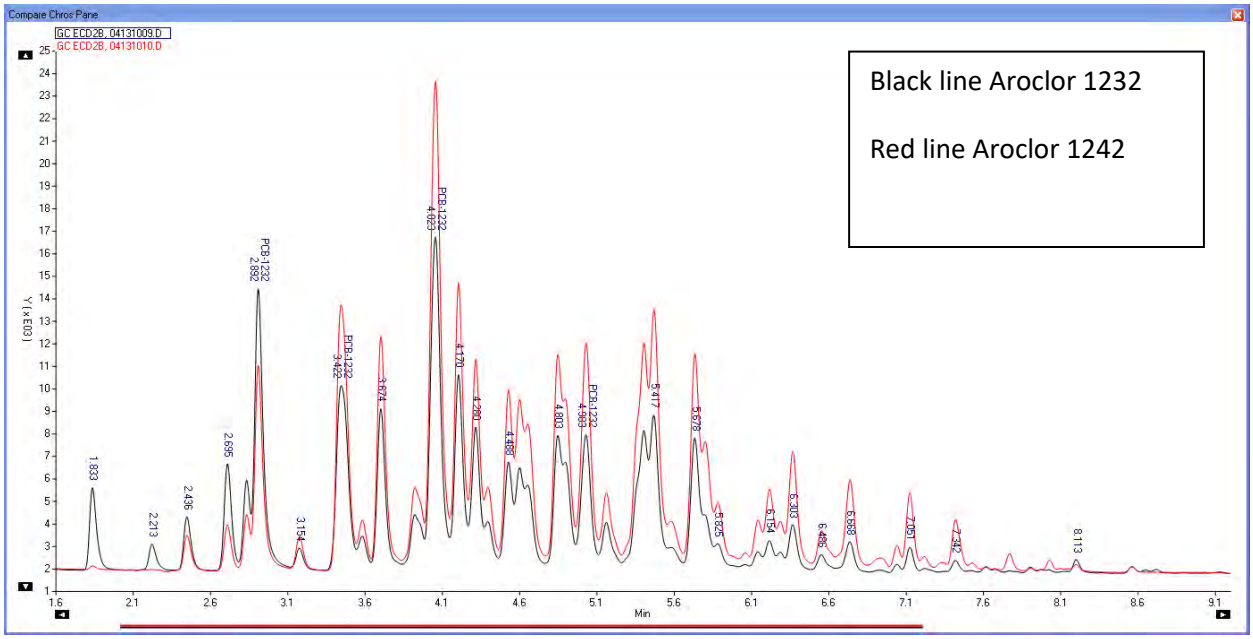


For 1221 vs. 1232, the front end of the chromatogram is identical, but 1232 has later peaks that are not present in 1221.

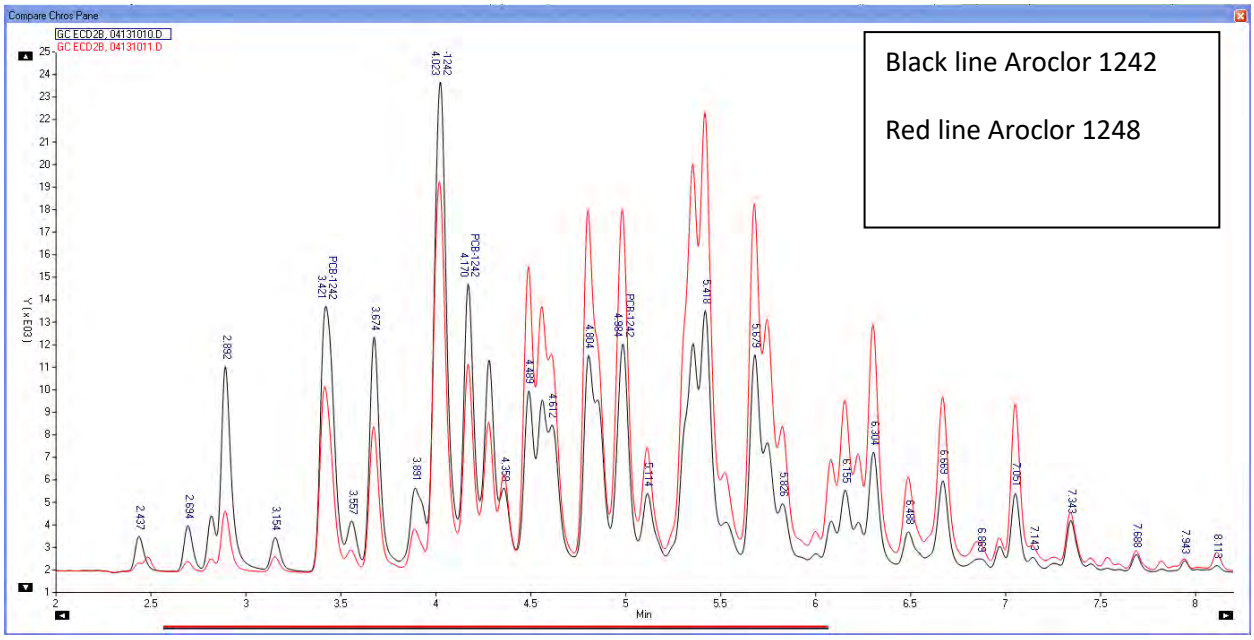


Attachment 1. Aroclor Identification 101 (Continued)

1232 and 1242 are best distinguished by the early peaks in 1232 (1.83, 2.13) that are not present in 1242. Also note that the peak at 2.89 is twice the height of that at 5.41 in 1232, whereas the 5.41 peak is slightly higher in 1242. This relative size of the front and back end of the envelope is a key tool for distinguishing Aroclors.

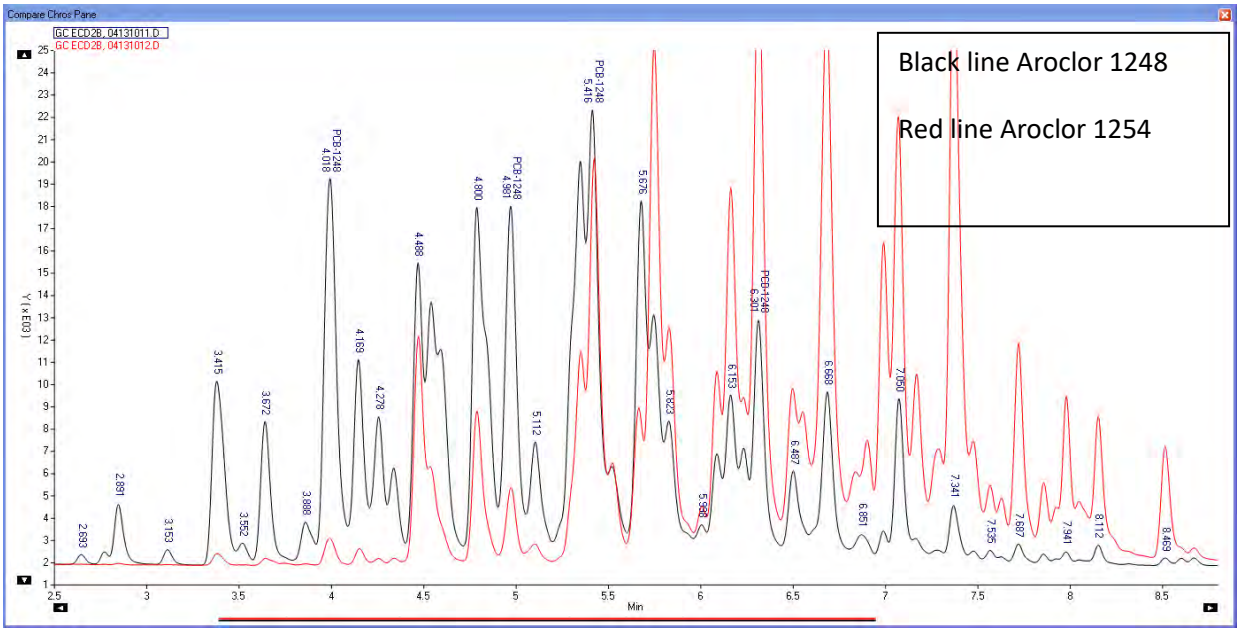


Aroclors 1242 and 1248 both have all of the same peaks, so the relative strength of the front and back of the envelope is the only way to distinguish. For example, in 1242, the peak at 3.42 is larger than that at 5.67, whereas for 1248, the 5.67 peak is considerably larger than the 3.42 peak.

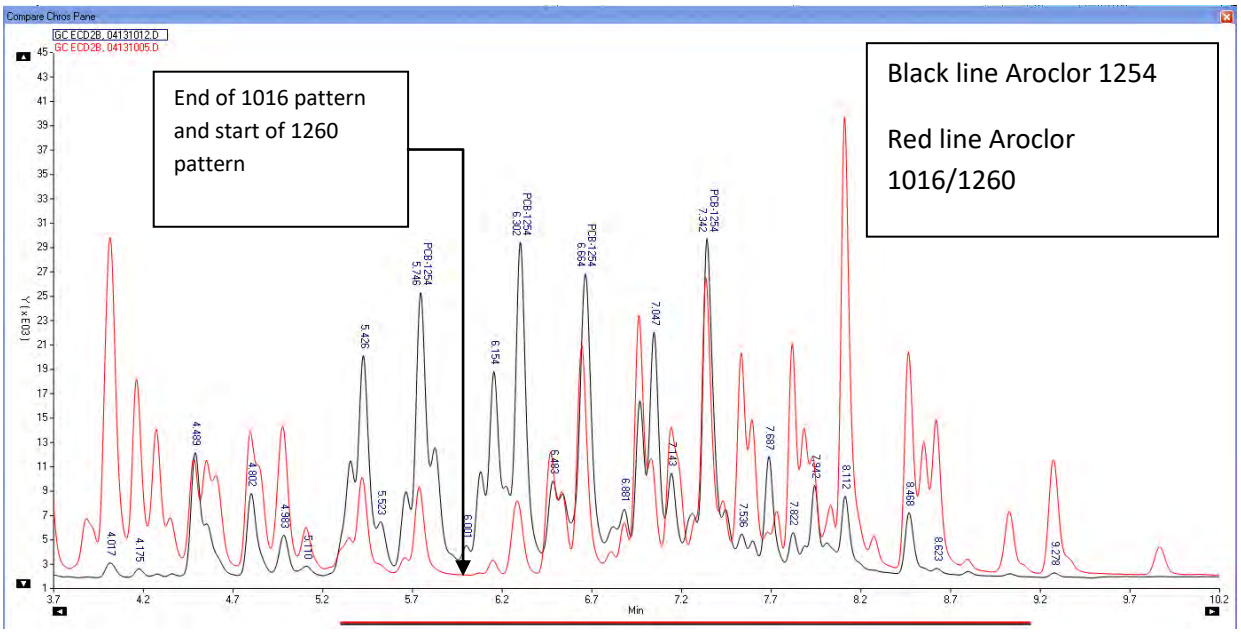


Attachment 1. Aroclor Identification 101 (Continued)

1248 vs 1254 is a relatively easy case, the front end of the envelope is much stronger in 1248.

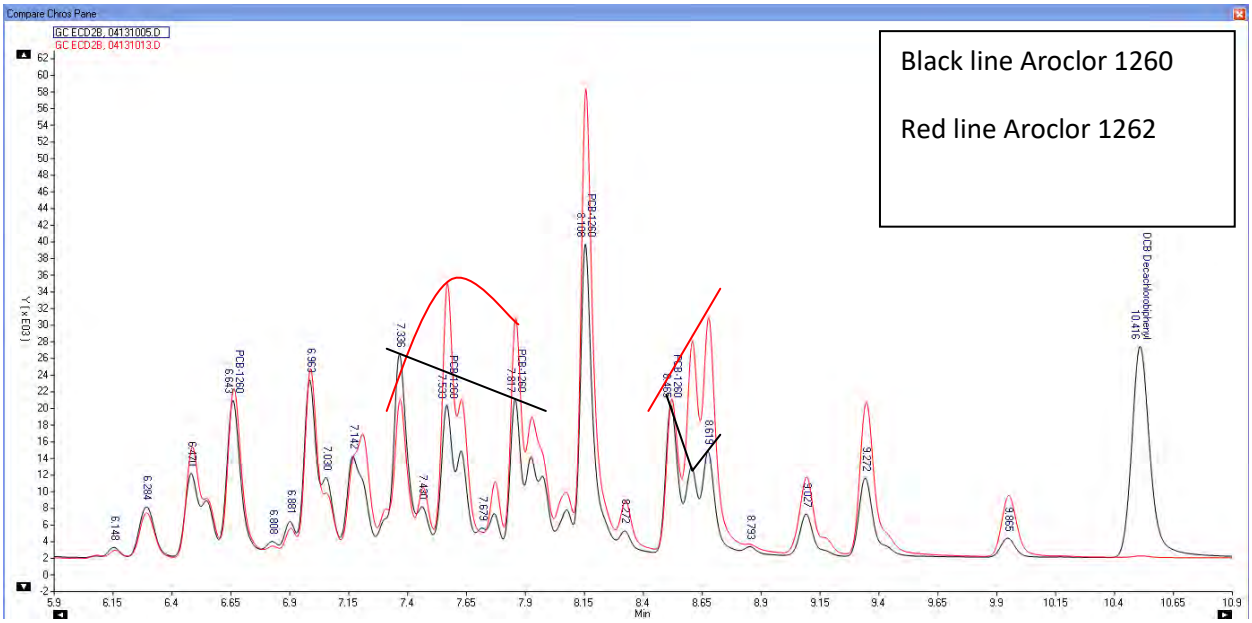


The 1254 vs. 1260 chromatograms are again a little masked by the inclusion of 1016 in the 1260 standard (peaks up to 5.9 min in the 1260 chromatogram actually belong to 1016). Keeping this in mind, the presence of peaks at 5.42 and 5.74 indicates 1254. The relative strength of peaks in the 7.5-9.3 range indicates 1260.

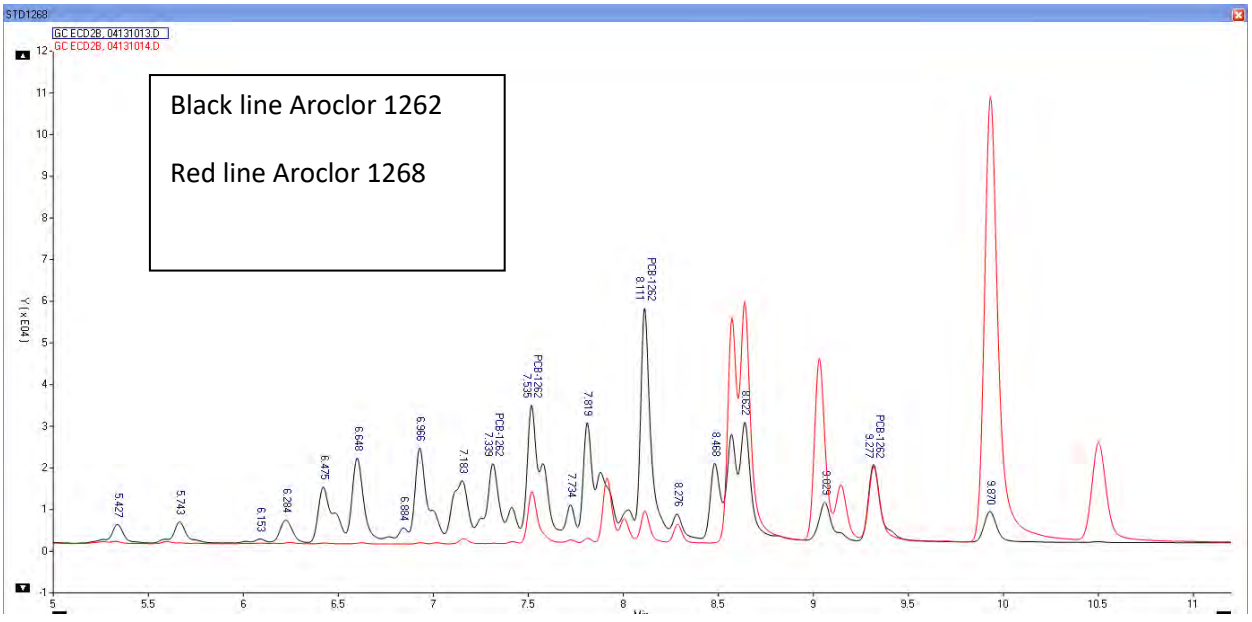


Attachment 1. Aroclor Identification 101 (Continued)

Many labs do not analyze for 1262, perhaps just as well since it is certainly challenging to distinguish from 1260. However, the shape of the envelope is again the key. Note that in 1262 the peaks around 8.6 minutes are as large as that at 6.96, whereas they are only half the size in 1260. Also note the shape of the envelope for the peaks in the 7-8 minute range – bow shaped for 1262 and a straight declining line for 1260. The envelope shape is also quite different in the 8.4-8.7minute range.

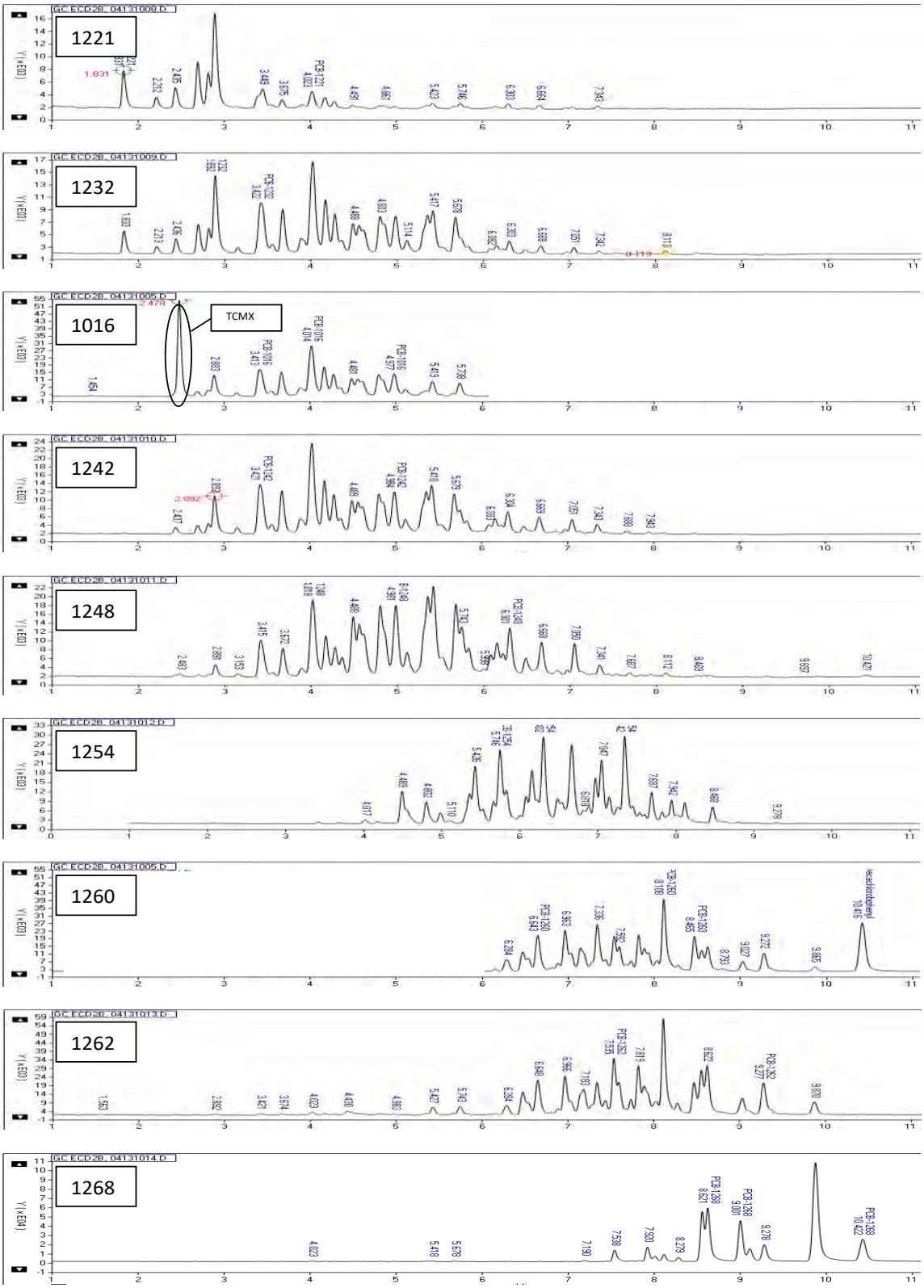


The really strong peak at 9.87 and the lack of much of a pattern between 6.0 and 7.5 minutes are good indicators of 1268.



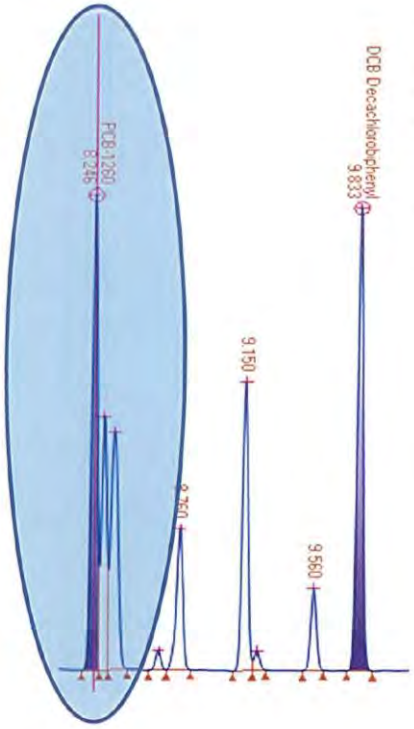
Attachment 1. Aroclor Identification 101 (Continued)

AROCLOR PATTERNS



Attachment 2.
Example of Minimum Resolution Requirement for Aroclor 1260 Triplet

(See Work Instruction CA-T-WI-003 for more information)



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

Work Instruction No. CA-T-WI-003, dated 31 Mar 2015

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Characterization of RCRA Waste

Approvals (Signature/Date):

Doug Gomer 6/27/19
Doug Gomer Date
Environmental Health and Safety Coordinator

Roxanne Sullivan 6/27/19
Roxanne Sullivan Date
Quality Assurance Manager

Charles Newton 6/27/19
Charles Newton Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 PURPOSE

1.1 This SOP is a statement of policy that stipulates the process used by TestAmerica Denver to properly characterize a waste to determine the correct disposal method. This is one of the key operational procedures required under TestAmerica Denver's Waste Management Plan (TestAmerica Denver Policy DV-HS-001P).

1.2 The ultimate objective is that TestAmerica Denver's waste disposal practices are compliant with local, state, and federal requirements.

1.3 This SOP does not describe the additional procedures required for the characterization and management of radioactive wastes. See SOP DV-RS-0017, *Radioactive Waste Disposal*, for those details.

1.4 Management of spills or accidents involving waste is described in the TestAmerica Denver Contingency Plan which is incorporated into the TestAmerica Denver Facility Addendum.

2.0 RESPONSIBILITIES

2.1 Waste Disposal Technician or Specialist (or designee) has the following responsibilities:

2.1.1 Sampling waste containers in accordance with 6 CCR 1007-3 Part 265.13.

2.1.2 Labeling waste containers properly to reflect the results of characterization.

2.2 Waste Coordinator (or designee) has the following responsibilities:

2.2.1 Disposing of waste properly, in accordance with applicable requirements.

2.2.2 Providing training to employees as to the extent of their job function.

2.2.3 Preparing summary reports for analytical characterizations and signing off on documentation.

2.2.4 Maintaining all documents associated with this procedure.

3.0 SAFETY

3.1 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

3.2 This SOP is an administrative statement of policy. Actual tests performed to characterize waste are described in technical SOPs.

3.3 Eye protection that satisfies ANSI Z87.1 (per TestAmerica Denver Environmental Health and Safety Manual), laboratory coat, and appropriate gloves must be worn while working in laboratory areas and handling wastes.

4.0 DEFINITIONS AND ACRONYMS

4.1 Radioactive Waste: Waste with known or suspected radioactive material.

4.2 Hazardous Waste: Solid wastes designated by 40 CFR Part 261 and regulated as hazardous waste by the United States Environmental Protection Agency.

4.3 Mixed Waste: A hazardous waste that also exhibits radioactivity.

4.4 Non-radioactive Waste: Waste with no known or suspected radioactive contamination.

4.5 POTW: Publicly Owned Treatment Works, i.e., sewer.

4.6 TSDF: Treatment Storage and Disposal Facility.

5.0 POLICY

5.1 Any deviation from this procedure must be documented as a nonconformance, with a cause and corrective action described.

5.2 Summary

Generators are required to accurately characterize their waste. A hazardous waste determination may be obtained by applying knowledge of the waste's chemical or physical properties or by analytical testing.

5.3 Characterization of Waste Streams

5.3.1 TestAmerica Denver Waste Streams are listed in Attachment 1 of SOP DV-HS-0002, *Collection and Accumulation of Hazardous Waste*.

5.3.2 All wastes generated are classified as hazardous or non-hazardous as defined in 6 CCR 1007-3 Part 261. Regulations identify three separate categories of hazardous waste, as follows:

- Characteristic wastes: exhibiting the characteristics of ignition, corrosiveness, reactivity, and/or toxicity.
- Listed wastes: F, K, P, or U listed in 6 CCR 1007-3 Parts 261.30 through 261.33.
- Mixture of solid waste and listed or characteristic hazardous waste.

If a material fits one of these categories, it is regulated under RCRA and considered hazardous.

5.3.3 Characterization of TestAmerica Denver waste streams are determined either by testing or by documented process knowledge as described in 6 CCR 1007-3 Part 262.11. Once established, the profile may be generated by the vendor electronically. At a minimum, characterization should be performed in the following situations:

5.3.3.1 To determine if the waste is restricted from land disposal.

5.3.3.2 When the process that generates the waste has changed.

5.3.3.3 When TestAmerica Denver is notified by the Treatment Storage and Disposal Facility (TSDF) that the characterization of the waste does not match the pre-approved specifications profile.

5.3.3.4 When a new analytical process is introduced into the laboratory.

5.3.4 In addition to the above, waste stream profiles are reviewed annually.

5.3.5 When the use of process knowledge is not practical or is inconclusive, the waste is tested for toxicity characteristics. Complete toxicity characterization consists of the tests/analyses listed below. 6 CCR 1007-3 Part 261.24 Table 1 provides a complete list of the individual regulated chemicals and the maximum concentration allowable for the toxicity characteristic.

- pH
- Ignitability (Solid)/ Flashpoint (Liquid)
- Phenoxyacid Herbicides (RCRA List)
- Chlorinated Pesticides (RCRA List)
- Volatile Organics (RCRA List)
- Metals (RCRA List)
- Cyanide
- Sulfide
- Radioactivity (Gross Alpha and Beta/Gamma)

NOTE: The determination of radioactivity is not required for RCRA characterization, but is done to ensure that the laboratory is in compliance with its radioactive materials handling license.

5.3.6 Analytical characterization may be reduced to periodic monitoring if repeated measurements show that a toxic contaminant is not present or indicative of a particular waste stream. Documentation to this effect shall be maintained by the Waste Coordinator or designee.

5.4 Disposal Methods

5.4.1 Waste is disposed of by one of the following four options:

5.4.1.1 Characterized as non-hazardous and released into the POTW (Publicly Owned Treatment Works, i.e., sewer).

5.4.1.2 Characterized as non-hazardous and shipped off site to an EPA-approved TSDF.

5.4.1.3 Characterized as hazardous and shipped off site to an EPA-approved TSDF.

5.4.1.4 Characterized as non-hazardous/radioactive or mixed waste and shipped off site to a TSDF that is approved to accept and dispose of radioactive waste.

5.4.2 Waste Release into the POTW

5.4.2.1 Release of waste into the POTW is acceptable provided that the waste is characterized as non-hazardous at the discharge point, and meets the "Metro District Limitations", Section 6.18.1, provided by Metro District's Rules and Regulations, 5/30/2003 as listed in Attachment 1.

5.4.2.2 If a waste is characterized as non-hazardous but fails to meet the limitations listed in Attachment 1, a calculation may be performed that takes into account the average daily effluent discharge rate from the facility. If the calculation proves that the effective discharge concentration for the component is below levels of concern at the discharge point, the waste may be released into the POTW (See DV-HS-0005 Section 4.3.4.4 for details).

5.4.2.3 TestAmerica Denver does not knowingly release radioactive material into the POTW in accordance with the Colorado Rules and Regulations Part 4 for Standards for Protection against Radiation.

5.5 Documentation

5.5.1 Analytical results and supporting quality control data for waste characterization shall be maintained by the Waste Coordinator or designee and kept indefinitely in accordance with SOP DEN-QA-0005, *Document Archiving Procedure*.

5.5.2 A summary report shall be prepared by the Waste Coordinator or designee that describes the type of analyses performed, a discussion of the results, flow rate calculations (if applicable), and disposal method. The summary report shall also include the date the waste was disposed of by the Waste Coordinator or designee.

6.0 REFERENCES / CROSS-REFERENCES

6.1 Environmental Health and Safety Manual, TestAmerica Denver, current revision.

6.2 Quality Assurance Manual (QAM), TestAmerica Denver, current revision.

6.3 Radiation Safety Manual (RSM), TestAmerica Denver, current revision.

6.4 Colorado Hazardous Waste Regulations (6 CCR 1007-3).

6.5 Clear Creek Valley Water and Sanitation District Industrial Pretreatment Resolution, current revision.

6.6 Metro District's Rules and Regulations, 11/3/2013.

7.0 ATTACHMENTS

Attachment 1: Metro District Limitations for Discharge

8.0 REVISION HISTORY

Revision 11, dated 30 June 2019
- Basic Annual Review
- Updated copyright information

Revision 10, dated 30 June 2018
- Basic Annual Review

Revision 9, dated 30 June 2017
- Basic Annual Review

Revision 8, dated 30 June 2016
- Basic Annual Review
- Removed revision histories 2011 and earlier

Revision 7, dated June 30, 2015
- Basic Annual Review
- Minor reformatting throughout
- Corrected reference in Section 5.3.1 to Attachment 1
- Corrected Section 5.3.5 to link ignitability with solid samples and flashpoint with liquid samples
- Added reference in Section 5.4.2.2 regarding waste disposal to the sewer

Revision 6, dated June 30, 2014
- Basic Annual Review
- Re-formatted SOP

Revision 5, dated June 30, 2013
- Basic Annual Review
- Updated Attachment 1 to reflect Metro District's Rules and Regulations
- Formatting and grammatical changes throughout

Revision 4.4, dated June 22, 2012
- Basic Annual Review

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Metro District Limitations for Discharge

Component	Limit (mg/L)
Arsenic	**0.33**
Cadmium	**3.4**
Chromium	**3.6**
Copper	**6.1**
Lead	**2.2**
Mercury	**0.13**
Molybdenum	**0.43**
Nickel	**5.6**
Selenium	**0.66**
Silver	**2.9**
Tetrachloroethene	**1.5**
Zinc	**15.6**

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Excess Sample Material Management

Approvals (Signature/Date):

Doug Gomer 6/27/19
Doug Gomer Date
Environmental Health and Safety Coordinator

Roxanne Sullivan 6/27/19
Roxanne Sullivan Date
Quality Assurance Manager

Charles Newton 6/27/19
Charles Newton Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 PURPOSE

1.1 This procedure describes TestAmerica Denver's handling of sample material remaining after all chemical testing ordered by our clients is completed. This includes tracking individual samples from the time they are removed from the storage areas for active samples, through archival storage, and ending with final disposition of the excess sample materials. This is one of the key operational procedures required under TestAmerica Denver's general *Waste Management Plan* (Policy DV-HS-001P).

1.2 The disposal of all wastes shall comply with all federal, state, and local regulations concerning hazardous waste disposal.

2.0 RESPONSIBILITIES

2.1 Any employee who receives, prepares, or offers hazardous materials for transportation must receive DOT training to the degree of his/her job responsibility as described in SOP DV-QA-0024 and 49 CFR Section 171.2(a). All waste personnel who dispose of waste shall also have knowledge of Clear Creek Valley Water District (CCVWD) discharge regulations and RCRA regulations.

2.2 Waste Coordinator (or designee) responsibilities include:

2.2.1 Overseeing personnel involved in handling samples after they have been relinquished from the active sample storage areas.

2.2.2 Determining the analytical testing that is needed to properly characterize bulked samples.

2.2.3 Identifying the correct method of disposal for each container of excess sample material.

2.3 Waste Technician/Specialist (or designee) is responsible for:

2.3.1 Documenting that samples have been archived by scanning the bar codes into the TestAmerica LIMS (TALS).

2.3.2 Tracking individual samples in the Sample Archive area.

2.3.3 Bulking samples in drums at the end of the required archive time period.

2.3.4 Collecting representative subsamples from the drums of bulked samples, and submitting those samples to the laboratory for testing as directed by the Waste Coordinator.

2.3.5 Disposing of samples as directed by the Waste Coordinator.

2.3.6 Maintaining all records related to bulk sample disposal.

2.3.7 Generating a list of samples that will require approval before disposal.

2.3.8 If waste personnel transfer the sample to another box or container, they shall mark the box with all the labels and markings on the original box.

2.4 Radiation Safety Officer (RSO) (or designee):

The RSO will direct the activities described in Section 2.3 as they pertain to radioactive samples.

2.5 Project Manager/Project Assistant is responsible for:

2.5.1 Identifying to the Waste Coordinator any known hazards in a sample.

2.5.2 Notifying the Waste Coordinator in writing of samples that have been analyzed and are to be returned to the client, rather than accumulated and disposed of by the laboratory.

2.5.3 Communicating in writing to the Waste Coordinator special client or project requirements for archiving samples, e.g., refrigerated or > 30 days.

2.5.4 Notifying Waste Management personnel in writing if the analyses for a sample lot have been canceled.

2.5.5 Contacting clients for disposal approval for samples requiring client approval before disposing of the samples.

2.5.6 Ensuring project information in TALS is correct, including but not limited to, the correct disposal code and the correct number of days sample is to be held before disposal if the sample requires a longer retention time than 30 days.

2.6 Sample Control Staff

2.6.1 Sample Control personnel track the locations of samples in the main and satellite sample storage areas using TALS.

2.6.2 Sample Control personnel will label sample containers and outer containers with descriptions of known hazards, i.e., Rad Cat levels, PCB contents, Explosive Hazards, etc. Sample Control personnel will label boxes (or other organizing container) containing sample bottles with extended holding time and special disposal requirements.

2.6.3 Sample Control personnel will remove all unbroken samples from coolers containing broken/damaged hazardous samples before turning the cooler over to Waste Management. If Sample Control needs guidance on cleaning intact sample bottle from a cooler of hazardous samples, the Waste Coordinator (or designee) shall be contacted. Sample Control personnel shall clean broken/damaged samples and coolers that are not hazardous. The DOT shipping papers shall be used to determine if a sample is hazardous or non-hazardous. If a cooler containing broken samples is transferred to Waste Management, it should be accompanied

by a written description that includes the number and type of broken containers.

2.6.4 Sample Control personnel will remove samples from the walk-in coolers after the sample jobs have been invoiced and the appropriate storage time has passed. The samples will be placed on carts and delivered to the Waste Control group for archiving.

3.0 SAFETY

3.1 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

3.2 Procedures shall be carried out in a manner that protects the health and safety of all TestAmerica associates. This procedure must be performed in accordance with the TestAmerica Environmental Health and Safety Manual (EHSM).

3.3 Eye protection that satisfies ANSI Z87.1 (as per the Environmental Health and Safety Manual), laboratory coat, and appropriate gloves must be worn while samples are being handled. Normally, latex or nitrile gloves are worn when handling excess sample waste. If handling broken glassware, cut resistant gloves are required. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.4 Avoiding Spills and Spill Containment

3.4.1 Be aware of the location of spill kits in the area. Do not proceed until instructed on the proper use of spill kits and the principles of spill containment.

3.4.2 To avoid spills, proceed slowly when transferring the contents from one container to another.

3.4.3 Immediately notify EH&S personnel if a large spill occur.

3.4.4 Dispose of the materials used to neutralize or contain the spilled material in accordance with SOP DV-HS-0003, *Characterization of RCRA Waste*.

3.4.5 Refer to the EHSM for additional information concerning spill control.

3.5 The lids (or bungholes) must be kept closed on waste containers when not being filled.

3.6 Hearing protection is required in the Sample Archiving Room when the glass crusher and/or the plastic shredder are in operation.

3.7 Hearing protection is required in the explosives grinding room when either grinding apparatus is in operation.

3.8 When pouring sample waste into drums or crushing or shredding glass and plastic, a face shield with safety glasses or splash-resistant goggles shall be worn.

4.0 PROCEDURE

4.1 Archiving samples

4.1.1 When empty shelves in the walk-in coolers are filled with new sample receipts by Sample Control personnel, the sample jobs on the next shelf (numerically) will be reviewed in TALS to determine if they have been invoiced and are not on hold. The current job status may be viewed in the internal chain of custody (ICOC) module in TALS. For each job the ICOC must show dates in both the "Disposal After Date" and "Last Invoice Date" columns and must be checked in TALS to ensure it is not on hold before the samples may be removed from the shelf. If the jobs have been invoiced and are not on hold the samples may be removed from the shelf, placed on a cart, and moved to the Sample Archive area. If the samples have not yet been invoiced or have an "on hold" status in TALS, they shall be returned to the shelf.

4.1.2 Alternatively, Sample Control personnel may run a query in TALS using a module called Disposal Report under the Sample Management menu. After selecting the Disposal Report module click the button marked "OK" and then select the report titled "Disposal Report Selected by Disposal Date". This query is a report developed in TALS and pulls the sample job numbers by invoice date for a specified date range. This report also contains all storage locations for the samples. This query will not pull in samples which are on hold. Any job that appears on this report may be removed from the walk-in cooler shelves, placed on a cart, and delivered to the Sample Archive area.

4.1.3 It is critical that all digits of each job number on the report are verified against sample bottles in the shelf boxes. All samples from the sample receiving coolers and satellite coolers shall be placed on rolling carts. Excess material in original sample containers shall be consolidated into as few storage boxes as possible to minimize storage space in Sample Archive.

4.1.4 The excess sample material is moved from the cart onto a shelf in the Sample Archive area.

4.1.4.1 The excess sample material that is Cat 2 or higher is moved from the cart onto a shelf in the Radiological Sample Archive area.

4.1.4.2 Quarantined soil samples shall be segregated from other soil samples while in archive storage.

4.1.4.3 All samples shall be stored in an organized manner to allow easy retrieval of sample material.

4.1.4.4 The excess sample archive storage location is logged in TALS.

The projected sample disposal date is recorded on the sample storage box in red.

4.1.4.4.1 Excess sample archive disposal dates are determined by agreement between the client and the Laboratory by contract or good faith agreement. The regular archive holding time is 30 days past invoice date at room temperature storage. Any changes from the regular storage practices will be conveyed to the Sample Login Staff by written comment in the project in TALS. These special requirements are written and stickers attached to the box used for storage. These stickers alert the waste disposal staff to special requirements.

4.1.4.4.2 Project Managers are responsible for obtaining clients permission to dispose of samples that require client permission.

4.1.5 Periodic sweeps are made of all sample storage areas to ensure that all invoiced samples that are not on hold have been archived. The Sample Control supervisor determines samples to be considered for disposal by older job numbers. When an older sample is identified, it must be verified as needing to be moved prior to relocation to Sample Archive.

4.2 Storage

4.2.1 Ensure that samples are standing upright and are not leaking.

4.2.2 When samples are retrieved from archive storage for reanalysis, the person requesting the samples shall record the transfer in TALS. If samples are not relogged into TALS with a new job number, they should be checked back into TALS and returned to the storage location where they were found (if possible). If a new location is required the storage location must be updated in TALS.

4.2.3 Keep the sample storage area cleaned and swept.

4.2.4 Grease the plastic shredder and glass crusher on at least a monthly basis, but do not over-lubricate. Refer to the owner's manual for maintenance instructions on how to care for the machine. The machine should be cleaned inside and out to prevent fires from accumulated plastic.

4.2.5 The Sample Archive and Radiological Sample Archive areas are inspected weekly by the Waste Coordinator per RCRA.

4.3 Disposal

4.3.1 Archived samples shall be disposed into the appropriate waste container on or after the disposal date written on each sample lot box.

4.3.1.1 A neutralizing agent may be added to the barrel prior to dumping for liquids prior to disposal.

4.3.1.2 Ensure the drum is compatible with the material being disposed and that the drum meets DOT specifications for shipping the materials if the drum contents are determined to be hazardous.

4.3.2 Prior to disposing of any sample the Waste Technician/Specialist (or designee) will confirm the lot number on the sample matches the lot number on the box.

4.3.3 Samples are scanned into TALS for tracking disposal. When samples are scanned into TALS for disposal prior to invoicing a pop-up will be generated requesting confirmation of the disposal of the sample. If the pop-up occurs and the sample bottle is **not** empty; Log-in **must** be notified prior to proceeding forward.

4.3.4 Dump the liquid samples into a numbered drum for liquids and record this information in TALS.

4.3.4.1 Radioactive samples shall be disposed into separate waste drums and checked for activity. The monthly effluent discharge activity shall not exceed the limits of the Colorado Department of Public Health and Environment, Rules and Regulations for Radiation Control, Parts 1-20.

4.3.4.2 When liquid drums are full, remove the drum from service and cap the drum. All drums shall be sampled and the results received prior to discharging any sample. To sample a drum, fill the sample bottles according to the sampling COC for quote 28007252 and deliver to Sample Receiving for login.

4.3.4.3 Move the filled drums if needed to an appropriate holding area.

4.3.4.4 Upon receipt of the analytical results from the sampled drum, the drum is evaluated for disposal to the sewer system using Attachment 1 and, if necessary, Attachment 2. Attachment 1 is completed by entering the results from TALS into the spreadsheet which calculates the concentration at the facility release point to the sewer.

4.3.4.4.1 If the discharge concentration is above CCVWD POTW restrictions and the drum cannot be discharged in smaller aliquots, it will be profiled for shipment as hazardous waste.

4.3.4.4.2 If the drum is below the CCVWD restrictions, the pH of the drum is checked, adjusted if necessary, and discharged to the drain. The pH shall be between 5 and 9. It requires the signatures of two Waste Management personnel to discharge the drum to the

drain.

4.3.4.5 The drum number and the date it was disposed to the POTW or shipped offsite (as well as the TSDF site if shipped) are recorded in the TALS database.

4.3.5 Dispose of the entire soil sample with container into a numbered drum for soils and record this information in TALS.

4.3.5.1 Quarantined soil samples shall be segregated from other soil samples while in archive storage.

4.3.5.2 Move the filled drums if needed to an appropriate holding area.

4.3.5.3 Soil samples are shipped to an authorized TSDF site for incineration and disposal.

4.3.5.4 Cat 1 samples shall be disposed as non-radiological samples after being surveyed.

4.3.6 Crush/shred all empty bottles to destroy any and all labeling. The crushed glass and shredded plastic should be recycled if possible.

4.3.7 Excess sample material to be returned to the client (as determined by contract or request) will be shipped according to DOT regulations. Copies of the shipping documents will be kept in the Waste Coordinator's office for at least 3 years before being archived off site.

4.3.8 Drum tracking is documented in the Waste Storage and Container Inspection logbook, as well as in TALS and RADACC.

5.0 DEFINITIONS

5.1 Quality Assurance Summary (QAS) - A document that describes all conformance requirements regarding sample analysis, reporting, safety and waste disposal issues.

5.2 POTW – Public Owned Treatment Waterworks.

5.3 TSDF – Transfer Storage Disposal Facility

5.4 DOT – Department of Transportation

5.5 RADACC – The database used to track aspects of radiation safety, including radioactive sample screens and radioactive material inventory control.

6.0 ATTACHMENTS

Attachment 1: Discharge to CCVWD Evaluation Sheet (Example)
Attachment 2: Radiological Discharge to CCVWD Evaluation Sheet (Example)

7.0 REVISION HISTORY

Revision 14, dated 30 June 2019

- Annual review
- Removed the reference in Section 4.3.4.3 about the drums being non-hazardous.
- Updated Section 4.3.5.1 about Quarantine soil segregation.

Revision 14, dated 31 May 2018

- Annual review
- Updated Attachment 1 reference to new spreadsheet in Section 4.3.4.4
- Updated Section 4.3.4.5 to say TALS database
- Updated Attachment 1 to new form

Revision 13, dated 31 May 2017

- Annual review
- Added new Section 2.6.4 describing Sample Control's responsibilities for archiving
- Rewrote Sections 4.1.1 and 4.1.2 to match current procedure

Revision 12, dated 31 December 2016

- Annual review

Revision 11, dated 31 December 2015

- Added language to Section 2.6.3 to require that coolers with broken samples transferred to Waste Management include a description of the broken samples.

Revision 10, dated 31 October 2015

- Annual review
- Minor reformatting throughout
- Language corrections throughout
- Added RADACC definition

Revision 9, dated 31 October 2014

- Added section 4.1.2 to reflect the sample/job verification process when archiving samples/jobs.
- Added note to section 4.3.3 regarding the scanning of samples for disposal and possible "pop-ups".

Revision 8, dated 31 January 2014

- Formatting and grammatical corrections made throughout SOP
- Revised section 2.3.1 to reflect current practice
- Minor clarifications added throughout Sections 4.1 and 4.2
- Updated section 4.1.1 to reflect current report used
- Removed section 4.1.1.1 (redundant information)

- Added clarification to sections 4.2.5 and 4.3.1.1 on current practice
- Updated section 4.3.3.4 to reflect current database used
- Removed section 4.3.4.2 (no longer performed)

Revision 7, dated 30 June 2013

- Basic Annual Review

Revision 6.2, dated 22 June 2012

- Basic Annual Review

Revision 6.1, dated 23 May 2011

- Basic Annual Review
- Updated the language to read TALS in place of LIMS

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Drum Discharge Approval Sheet (Example)

TestAmerica

Discharge Approval Sheet

Waste Technician:		Date:	
Drum Number:		TALS ID:	

Test:	ICP	Hg	PCE
Date Analyzed:			

Average Water Effluent Per Day:	15811	liters

Amount in Drum:	208	liters

Analyte	Result	Units	Average Effluent Concentration	Units	CCVWD Effluent Limit	Units	Evaluation	RCRA Limit	Units	Shipping Classification
Arsenic		mg/L	0.0132	mg/L	0.33	mg/L	Passed	5.0	mg/L	Non-RCRA
Barium		mg/L	0.0132	mg/L	100	mg/L	Passed	100.0	mg/L	Non-RCRA
Cadmium		mg/L	0.0132	mg/L	3.4	mg/L	Passed	1.0	mg/L	Non-RCRA
Chromium		mg/L	0.0132	mg/L	3.6	mg/L	Passed	5.0	mg/L	Non-RCRA
Copper		mg/L	0.0132	mg/L	6.1	mg/L	Passed	NA	mg/L	Non-RCRA
Lead		mg/L	0.0132	mg/L	2.2	mg/L	Passed	5.0	mg/L	Non-RCRA
Molybdenum		mg/L	0.0132	mg/L	0.14	mg/L	Passed	NA	mg/L	Non-RCRA
Nickel		mg/L	0.0132	mg/L	5.6	mg/L	Passed	NA	mg/L	Non-RCRA
Selenium		mg/L	0.0132	mg/L	0.66	mg/L	Passed	1.0	mg/L	Non-RCRA
Silver		mg/L	0.0132	mg/L	2.9	mg/L	Passed	5.1	mg/L	Non-RCRA
Zinc		mg/L	0.0132	mg/L	15.6	mg/L	Passed	NA	mg/L	Non-RCRA
Mercury		mg/L	0.01	mg/L	0.13	mg/L	Passed	0.2	mg/L	Non-RCRA
PCE		mg/L	0.01	mg/L	1.5	mg/L	Passed	1.0	mg/L	Non-RCRA

If any CCVWD Acceptable response indicates a response other than "Pass", the drum must be evaluated. The drum can be either discharged over a period of days (no more than 3 days) or must be shipped offsite. A shipping classification indicating a RCRA Hazardous material means the drum must be shipped as RCRA Hazardous if the drum is shipped offsite. All RCRA Hazardous materials must be listed on the shipping manifest.

DV-F-0075-5-25-2018-Rev 0

Page 1 of 1

Attachment 2

Radiological Discharge to CCVWD Evaluation Sheet (Example)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Denver

Alpha Calculation

Month May-04

10500 l/day	31 days/mont	2000 pCi/l	6.51E+08 pCi/month

Drum #	Drum's Alpha Activity	Drum Volume	Total Beta Activity in Drum	Remining Activity After Discharge
w10c13-14	1.60E+03 pCi/l	208 l	3.33E+05 pCi	6.51E+08 pCi
J10C03-5	1.90E+03 pCi/l	208 l	3.95E+05 pCi	6.50E+08 pCi
F10C03	0.00E+00 pCi/l	208 l	0.00E+00 pCi	6.50E+08 pCi
w10c15-16	1.04E+04 pCi/l	208 l	2.16E+06 pCi	6.48E+08 pCi
w10c17-19	2.10E+03 pCi/l	208 l	4.37E+05 pCi	6.48E+08 pCi
F10C05-6	3.00E+02 pCi/l	208 l	6.24E+04 pCi	6.48E+08 pCi
W10D01-4	5.40E+03 pCi/l	208 l	1.12E+06 pCi	6.46E+08 pCi
J10D01	1.80E+03 pCi/l	208 l	3.74E+05 pCi	6.46E+08 pCi
T10D01	5.00E+02 pCi/l	208 l	1.04E+05 pCi	6.46E+08 pCi
W10D05-9	3.40E+03 pCi/l	208 l	7.07E+05 pCi	6.45E+08 pCi
F10D02	3.00E+02 pCi/l	208 l	6.24E+04 pCi	6.45E+08 pCi
w10d10-15	1.10E+03 pCi/l	208 l	2.29E+05 pCi	6.45E+08 pCi
	pCi/l	208 l	0.00E+00 pCi	pCi
	pCi/l	208 l	0.00E+00 pCi	pCi
	pCi/l	208 l	0.00E+00 pCi	pCi
	pCi/l	208 l	0.00E+00 pCi	pCi

3.10E+06 max pCi/l concentration in a drum remaining

Beta Calculation

Month May-04

10500 l/day	31 days/mont	300 pCi/l	9.77E+07 pCi/month

Drum #	Drum's Beta Activity	Drum Volume	Total Beta Activity in Drum	Remining Activity After Discharge
w10c13-14	4.80E+03 pCi/l	208 l	9.98E+05 pCi	9.67E+07 pCi
J10C03-5	3.10E+03 pCi/l	208 l	6.45E+05 pCi	9.60E+07 pCi
F10C03	2.40E+03 pCi/l	208 l	4.99E+05 pCi	9.55E+07 pCi
w10c15-16	1.40E+03 pCi/l	208 l	2.91E+05 pCi	9.52E+07 pCi
w10c17-19	4.70E+03 pCi/l	208 l	9.78E+05 pCi	9.42E+07 pCi
F10C05-6	8.00E+02 pCi/l	208 l	1.66E+05 pCi	9.41E+07 pCi
W10D01-4	3.20E+03 pCi/l	208 l	6.66E+05 pCi	9.34E+07 pCi
J10D01	2.10E+03 pCi/l	208 l	4.37E+05 pCi	9.30E+07 pCi
T10D01	4.30E+03 pCi/l	208 l	8.94E+05 pCi	9.21E+07 pCi
W10D05-9	6.40E+03 pCi/l	208 l	1.33E+06 pCi	9.07E+07 pCi
F10D02	0.00E+00 pCi/l	208 l	0.00E+00 pCi	9.07E+07 pCi
w10d10-15	2.00E+02 pCi/l	208 l	4.16E+04 pCi	9.07E+07 pCi
	pCi/l	208 l	0.00E+00 pCi	pCi
	pCi/l	208 l	0.00E+00 pCi	pCi
	pCi/l	208 l	0.00E+00 pCi	pCi
	pCi/l	208 l	0.00E+00 pCi	pCi

4.36E+05 max pCi/l concentration in a drum remaining

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Acid Digestion of Aqueous Samples for Metals Analysis by ICP

Approvals (Signature/Date):

Doug Gomer 7/31/18
Doug Gomer Date
Technical Specialist

Doug Gomer 7/31/18
Doug Gomer Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 7/31/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 7/31/18
Richard Clinkscales Date
Laboratory Director

Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the acid digestion of aqueous samples by EPA Method 200.7, SW-846 Method 3005A or SW-846 Method 3010A prior to the determination of the concentration of individual metallic elements by inductively coupled plasma atomic emission spectroscopy (ICP). These methods include digestions for total, total recoverable, dissolved, and potentially dissolved analytes (see definitions in Section 3).

1.2 This SOP is applicable to ground water, surface water, domestic and industrial wastewater, TCLP leachates, and other aqueous media. This SOP is not applicable to oils or other liquids that are not miscible with water.

NOTE: Samples that are found to be immiscible with water, e.g., contain oil or other immiscible organic solvents, are subcontracted to other labs that are capable of handling such samples. If during the preparation process it is discovered that the sample is immiscible with water or is biphasic, the analyst notifies the Technical Specialist and Project Manager, who can subcontract the samples to a laboratory with the capability to handle the sample.

1.3 The following table summarizes the applicability of the various digestion methods referenced in this SOP. All sample digestates are analyzed by ICP in accordance with SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

Method	Title	Summary	SOP Section
3005A/200.7_Prep	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP	Preparation of surface and ground water samples for total recoverable or dissolved metals for analysis by ICP.	10.5
3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP	Preparation of aqueous samples, EP and mobility procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.	10.8

1.4 Sample digestion requirements are established by the laboratory Project Manager before samples are received. TestAmerica LIMS (TALS) method codes are applied to samples at Login to indicate which digestion is to be used for each sample.

1.5 This procedure can be used for all of the elements listed in Table 1. Additional elements may be analyzed using the digestion methods in this SOP provided the method performance criteria specified in Section 12 and the Quality Control (QC) acceptance criteria specified in Section 9 of this SOP and the ICP determinative SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021 are met.

1.6 All samples require digestion prior to analysis, with the possible exception of "direct analysis" of dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples. This must be determined by the laboratory Project Manager before projects start, and is communicated to the analysts through Method Comments in TALS.

2.0 Summary of Method

2.1 Method 3005A/200.7_Prep, Total Recoverable, Dissolved Metals or Potentially Dissolved Metals

A representative portion of sample is heated with diluted nitric and hydrochloric acids until substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2 Method 3010A Total Metals

A representative portion of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary), and brought up to volume.

3.0 Definitions

3.1 Dissolved Analyte: The concentration of analyte in an aqueous sample that will pass through a 0.45-µm membrane filter prior to acidification (sample is acidified after filtration).

3.2 Potentially Dissolved Metals: The concentration of elements in solution after acidifying the sample with nitric acid to pH < 2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-µm membrane filter. This definition is based on the Colorado surface water regulations.

3.3 Total Recoverable Analyte: The concentration of analyte determined by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s).

3.4 Total Metals: The concentration of elements in an unfiltered sample subject to a more rigorous nitric acid / hydrochloric acid digestion than is used for total recoverable metals.

3.5 General Analytical Terms: Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, “Quality Assurance Program,” for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work

areas, and atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not miscible with acids. If physical interferences are present, they should be documented in the final report case narrative.

4.3 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented in the final report case narrative.

4.4 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.5 Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. Method 3005 or 3010 samples containing more than 1 mg/L silver are redigested at a reduced sample volume and reanalyzed to produce more accurate results. Method 200.7 requires samples to be redigested if the silver is greater than 0.1 mg/L.

4.6 Specific analytical interferences are discussed in the ICP determinative methods. See SOPs DV-MT-0012, DV-MT-0019, and DV-MT-0021.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids are added.

5.3.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the digested samples.

5.4 Primary Materials Used

5.4.1 The following is a list of the materials used in this method which have a serious or significant hazard rating.

5.4.2 A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time.

Material (1)	**Hazards**	**Exposure Limit(2)**	**Signs and Symptoms of Exposure**
Stock Standard Solutions	Oxidizer Corrosive Poison	5 mg/m^3 as HNO_3	Toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid (HNO_3)	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (HCl)	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 Digestion blocks, with adjustable heating, capable of maintaining a sample temperature of 90 - 95 °C.

6.1.2 Thermometer that covers a temperature range of at least 80 - 110 °C, in increments of 1 °C.

6.1.3 Liquid-filled thermometers must have a tag indicating that the accuracy was checked by the QA group within the last 12 months.

6.1.4 Digital thermometers must have a tag showing that they were checked within the last three months.

6.1.5 See SOP DV-QA-0001 for details of the thermometer calibration procedure.

6.1.6 Centrifuge (when the desired method of removing particulates is centrifugation).

6.1.7 Calibrated mechanical pipettes with disposable pipette tips. Pipette calibration is checked in accordance with SOP DV-QA-0008.

6.2 Supplies

6.2.1 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.2 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.3 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters (No. 6973-2504), for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.4 Syringes or equivalent filtration apparatus.

6.2.5 Re-pipettors or suitable reagent dispensers.

6.2.6 Class A volumetric graduated cylinders.

6.2.7 pH indicator strips.

6.2.8 Plastic digestate storage bottles.

7.0 Standards and Reagents

7.1 Standards must be NIST traceable, where available. Multi-element standards are verified against a second-source standard before they are put into use (the only exception is standards purchased directly from NIST), which is described in SOP DV-QA-0015.

7.2 Stock standards are purchased as custom multi-element mixes or as single-element solutions. Standards are logged into the TALS Reagent Module and are assigned unique identification numbers that can be used to access traceability information.

7.3 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles.

7.4 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.5 Standards containing silver must be protected from light using either a cardboard box or amber containers.

7.6 Shelf-Life

7.6.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.6.2 Intermediate concentration standards or working standards may be used for up to six months. The expiration date cannot be later than the date assigned to the stock standard.

7.6.3 Any suspect standards are re-verified, and replaced if re-verification fails.

7.7 Laboratory Control Sample (LCS) Spike Stock Standards

The LCS spike stock standards are custom-made standards purchased from Inorganic Ventures. The standards are designated ICP-SPK-3A (ICP-1) and ICP-SPK-2B (ICP-2) and contain the following elements at ready-to-use concentrations:

LCS Spike Stock Standards

Elements in LCS Spike	Concentration in ppm (µg/mL)
Ca, K, Mg, Na	5,000
P, Si	1,000

Elements in LCS Spike	Concentration in ppm (µg/mL)
Al, Ba, Bi, Se, Tl, U, Sn, S	200
Fe, Sr, Li, B, Mo, Ti, As, Th	100
Co, Mn, Ni, Pb, V, Zn, Sb, Zr	50
Cu	25
Cr	20
Cd	10
Ag, Be	5

7.8 TCLP Spike Stock Standard (TCLP Spike)

The TCLP spike stock standard is purchased from commercial sources. The stock is a custom-made standard purchased at ready-to-use concentrations and designated as TCLP Spike, as follows:

TCLP Spike Stock Standard

Elements in TCLP Spike	Concentration in ppm (µg/mL)
Ba	1,000
Cr, Pb	500
As	300
Cu, Zn	200
Ag, Cd, Se	100

7.9 TCLP Mercury Spike Solution

TCLP leachate matrix spike samples are spiked for both ICP elements and mercury at the time of sample preparation but before preservation. The mercury spike standard is prepared by the mercury analyst as the mercury daily spike solution (Hg Daily Spk) at a concentration of 100 µg/L (SOP DV-MT-0015).

7.10 Reagent Water

Reagent water must be produced by a Millipore de-ionized system or equivalent and must achieve the performance specifications for ASTM Type II water, i.e., conductivity < 1.0 µmhos/cm; resistivity > 1.0 megohms-cm; silica < 3.0 µg/L. In

addition, the reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs DV-MT-0012, DV-MT-0019, and DV-MT-0021.

7.11 Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.12 Hydrochloric acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	HDPE	500 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD/DOE QSM 5.0 or 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. Ongoing proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details on initial demonstrations of capability, analyst training and qualification.

9.2 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

9.3 Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are not included in the sample count unless specifically requested by the client. The prep batch consist of the laboratory generated QC and no more than twenty field samples.

9.4 Method Blank (MB)

9.4.1 The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples.

9.4.2 TCLP method blanks are prepared by taking 10 mL of TCLP leachate fluid (see SOP DV-IP-0012) through the appropriate procedure as described in Section 10. TCLP method blanks are referred to as LB (extraction fluid 1) and LB2 (extraction fluid 2) in TALS and on the final reports.

9.4.3 One method blank must be processed with each preparation batch. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data. Method blank results are evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

9.4.4 Acceptance Criteria

The method blank should not contain any analyte of interest at or above ½ the reporting limit (RL) or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher. In other words, the sample result must be a minimum of 10 times higher than the blank contamination level. Method blank results that are greater than ½ the RL may also be reported if the associated sample results fall below the RL and the client accepts the data.

9.4.5 Corrective Action

If the method blank does not meet the acceptance criteria, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.5 Laboratory Control Sample (LCS)

9.5.1 One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples.

9.5.2 An LCS for a batch of aqueous samples is prepared by adding 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), to 50 mL of reagent water. This produces the final concentrations shown in Table 1.

9.5.3 An LCS for a TCLP batch is prepared by adding 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), plus 0.5 mL of the TCLP Spike stock standard (Section 7.8) to 50 mL of the TCLP leachate solution (see SOP DV-IP-0012). This produces the final concentrations shown in Table 2.

9.5.4 The LCS is used to monitor the accuracy of the analytical process. LCS results are evaluated by the ICP analyst as described in SOP DV-MT-0012. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.5.5 Acceptance Criteria

LCS recovery control limits are set at ± 3 standard deviations about the historical mean. These limits must not be wider than 85 - 115 % recovery

for Method 200.7 or 80 - 120 % for Method 6010. The control limits are maintained in TALS.

9.5.6 Corrective Action

If the LCS percent recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be documented in an NCM and explained in the case narrative.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Normally, one MS/MSD pair is digested with each preparation batch. Samples identified as field blanks, equipment blanks, or rinse blanks are not appropriate for use as the batch MS/MSD.

9.6.2 Some programs (e.g., South Carolina and North Carolina) require that MS/MSD pairs are run at a 10% frequency. Also, some clients may require unspiked duplicate samples in place of or in addition to an MS/MSD pair. Check special project instructions attached as Method Comments in TALS and any project QASs before starting the batch.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, the LCS and LCSD are used to measure precision.

9.6.3 If insufficient sample is available to process an MS/MSD pair, then a duplicate LCS must be processed and an NCM generated. The LCS pair is then evaluated according to the MS/MSD criteria. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

9.6.4 The purpose of analyzing matrix spike samples is to assess the effect of the sample matrix on the accuracy and precision of the analysis. MS/MSD results are evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021. If the MS/MSD results fail to meet control limits while the LCS results are in control, then something about the sample matrix is interfering with the analysis.

9.6.5 Matrix spikes for aqueous sample batches are prepared by adding 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), to a digestion tube containing 50 mL of the selected sample. The final spike concentrations are shown in Table 1.

9.6.6 Matrix spikes for TCLP batches are prepared by adding 0.5 mL of the TCLP Spike stock standard (Section 7.8) plus 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), to 50 mL of the parent TCLP aliquot. A second aliquot is spiked for mercury analysis at by adding 1.5 mL of the 100 mg/L Hg standard (Hg Daily Spk) to 30ml of parent sample. The matrix spike samples are then preserved with HNO_3 to pH < 2. The final spike concentrations are shown in Table 2.

NOTE: The MS and MSD must be spiked prior to preservation of the leachate.

9.6.7 Acceptance Criteria

The recovery for each analyte must fall within established limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. If any analyte recovery or relative percent difference (RPD) between the MS and MSD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS.

9.6.8 Corrective Action

If MS/MSD results fail to meet control limits, but the LCS results are within limits, then samples do not require re-preparation and reanalysis unless the results indicate that a spiking error may have occurred. If the recovery of the LCS also failed acceptance criteria, then corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. One possible exception is an LCS recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be documented in an NCM and explained in the case narrative.

9.7 Continuing Calibration Verification Standard (CCV)

Continuing calibration verification standards (CCVs) are not digested but are instead created and evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

9.8 Second-Source Initial Calibration Verification (ICV) Standard

Initial calibration verification standards (ICVs) are not digested but are instead created and evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a non-conformance memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 All data shall be recorded directly on the described forms, logbooks, electronic forms, or directly in TALS at the time of data generation. It is not acceptable to record data on loose papers, scraps of paper, gloves, sample vials, or "Post-It" notes. Data may be recorded on paper bench sheets if the sheets are subsequently scanned and saved in a designated folder on the company server.

10.4 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.5 Sample Preparation

10.5.1 Samples are typically logged in as either water or solid. Waste such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), contact the project manager and the laboratory Technical Specialist for further instructions. It may be necessary to subcontract these samples to a laboratory with the capability to digest organic matrices.

NOTE: TestAmerica Denver has not implemented digestion methods for water-immiscible organic matrices, e.g., oils. Samples that are known to be incompatible with TestAmerica Denver digestion techniques are typically subcontracted to other laboratories.

10.5.2 All samples are to be electronically checked out of sample control using the TALS Internal Chain of Custody (ICOC) module.

10.5.3 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.5.4 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through

the lab, data review and reporting.

10.5.5 Guidelines are provided in Appendix 1 on procedures to minimize contamination of samples and standards.

10.6 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure. The sample preparation procedures for Methods 3005A and 3010A detailed in the following sections are also summarized in work instruction WI-DV-016.

10.6.1 Verify sample pH

10.6.1.1 Measure the sample pH with pH paper using a separate aliquot of sample. This can be done using disposable plastic droppers or pouring the sample on to the pH paper. Do not put the pH paper directly into the bottle. Record the pH on a copy of the internal chain of custody (ICOC). When all of the samples have been tested, initial and date the copy of the ICOC, scan it, and save it to the Metals folder on the G: drive.

10.6.1.2 All water sample pH's must be verified and documented in the batch record before digestion.

10.6.1.3 If the pH>2 for a sample requiring acidic preservation, record the job in the Sample Filtration and Preservation Logbook.

10.6.1.4 If laboratory preservation is required, add 1-2 mL of conc. HNO_3 to the sample. Replace the lid and mix the sample. If the pH is still >2 add another addition of HNO_3. Do not add more than 5 mL. If the pH is still >2 create an NCM saying the sample will not preserve.

10.6.1.5 Allow the sample to sit for 24 hours following acidification.

10.6.1.6 Recheck the pH of the sample. If the pH>2, repeat Section 10.5.1.4 until the pH holds at <2 or 5 mL of HNO_3 has been added. If the pH is still >2 after the addition of 5 mL of HNO_3 create an NCM saying the sample will not preserve.

10.6.1.7 Samples cannot be digested for 24 hours after preservation. Note the date/time of this pH recheck in the Metals Prep Log in TALS.

10.6.1.8 Leachates or portions of leachates for metallic analyte deteminations must be acidified with nitric acid to a pH <2 unless precipitation occurs. Test a small portion of sample to see if precipitation occurs. If a precipitate forms do not acidify the leachate and analyze as soon a possible. Leachates may be digested as soon as they are acidified.

10.6.2 Select the unfiltered fraction for a total or total recoverable analysis or the filtered fraction for a dissolved analysis. If requested by the client, select the filtered fraction for a total dissolved analysis. For TCLP and SPLP, select the proper sample leachates.

10.6.2.1.1 Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number.

10.6.2.1.2 Samples and batch QC requiring filtration are to be put into a filtration batch. A filtration batch is to have no more than 20 samples.

10.6.2.1.3 Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples. The performance of the filtration process is recorded in TALS.

10.6.3 Mix the sample by shaking the container.

10.6.4 Measure and transfer 50 mL of the sample into a digestion tube (record the lot number of the digestion tubes used in TALS). When using calibrated digestion tubes, pour the sample into the tube to the 50 mL mark. For TCLP sample batches pour 10 mL of samples and bring to 50 mL with reagent water. Unless specifically required for a project, all samples are measured by volume and not by weight. Record the volume and units on the preparation bench sheet in TALS. If the digestion cup is filled beyond the required mark, the excess sample must not be poured back into the original container, but must be disposed of as waste.

10.6.5 Mix the sample by shaking the container and then measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot as described in Section 9.6. Refer to Section 9.6.6 for specific instructions for spiking the selected TCLP sample. Record the standards and pipette identifications in TALS.

10.6.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested (TALS 3005A), use filtered reagent water for the method blank. For TCLP sample batches, measure 10 mL of the TCLP leachate solution and bring to 50 mL with reagent water for the blank. See Section 9.4 for a detailed description of the method blank.

10.6.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add the spiking solutions as described in Section 9.6.2. For TCLP sample batches, use 10 mL of TCLP leachate fluid and bring to a final volume of 50 mL with reagent water for preparing the LCS (Section 9.5.3). Record the standards and pipette identifications in TALS. If determination of dissolved metals is requested and one or more samples were filtered in the laboratory, then filter the LCS using a filter of the same type that was used to filter the sample(s).

10.6.8 If the analysis is for total recoverable, dissolved metals, or potentially dissolved metals, continue on with Section 10.5. If the analysis is for total metals, skip Section 10.6 and go to Section 10.7.

10.7 Total Recoverable, Dissolved, or Potentially Dissolved Digestion for Waters by 3005A and 200.7_Prep.

10.7.1 Add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl to the sample in the digestion tube.

10.7.2 Heat at 90 - 95 °C until the volume is reduced to approximately 10 mL. Record the start and stop times, digestion block temperature (observed and corrected) and the thermometer ID in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.7.3 Allow the digestion tube to cool in a fume hood.

10.7.4 Wash down the digestion tube walls with reagent water.

10.7.5 Add 1.5 mL of concentrated HNO_3 to the digestate.

10.7.6 Revolume to 50 mL with reagent water. Cap and shake to mix.

10.7.7 If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

NOTES: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples may be diluted and mixed and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

10.7.8 The sample is now ready for analysis.

10.8 Total Metals Digestion for Waters or TCLP Leachates by 3010A

10.8.1 Add 1.5 mL of concentrated HNO_3 to the sample in the digestion tube.

10.8.2 Heat at 90 - 95 °C until volume is reduced to approximately 5 mL. Record the start and stop times, digestion block temperature (observed and corrected) and the thermometer ID in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.8.3 Allow the digestion tube to cool in a fume hood.

10.8.4 Add another 1.5 mL portion of concentrated HNO_3 and cover the sample with a watchglass.

10.8.5 Continue refluxing until the digestion is complete.

NOTE: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient. Additional aliquots of nitric acid may be added if necessary.

10.8.6 Evaporate to a low volume of 5 to 10 mL. If the sample does go to dryness, the digestion must be started over using a fresh portion of sample.

10.8.7 Allow the digestion tube to cool in a fume hood.

10.8.8 Add 2.5 mL of concentrated HCl.

10.8.9 Cover and reflux for an additional 15 minutes to dissolve any precipitate or residue.

10.8.10 Wash down the digestion tube walls and watch glass (or digestion tube cover) with reagent water.

10.8.11 Adjust to 50 mL final volume with reagent water. This must be done volumetrically, and not using a balance.

10.8.12 If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

NOTES: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples may be diluted and mixed and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

10.8.13 The sample is now ready for analysis.

10.9 Calibration

10.9.1 The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded in TALS. The temperature must be monitored by measuring the temperature of reagent water contained in a capped digestion tube that is placed in each digestion block. The thermometer used and the start and end times for all temperature cycles are recorded in TALS.

10.9.2 The thermometer is calibrated in accordance with SOP DV-QA-0001, Thermometer Calibration Procedures.

11.0 Calculations / Data Reduction

11.1 This SOP does not produce any analytical data. See the determinative method SOPs DV-MT-0012, DV-MT-0019 or DV-MT-0021 for data analysis and applicable calculations.

11.2 Documentation

11.2.1 All of the preparation information is recorded and stored in TALS.

11.2.2 The preparation information includes:

11.2.2.1 Batch number, job and sample numbers, preparation date, and analyst name;

11.2.2.2 Matrix and prep type;

11.2.2.3 Initial sample pH, Initial sample volume and final volume;

11.2.2.4 Reagent manufacturer and lot number for each reagent used;

11.2.2.5 Digestion tube lot information;

11.2.2.6 Standard identification number for each standard used;

11.2.2.7 Start and stop times for digestions;

11.2.2.8 Observed and corrected temperature readings during digestion;

11.2.2.9 Identification numbers of calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

11.3 Reporting

11.3.1 Reporting units are mg/L for water samples.

11.3.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.3.3 All associated data are entered or uploaded into TALS as required.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.4 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with TestAmerica SOP CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. MDL verifications are performed quarterly.

12.1.2 The current MDL values are maintained in TALS.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1 - 2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.2 Method 200.7, Determination of Metals And Trace Elements In Water And Wastes By Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, 1994.

16.0 Method Modifications

16.1 Modifications Specific to MCAWW Methods (200.7_Prep)

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 10. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness versus an exact volume).

16.2 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above ½ the reporting limit. Common laboratory contaminants are allowed up to the reporting limit in the blank following consultation with the client.

16.3 The referenced methods use 100 mL of sample for digestion. This SOP uses a 50 mL aliquot, with a proportional reduction in digestion reagents. This change is made to allow better control of temperature and potential sample contamination with the use of the digestion block. It is also considered one of the laboratory's hazardous waste reduction initiatives.

16.4 The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document states "flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1)

and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.0 Attachments

Table 1. Matrix Spike and Aqueous Laboratory Control Sample Levels

Table 2. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels

Appendix 1. Contamination Control Guidelines

18.0 Revision History

- Revision 11 dated 31 July 2018
  - Annual technical review
  - Updated Section 10.7.2 to reduce volume to approximately 10 mL
  - Updated Section 10.8.2 to reduce volume to approximately 5 mL

- Revision 10 dated 30 June 2017
  - Annual technical review
  - Updated Section 9.1.2 to include QSM 5.1
  - Added current Section 10.4 referencing support equipment IDs and renumbered remaining sections
  - Added Sections 11.3 and 11.4
  - Updated Section 12.1.1 MDLV language for consistency with other SOPs
  - Added current Section 12.2 regarding LOQVs and renumbered remaining sections
  - Updated the language in Sections 12.3 and 12.4 for consistency with other SOPs

- Revision 9 dated 30 June 2016
  - Annual technical review
  - Added "document in the batch record" regarding sample pH to Section 10.5.1.2.
  - Updated Section 9.5.2 to say 10ml of TCLP fluid is added to the method blank.
  - Updated Section 10.5.1.5 to wait 24 hours after acidification before checking pH
  - Converted the note in Section 10.5.2 into subsections 10.5.2.1.1 - 10.5.2.1.3
  - Added definition of filtration batch to section 10.5.2.1.2
  - Updated Section 16.2 to say we control common laboratory contaminates to the reporting limit

- Revision 8 dated 30 June 2015
  - Updated Section 10.5.1.1 to include statement about not putting the pH paper into the bottle
  - Added language to Section 4.5 for clarification
  - Added new Section 10.3 reminding analysts to enter data directly at time of acquisition

- Revision 7 dated 31 October 2014
  - Annual technical review
  - Removed reference to SOP DV-IP-0017 for oils in section 1.2
  - Added maximum silver concentration to section 4.5 for method 200.7
  - Updated standard ID's for sections 7.7 and 7.8 and added Sulfur to the spike list
  - Corrected intermediate standard expirations from three months to six months
  - Removed duplicate analyte spike levels in ICP spike standards

- Changed references from LIMS to TALS
- Corrected concentration of Hg Daily spike standard
- Removed Figures 1 and 2
- Corrected various grammar and language errors
- Corrected analyte spike levels in Table 1

- Revision 6 dated 08 October 2013
  - Updated sections 10.4.1.3, 10.4.1.4 and 10.4.1.6 about preservation procedure and removed the comment about recording the amount of acid added in the preservation logbook

- Revision 5, dated 15 July 2013
  - Annual review
  - Changed section 10.5.5, 7.3, 9.4, 9.5.2, 9.5.4, 10.3.1, 10.3.2, 10.4, 10.4.4, 10.5.2, 10.6.2, 11.2.2, 12.1.1 and 12.3 to reflect current practices
  - Corrected formatting and grammatical errors
  - Clarified sample matrices for this method in section 1.2
  - Corrected references in table associated with section 1.3
  - Added ICP determinative SOPs to sections 1.5, 4.6, 7.10, 9.5.3, 9.7.4
  - Added 200.7_Prep whenever 3005A was referenced
  - Edited section 3.5 to reflect current reference
  - Removed note associated with section 5.4.1
  - Added SOP reference to section 6.2.1
  - Removed references to Denver Standards Log and replaces those references with TALS reagent module
  - Correct standard names in section 7.7
  - Removed references to Supplemental Metals Prep Sheet
  - Updated sections 10.4.4, 10.4.6 and 10.4.7 for 10 mL TCLP sample aliquot
  - Added reference to 200.7 in Section 15

- Revision 4.7, dated 18 July 2012
  - Annual review
  - Updated Section 9.1, 10.1 and 10.2 to reflect current practice
  - Updated Section 9.7.6 on spiking TCLP aliquots
  - Added section 10.4.1.9 for TCLP preservation
  - Removed Appendix 2. Added reference to work instruction in Section 10.4
  - Updated Figures 1 and 2 to reflect current practice.
  - Formatting and editorial changes throughout

- Revision 4.6, dated 24 August 2011
  - Added recommendation to use disposable bulbs for pH checking in section 10.8.1.
  - Added requirement to store samples with a Rush form after preserving in section 10.8.1.2.

- Revision 4.5, dated 31 January 2011
  - Change note in section 10.8.1.8 to be 24 hours before preparation.

Earlier revision histories have been archived and are available upon request.

Table 1.

Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	LCS Concentration (µg/L)	Matrix Spike Concentration (µg/L)
Aluminum	2,000	2,000
Antimony	500	500
Arsenic	1,000	1,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	100	100
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO2)	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Thallium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	500	500

Table 2.

TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels

Element	RL (mg/L)	Regulatory Limit (mg/L)	Spike Level (mg/L)
Arsenic	0.1	5,000	5.0
Barium	1.0	100,000	12.0
Cadmium	0.05	1,000	1.05
Chromium	1.0	5,000	5.2
Lead	0.03	5,000	5.5
Selenium	0.05	1,000	3.0
Silver	0.1	5,000	1.05

Appendix 1.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) [Method No(s). SW846 1311 and 1312]

Approvals (Signature/Date):

Andrew Pepping 12/3/18 Technical Specialist Date	Doug Gomer 12/3/18 Doug Gomer Date Health & Safety Manager / Coordinator
Roxanne Sullivan 12/4/18 Roxanne Sullivan Date Quality Assurance Manager	Richard Clinkscales 12/4/18 Richard Clinkscales Date Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW-846 Method 1311. The Toxicity Characteristic (TC) of a sample is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a sample. The TC is one of four criteria in 40 CFR Part 261 to determine whether a sample is classified as a hazardous waste. The other three are corrosivity, reactivity and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the landfill fluids will dominate the acid/base characteristics of the waste. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2 The specific list of TC analytes and regulatory limits may be found in Attachment 1.

NOTE: The list in Attachment 1 does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with TestAmerica Laboratories Technical Specialists before establishing the instrumental methods for these regulations.

1.3 This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW-846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Attachment 1. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4 The procedure is applicable to liquid, solid, and multiphase wastes. Currently TestAmerica Denver does not have the capability to digest organic wastes for metals analysis. Therefore if the sample produces a leachate that includes an organic phase, and the client is asking for metals analysis, TestAmerica Denver cannot accept the sample.

1.5 The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6 The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7 If a total analysis of the waste demonstrates that individual analytes are not present in the waste or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8 If an analysis of any one of the liquid fractions of the leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

2.0 Summary of Method

2.1 For liquid samples that contain less than 0.5% dry solid material, the sample, after filtration through 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP leachate and reagent water is used as the blank fluid.

2.2 For samples containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is leached with an amount of leach fluid equal to 20 times the weight of the solid phase. For TCLP, the leach fluid employed for the leaching of non-volatile analytes is a function of the alkalinity of the solid phase of the sample. For SPLP, the leach fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following leaching, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 μm fiber filter.

2.3 If the initial liquid phase of the sample (the filtrate) is miscible with the leachate, then they are combined, prepared, and analyzed together. If not miscible, the filtrate and leachate are analyzed separately and the results can be mathematically combined to yield a volume-weighted average concentration.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.2 Leachate: The TCLP solution generated after solids are tumbled with leaching fluid.

3.3 Filtrate: The liquid fraction of a sample that passes through a 0.6 to 0.8 μm fiber filter.

3.4 Final Leachate: The final solution generated from this procedure - either a leachate or a leachate combined with filtrate.

3.5 Leach Batch: A Leach Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same leaching procedure, reagents, and blank fluid type within the same time period. One TCLP leach blank (LB) will be prepared with each TCLP leachate batch.

3.6 Percent Wet Solids: The fraction of a sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4.0 Interferences

4.1 Oily samples may present unusual filtration and drying problems. Oils may contaminate the ZHEs and filtration apparatus. Therefore it is important to use filter apparatus designated to oily samples and do extra cleaning after filtration.

4.1.1 For oily wastes that filter completely, the filtrate is the leachate and should be sent on for analysis. If the client is requesting metals analysis on these wastes, the sample cannot be analyzed at TestAmerica Denver. For filterable oily wastes requiring semi-volatile organic analysis, the sample should be logged into the LIMS as a waste matrix for method 1311_T with a waste dilution extraction method 3580. For filterable oily samples requiring volatile organic analysis, the sample should be logged into the LIMS as a waste matrix for method 1311_Z with a 5030B_H prep method.

4.1.2 For oily wastes that do not filter completely, any filtrate will have to be logged as a separate sample according to Section 4.1.1 above while the portion of the sample that does not filter will have to be leached. The results from the leachate and the filtrate will have to be reported separately and then mathematically re-combined in proportion to give a final result.

4.2 Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing leach blanks as described in the Section 9.4 and the individual determinative SOPs.

4.3 Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.4 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.5 Over exposure of the sample to the environment will result in the loss of volatile components. Samples that are being leached for volatiles should be kept cold. They should not be removed from cold storage until immediately before aliquotting, or alternatively can be kept in an ice bath in the TCLP lab.

4.6 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized.

CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.2.3 A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene over-wraps of the glass jar, may be necessary.

5.2.4 Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.2.5 During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure. This is more common with samples being leached with TCLP fluid 2. If necessary, secure the lid with duct tape to ensure the vessel stays sealed for the entire leaching period.

5.2.6 Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

5.2.7 Do not attempt to manually stop a rotating piece of equipment. Keep all hanging objects, such as ties, hair, necklaces, etc., away from rotating equipment. Guards must be used when the apparatus is rotating to prevent loose clothing or limbs from getting caught.

5.2.8 Glass vials can break when the caps are being tightened. Cut resistant gloves should be worn whenever caps are being tightened.

5.2.9 When cleaning ZHE's a methanol rinse is used to remove any residual volatile compounds. After the rinse, the ZHE is put in an oven as a final cleaning procedure. It is very important that after the rinse the ZHE is allowed to dry for two hours in a fume hood before it is put in the oven. If this is not done then methanol vapor will acuminate in the oven resulting in a hazard. This hazard can cause a fire, explosion, or methanol exposure to the face and/or eyes when the door to the oven is opened.

5.2.10 After performing the procedure, the analyst must separate solid wastes from liquid wastes. This is done by filtering the waste through cloth. The corners and edges of the cloth are gathered together and the liquid is wrung out of the cloth into a drum. The cloth and the trapped solids are then immediately transferred to a waste container. No waste shall be left outside of a closed container.

5.3 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not contain all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.
Acetic Acid, Glacial	Corrosive Poison Flammable Liquid and Vapor	10 ppm (TWA)	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

NOTE: Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

6.1 Leach Vessels

6.1.1 For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Attachment 6). Cleaned by the following steps:

6.1.1.1 Remove the top and bottom flange from the barrel.

6.1.1.2 Remove support screens and o-rings from the top flange.

6.1.1.3 Remove the piston from the barrel and remove the o-rings and wiper seal from the piston.

6.1.1.4 Wash all parts in hot soapy water, rinse with hot tap water, and rinse with DI water.

6.1.1.5 Rinse top Flange, barrel, and piston with methanol, and allow to dry in a hood for at least 2 hours before placing in an oven heated to approximately 75ºC for at least 4 hours.

6.1.1.6 O-rings, wiper seals, and screens are placed in a disposable 2L HDPE bottle filled with methanol and tumbled 2-3 hours. They are then allowed to dry in a hood for at least 2 hours before placing in an oven heated to approximately 75ºC for at least 4 hours.

6.1.1.7 Disposable screens can be used instead of re-usable metal screens. Environmental Express part number F2090MM.

6.1.2 For metals - either disposable borosilicate glass jars (1 gallon, with Teflon lid inserts) or disposable 2 L HDPE (Nalgene® or equivalent) bottles may be used.

6.1.3 For non-volatile organics – disposable borosilicate glass jars must be used.

6.2 Vacuum Filtration Apparatus - Capable of 0 - 50 psi. For the filtering of leachates for metal analysis only as the apparatus is constructed of plastics. Cleaned by disassembling completely, washing with warm soapy water, rinsing with hot tap water, rinsing with DI water, and allowing to dry.

6.3 Stainless Steel Pressure Filtration Apparatus – 142 mm diameter. Capable of 0 - 50 psi. (See Attachment 7). For the filtering of leachates for semi-volatile organics and metals. For the percent wet solids determination. Cleaned by disassembling completely, washing with warm soapy water, rinsing with hot tap water, rinsing with DI water, rinsing with methanol, and allowing to dry.

6.4 Acid Washed, Low Metal, Borosilicate Glass Fiber Filters - 0.6 - 0.8 μm (Ahlstrom Grade 26). Certified for low metal content. 14.2 cm in diameter for pressure filter use. 4.7 cm in diameter for vacuum filter use. Glass fiber filters are fragile and should be handled with care.

6.5 Glass Fiber Filter Paper – 90 mm in diameter. For use in the ZHE.

6.6 Rotary Agitation Apparatus - Multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Attachment 6). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm. The RPM is checked before each use.

6.7 Gas-Tight Syringes - 100mL capacity, Luer Lock Hamilton 0158330 or equivalent

6.8 Top Loading Balance - Capable of 0g – 4000g ± 0.01g. The balance accuracy is verified each day of use in accordance with SOP DV-QA-0014.

6.9 pH Meter and Probe - Capable of reading to the nearest 0.01 unit, and with automatic temperature compensation. Calibrated daily. See Attachment 13 for detailed instructions.

6.9.1 Always use fresh aliquots of the pH buffers in fresh cups.

6.9.2 Always keep the probe immersed in pH electrode storage solution when not in use.

6.9.3 Calibrate the meter using buffers at pH 2, 4, 7, & 10.

6.10 Narrow Range pH Strips – Can be used to measure pH in place of the pH meter when dealing with especially oily samples that may damage the pH probe.

6.11 Magnetic Stirrer/Hotplate and Stirring Bars – For use in the leach fluid determination.

6.12 VOA Vials – 20 mL, with caps and septa. For the storage of leachates for volatile organic compounds analysis.

6.13 Glass Jars - 1/2 to 1 gallon, with Teflon lid-inserts. For the storage of leachates for sem-volatile organic compounds analysis.

6.14 Nalgene Plastic Bottles – 250mL to 1 L. For the storage of leachates for metals analysis.

6.15 Pipette - Calibration checked daily per SOP DV-QA-0008.

6.16 Bottle-top Pump – Calibration checked daily per SOP DV-QA-0008 to deliver 96.5mL of water.

6.17 Log Tag – An automated temperature data recorder used to monitor the temperature of the room during the 16-20 hour leach. See WI-DV-0067 for instructions on how to download the temperature readings.

6.18 Miscellaneous laboratory glassware and equipment.

6.19 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent Water **–** TestAmerica Denver has three ELGA Analytical water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026. Either water from the ELGA system or bottled HPLC grade water may be used in this procedure.

7.2 Hydrochloric Acid, 1 N "**1N HCl**" - For use in leach fluid determination. Add approximately 800mL of reagent water to a 1 liter Class A graduated cylinder. Using a 100mL Class A graduated cylinder, measure out 83mL of concentrated reagent grade HCl and carefully add the acid to the reagent water. Dilute to 1 liter with reagent water. Transfer to a 1 liter glass bottle, cap and shake to mix well.

7.3 69%-70% Trace Grade Nitric Acid - For the preservation of final leachates prior to metals analysis. Purchased ready to use.

7.4 Sodium Hydroxide, 10 N **"10N_NaOH"**- For use in TCLP Fluid #1. Purchased ready to use

7.5 Glacial Acetic Acid **"Acetic Acid"**– For use in TCLP Fluid #1 and #2. Concentrated, reagent grade liquid (HOAc).

7.6 pH Calibration Solutions - Buffered to a pH of 2, 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis.

7.7 TCLP Leaching Fluids

The pH of both types of TCLP leaching fluids will be monitored and recorded daily before

use by mixing the fluid well and testing with a calibrated pH meter.

The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

7.7.1 TCLP Fluid #1: This reagent is prepared in a manner so that 5.7mL of glacial acetic acid and 64.3mL of 1 N NaOH is diluted to 1 liter in reagent water. When correctly prepared, the pH of this solution is 4.93 ± 0.05. The laboratory makes this reagent in large quantities by measuring 289mL of 10N NaOH and 256mL of glacial acetic acid and diluting up to 45L of reagent water. (Note that 289mL of 10N base is used instead of 2893mL of 1N base.) The reagent is mixed well as it is being prepared and the pH is checked. If the pH is not within the 4.93 ± 0.05 range, the fluid is not used.

7.7.2 TCLP Fluid #2: For every liter of fluid to be prepared, carefully add 5.7 mL glacial acetic acid and dilute up to volume with reagent water. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.7.3 For water samples that are determined to be less than 0.5% solids, the leach fluid used to prepare the leach blanks is reagent water.

7.8 60/40 Sulfuric Acid / Nitric Acid - (60/40 weight percent mixture H2SO4/HNO3) For use in SPLP fluids. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.9 SPLP Leaching Fluids

SPLP solutions are un-buffered. The pH of SPLP fluids will be checked daily prior to use. Mix well and check with a calibrated pH meter. If not within specifications, the fluid may be discarded and fresh fluid prepared or the fluid must be adjusted using additional acid or reagent water to achieve proper pH.

NOTE: All SPLP waste waters/wastes **must** be SPLP fluid 1, no matter if they are east or west of the Mississippi. If a sample is a wastewater/wastes, the PM will be logged as 1312E, which will indicate to the analyst to use SPLP Fluid #1.

7.9.1 SPLP Fluid #1: This fluid is used for soils from a site that is east of the Mississippi River. Add 60/40 weight percent mixture of sulfuric and nitric acids to approximately 20 liters of reagent water until the pH is 4.20 ± 0.05. Test with a calibrated pH meter. If the pH is above 4.25 add more acid until the pH is in range. If the pH is below 4.15 dilute by adding more reagent water. Use the spreadsheet described in Attachment 14 to determine how much water to add.

7.9.2 SPLP Fluid #2: This fluid is used for soils from a site that is west of the Mississippi River. Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. Test with a calibrated pH meter. If the pH is above 5.05, add more acid until the pH is in range. If the pH is below 4.95

dilute by adding more reagent water. Use the spreadsheet described in Attachment 14 to determine how much water to add.

7.9.3 SPLP Fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas. This fluid is also used as the blank fluid for SPLP water samples. If the samples are to be analyzed for common lab contaminates like acetone and methylene chloride by method 8260, the reagent water should first be boiled and purged per DV-MS-0010.

7.10 Metals Spike Standards

7.10.1 TCLP Spike – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 2.

7.10.2 ICP SPK 2B – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 3.

7.10.3 ICP SPK 3A – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 4.

7.10.4 Hg Daily Spk – Prepared in 1% nitric acid at the concentration listed in Attachment 5.

7.11 Methanol – Used in cleaning ZHEs and steel pressure filters.

7.12 Methylene chloride - used to aid in cleaning oil contaminated equipment.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

8.2 Samples being analyzed for metals only can be collected in either glass or polyethylene containers. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

8.3 When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes. Water samples should be collected in Teflon lined septum capped vials. Soil samples should be collected in Teflon line 4 oz jars. Both water and soils should be collected with minimal headspace and stored at 4 ± 2 °C). Samples should be opened only immediately prior to leaching. A second container should be supplied for the percent solids determination.

8.4 Samples should be refrigerated to 4 ± 2°C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5 The physical state or states of the waste and the analytes of concern determine the minimum TCLP sample collection size. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing.

8.5.1 For multi-phasic samples containing between 0.5% and 10% solids, several kilograms of sample are required to complete the analyses.

8.5.2 The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low-density sample materials, such as rags or vegetation, will require larger volumes of sample.

8.5.3 For liquid samples (less than 0.5% solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required.

8.5.4 If matrix spike or duplicate control samples are requested, additional sample volume is required.

8.5.5 If sufficient sample volumes were not received, analyses cannot be started and the project manager should be notified as soon as possible.

8.6 Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid, then no more acid shall be added and the leachate shall be analyzed as soon as possible.

8.7 All leachates for semi-volatile organic analysis should be stored under refrigeration (4 ± 2 °C) until analyzed.

8.8 Leachates for volatile analysis must be stored under refrigeration (4 ± 2 °C) in VOA vials filled to eliminate all headspace.

8.9 Samples are subject to appropriate treatment within the following time periods:

HOLDING TIMES (DAYS)				
PARAMETER	**COLLECTION TO START OF LEACH**	**START OF TCLP TUMBLE TO PREPARATION**	**START OF TCLP LEACH OR SEMIVOLATILE PREP EXTRACTION TO ANALYSIS**	**TOTAL ELAPSED TIME**
Volatiles	14	N/A	14	28
Semi-Volatiles	14	7	40	61
Mercury	28	N/A	28	56

HOLDING TIMES (DAYS)				
PARAMETER	**COLLECTION TO START OF LEACH**	**START OF TCLP TUMBLE TO PREPARATION**	**START OF TCLP LEACH OR SEMIVOLATILE PREP EXTRACTION TO ANALYSIS**	**TOTAL ELAPSED TIME**
Other Metals	180	N/A	180	360

NOTE: The hold is the same for water and solids.

NOTE: The initial holding time is measured from date of collection to date TCLP leach started. (This should be the TCLP leach date in LIMS.) Semi-volatile method prep holding time is measured from the day leach was started to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is

described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batching Samples - Samples that are less than 0.5% solids (i.e. liquid samples) are batched separately from samples that are greater than 0.5% (i.e. solid samples or multi-phasic samples.)

9.3 A Leach Batch is a set of up to 20 field samples of similar general matrix (i.e greater than 0.5% solids or less than 0.5% solids) that behave similarly and are processed using the same leaching procedure, reagents, and blank fluid type within the same time period. One TCLP leach blank (Method Blank) will be prepared with each TCLP leachate batch.

9.4 TCLP Leach Blanks - One blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples leached that day in a particular vessel type. The leach blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). Leach fluid is tumbled with the samples in the same type of leach vessel (see Section 6.1) and filtered using the same filtration apparatus (see Section 6.2 and 6.3). Zero Headspace Extraction vessels are uniquely numbered. Each time a new batch is set up the blank should be rotated randomly to a different vessel to ensure all vessels are periodically checked. A vessel cannot be used in the leaching of more than 20 samples before it is used for the leaching of a blank. This is documented in the ZHE spreadsheet.

9.5 Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be created at the time of the preparative digestion or extraction by spiking an aliquot of the appropriate leach fluid used for that batch. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.6 Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. An MS/MSD pair is required with each batch. When an MS/MSD pair is not available, an LCS and LCSD are to be used to measure precision.

NOTE: Some clients interpret Section 8.2 of SW-846 1311 to mean that a matrix spike must be performed for each specific sample matrix. In other words, if the samples in the batch are visually distinct (clay, soil, sand, wood, plastic, metal) the lab must perform a MS/MSD on each distinct sample matrix type. If the client interprets the method in this way, this will be communicated through the Method Comment “MS per Specific Matrix”.

9.7 MS/MSD samples will be spiked after final leachate generation at the time of preparative digestion or extraction. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.8 Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.9 Consult the individual analysis SOPs for corrective action for blanks, LCSs, and MS/MSDs

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must also be documented in an NCM, with a cause and corrective action described.

NOTE: The worksheets referred to in this SOP can be found in G:\QA\Edit\FORMS\Organic Prep Forms\TCLP Worksheets Rev 9.

NOTE: See Attachment 12 for instructions on how to create batches in the LIMS system "TALS".

10.1 WORKSHEET 1, SECTION A, SAMPLE DESCRIPTION – Enter data on Worksheet 1.

10.1.1 Preliminary TCLP evaluations (percent solids, particle size, selection of leach fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of sample. This aliquot may also undergo the actual TCLP or SPLP extraction for non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

10.1.2 Record the number of phases observed in the sample. It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

10.1.3 If the sample has multiple phases and is received in more than one bottle, then the contents of each bottle should be combined in a single larger container prior to processing the sample further. However, the aliquot for volatile analysis should not be combined because that would expose the sample to headspace.

10.1.4 *LINE A.1* - Record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), describe the different phases in an NCM.

10.1.5 *LINE A.2* - Record the number of liquid phases observed in the sample according to apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case, a small drop of the liquid can be added to a small amount of water to test the relative density.

NOTE: If the sample contains an oil layer, see Section 4.1 for guidance.

10.1.6 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 10.3 (Leach Fluid Determination) for semi-volatile and metals analysis and proceed to Section 10.7 (ZHE Leaching Procedure) for volatile analysis. If only one jar was received, the ZHE procedure (Section 10.7) should be completed before proceeding to Section 10.3 for semi-volatile and metals analysis.

10.2 WORKSHEET 1, SECTION B – PERCENT SOLID PHASE

10.2.1 Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 10.7 (ZHE Extraction Procedure). Otherwise continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

10.2.2 Determine Type of Filtration Apparatus Needed –

- If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a liquid containing highly granular solids, either vacuum filtration or pressure filtration may be used
- If the sample is viscous (sludge or has high solids content), use pressure filtration.
- If the sample is oily, a glass vacuum flask and Büchner funnel can be used to filter the sample.

10.2.3 *LINE B.1 - Weight of Filter.* Measure and record this value before loading the filter into the filter holder. Assemble the filtration apparatus. Use care when handling the 0.6 to 0.8 μm filter so as not to bend the filter or to contaminate it with trace amounts of oil from your hands.

10.2.4 *LINE B.3.b – Tare Weight of filtration collection bottle.* Select an appropriate container to collect the filtrate into. Record the weight of the empty container as the tare weight of the filtrate. A plastic bottle can be used if only metals analysis is requested, but a glass container should be used if any organic analyses are requested.

10.2.5 *LINES B.2.a, B.2.b, and B.2.c - Weight of Subsample for Percent Solids Determination*.

10.2.5.1 Weigh the full sample container and document this as the gross weight (Line B.2.a). Whenever possible, the entire contents of the sample container should be used in the percent solids determination.

If the entire contents of the sample container are used, then transfer the entire contents to the filtration apparatus. It might be more conducive to filtering if any liquid portion is poured into the filtration apparatus first as not to pre-maturely clog the filter. If necessary, centrifugation can be used as well.

If there is limited sample volume, and the entire contents of the sample container cannot be used in the percent solids determination, then care must be taken to create a representative sub sample by creating a well-mixed slurry before taking the sub-sample.

10.2.5.2 Weigh the empty sample container with any residual sample and document this as the tare weight (Line B.2.b). The worksheet will then calculate the net weight of the sample used for the percent solids determination in Line B.2.c. If net weight is less than 100 g, an NCM should be written as the percent solids determination should be performed on an aliquot of at least 100 g.

10.2.6 Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no additional liquid has passed through the filter during a 2-minute period.

10.2.7 Increase the pressure in 10-psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2-minute period. This may require many hours to complete. The sample should not be filtered for more than 24 hours to avoid evaporation of the filtrate and thus miscalculation of the percent wet solids. If the sample filtration is not complete in 24 hours, then the client should be contacted.

NOTE: Some samples will contain liquid material that does not filter. Do not attempt to filter the sample again by exchanging filters. Viscous liquids or solids that do not pass through the filter are classified as a solid.

10.2.8 *LINE B.3.a – Gross Weight of Filtrate.* Remove the filtrate collection bottle, weigh and record the gross weight.

10.2.9 *LINE B.3.c – Net Weight of Filtrate.* The worksheet will calculate the net weight of the filtrate.

10.2.10 *LINE B.4 – Total Weight of Wet Solids.* The worksheet will calculate the total weight of wet solids by subtracting the net weight of the filtrate (Line B.3.c) from the net weight of the subsample (Line B.2.c)

10.2.11 *LINE B.5 – Weight Percent of Wet Solids*. The worksheet will calculate the percentage of wet solids in the sample based on weight by dividing the Total Weight of Wet Solids (Line B.4) by the Net Weight of the Subsample (Line B.2.c) and multiplying by 100.

10.2.12 *LINE B.3.d – Density of Filtrate.* If the percent solids determination result is greater than 0.5%, then determine the density of the aqueous phase of the filtrate using a calibrated pipette to measure the mass of 1 mL.

10.2.13 *LINE B.7* - The worksheet will then calculate the volume of the aqueous phase of the filtrate.

10.2.14 *LINE B.8* - If the filtrate is multi-phasic, pour the filtrate into a graduated cylinder. Measure and record the volume of the non-aqueous organic phase. If more than one organic phase is observed, enter "See Below" and provide a description at the bottom of Worksheet 1 and record this in a NCM.

10.2.15 Retain the filtrate for use in Section 10.3.3. If the sample is logged for metals analysis only, the filtrate can be stored in a plastic container at room temperature. If the sample is logged for any organic analyses, then the filtrate must be stored refrigerated in a glass container. If the sample is logged for analysis of VOCs and a separate container was not received, then a small portion of this filtrate must be stored refrigerated in a VOA vial with no headspace and an NCM written.

10.2.16 If the Weight Percent of Wet Solids in Line B.5 is greater than 5.0%, and semi-volatile and metals analyses are required, proceed to section 10.3. If the Weight Percent of Wet Solids in Line B.5 is greater than 5.0% and volatile analysis is required, proceed to Section 10.7.3.

10.2.17 If the Weight Percent of Wet Solids in Line B.5 is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate. If the sample is logged for method 8260B, refer to Section 10.7.1 (ZHE leaching of 100% Liquid Samples) to generate leachate and blanks for volatile analysis. If the sample is logged for semi-volatiles and metals analysis, generate a leach blank by passing reagent water through a clean filtration apparatus similar to the apparatus used in the percent solids determination of the sample. Deliver the leachates and the associated blank to the appropriate departments along with all completed documentation.

10.2.18 If the Weight Percent of Wet Solids in Line B.5 is greater than or equal to 0.5% but less than 5.0% and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 10.2.19 to complete the percent solids measurement on a dry-weight basis. If it is apparent to the analyst that the sample contains a significant amount of solids (>0.5%), the analyst can proceed to Section 10.2.19 to complete the percent solids measurement on a dry-weight basis to confirm this, or can proceed to Section 10.3 (Particle Size Reduction for Fluid Determination) for semi-volatile and metals analysis and Section 10.7.3 (ZHE Leaching of Samples Less than 100%, but greater than 0.5% Solids)

NOTE: If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter but instead proceed to Section 10.3 (Particle Size Reduction for Fluid Selection). Document in an NCM that the percent wet solids result is most likely biased high due to oily material trapped on the filter and that percent dry solids could not be performed.

10.2.19 *LINE B.6 – Weight Percent of Dry Solids*

NOTE: These steps are required only if it is noticed that a small amount of the filtrate is entrained in the wetting of the filter and the percent wet solids in Line B.5 is ≥ 0.5% and < 5.0 %.

- Remove the filter with the wet solids from the filtration apparatus. Take care to remove the entire filter. Often the filter will adhere to the apparatus.

- Dry the filter and solid phase at 100 ± 20 ° C. Record the observed temperature of the oven and the thermometer correction factor in Lines 6.d on Worksheet 1. Allow the filter to dry in the oven for at least 10 minutes.

- Remove the filter from the oven and allow to cool.

- Weigh and record the gross dry weight (Line B.6.a). The Worksheet will calculate the Weight Percent of Dry Solids in Line B.6.c using the equation in Section 11.5. If the Weight Percent of Dry Solids is less than 0.5%, then follow the guidelines in Section 10.2.17 for when percent wet solids is less than 0.5%. If the Weight Percent of Dry Solids is greater than 0.5%, repeat the drying step.

- Weigh and record the second gross dry weight (Line B.6.b). If the two weighings do not agree within 1%, perform additional drying and weighing until successive weights agree within 1%. Record the last two successive weights as Weight 1 and Weight 2 on Lines B.6.1 and B.6.2

- If the Weight Percent of Dry Solids is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 10.3 (Particle Size Reduction for Fluid Selection) using a fresh wet portion of sample.

- If the Weight Percent of Dry Solids result is ≥ 0.5% and the sample will be extracted for volatile constituents, proceed to Section 10.7.3 (ZHE Extraction Procedure).

- If the Weight Percent of Dry Solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Follow the guidelines in Section 10.2.17 for when percent wet solids is less than 0.5%.

10.3 WORKSHEET 2, SECTION C and D – LEACH FLUID DETERMINATION

If the solid content is greater than or equal to 0.5% and if the sample is being analyzed for metals or non-volatile organic compounds, the type of leaching solution must be determined.

The sub-sample used for fluid selection is taken from the non-filterable solid portion of the sample, but the aliquot must not have been subjected to the oven drying in Section 10.2.19.

Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

For SPLP, refer to Section 7.9 for fluid selection. The client must specify matrix type. Check special instructions, LIMS method, or the PM to determine if the sample is from east or west of the Mississippi River. Document on Line D.3, D.4, or D.5 the fluid type used and then proceed to Section 10.3.3 (Fluid Compatibility)

NOTE: All SPLP waste waters/wastes **must** be SPLP fluid 1, no matter if they are east or west of the Mississippi. . If a sample is a wastewater/wastes, the PM will be logged as 1312E, which will indicate to the analyst to use SPLP Fluid #1.

10.3.1 *LINE C.1 – Particle Size Reduction for Fluid Determination*

Reference WI-DV-0058. The sub-sample used for fluid determination must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used in the actual leach). The method requires smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full leaching. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

Surface Area Exclusion – Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If the surface area is less than or equal to 3.1 cm^2 per gram, enter “No” on Line C.1 and prepare an NCM documenting the surface area per gram of sample.

If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size. Enter “Yes” in Line C.1. Document in an NCM how the particle size reduction was performed.

Consult your supervisor and project manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick)

10.3.2 Determination of Appropriate Leach Fluid

10.3.2.1 Calibrate the pH meter with fresh aliquots of buffer solution in accordance with the manual. See Section 6.9.

10.3.2.2 *LINE C.2* – Calibrate a balance per DV-QA-0014 and record the balance ID.

10.3.2.3 *LINE C.3* - Weigh out a 5.0g ± 0.1g sub sample (less than 1mm particle size) of the solid phase into a 150mL beaker. This sub sample cannot have been subjected to the oven drying in Section 10.2.19

10.3.2.4 *LINE C.4* – Using a Class A graduated cylinder, or a bottle top pump calibrated per DV-QA-0008, add 96.5mL of reagent water to the sub

sample. Document on Line C.4.a the ID of the pipette or graduated cylinder.

10.3.2.5 *LINE C.5.a, Line C.5.b, –* Cover the 150mL beaker and stir vigorously for 5 minutes on a stir plate. Document the time the stirring started on Line C.5.a. Document the time the stirring stopped on Line C.5.b.

10.3.2.6 *Line C.5.c* - Measure and record the sample pH. Do not stir the sample during pH measurement. If the pH is less than or equal to 5.0, use TCLP Fluid #1. Place an "X" in LINE D.1 and proceed to Section 10.3.3 (Fluid Compatibility) If the sample matrix is especially oily, use narrow-range pH paper to measure the pH instead of the pH meter. This is done to protect the pH probe. Document the use of the narrow-range pH paper in an NCM.

10.3.2.7 *LINE C.6* - If the pH is greater than 5.0, add 3.5mL of 1 N HCl, using a calibrated pipette. Put a "X" on line C.6 and record the HCl Lot# and the Pipette ID in Lines C.6.a and C.6.b. Mix the sample briefly to create slurry.

NOTE: This can be done with either a glass rod or mixing motion of the hand – do not use the stir bar as a means of mixing.

10.3.2.8 *LINE C.7.a thru C.7.f* – Cover the sample with a watch glass and place the sample in a heated water bath and heat to 50ºC to 55ºC for 10 minutes. Do not stir the sample during this time. The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured. The temperature readings will be taken using a calibrated thermometer that is placed in a random sample in the water bath. Record the observed temperature and the thermometer correction factor.

10.3.2.9 Do not remove the cover and allow the sample to cool to room temperature. Measure the pH of the sample within 15 minutes of coming to room temperature. During this pH measurement, do not stir the sample. Document the reading on line 7.f. If the sample matrix is especially oily, use narrow-range pH paper to measure the pH instead of the pH meter. This is done to protect the pH probe. Document the use of the narrow-range pH paper in an NCM.

10.3.2.10 *LINE D.1 and LINE D.2* – If the pH is less than or equal to 5.0, use Fluid #1 If the pH is greater than 5.0, use Fluid #2.

10.3.3 Determination of Filtrate/Leach Fluid Compatibility

Skip this Section if the sample did not yield an initial filtrate from Section 10.2

10.3.3.1 Place 5mL of the appropriate leaching fluid (determined in the previous step) into a 25mL vial. Add 5mL of the initial filtrate, cap and shake.

10.3.3.2 If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation. Enter an "X" in LINE D.6 If the phases are not miscible, enter "NO". The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined. See Section 11.12.

10.4 WORKSHEET 3, SECTION E– DETERMINATION OF SAMPLE SIZE FOR BOTTLE LEACH PROCEDURE

10.4.1 The aliquot used in the Percent Solids determination described in Section 10.2 may be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or the preliminary aliquot was not oven dried proceed directly to Section 10.4.2 (Particle Size Reduction for Leaching). If the aliquot from the Preliminary Evaluation was oven dried then, using a fresh aliquot of sample, filter the sample to obtain wet solids and filtrate as described in Sections 10.2.2 through Section 10.2.15. The percent wet solids calculations may need to be repeated in order to correct for sub-sampling error. Then using this new aliquot of wet solids, proceed to Section 10.4.2

10.4.2 *LINE E.1 – Particle Size Reduction for Leaching*

Reference WI-DV-0058. Evaluate the solid portion of the sample for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the sample for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e, capable of passing though a 9.5 mm, 0.375 inch standard sieve). Size reduction is not required if the sample surface area to weight ratio is greater than or equal to 3.1 cm^2 per gram. (See Section 10.3.1)

Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick). Scissors or tin snips may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks or other solids amenable to grinding can be reduced using a jaw crusher. Document in an NCM how unusual samples were handled. Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

10.4.3 *LINE E.2* -Calibration check a top-loading balance per DV-QA-0014. Document the Balance ID on Line E.2.

10.4.4 Determine the total volume of leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to Table 2 below. Note that the volumes listed in Table 2 are the minimum volume required for one extraction and analysis. If possible, extra volume should be prepared for re-extractions and re-analysis. Additional volume for MS/MSD analysis should be provided for at least one sample per leach batch for every requested analysis. When an MS/MSD pair

is not available, an LCS and LCSD are required. The samples will be leached at a 20X dilution (i.e. 100g of solids will generate 2000mL of leachate).

Table 2. Minimum Required Leachate Volume

Analysis	Required Leachate Volume for TCLP (mL)	Required Leachate Volume for SPLP (mL)
Volatiles	20 (3 x 20mL vials are supplied to provide volume for screening and re-analysis)	40 (3 x 40mL vials are supplied to provide volume for screening and re-analysis)
Semivolatiles	200	1000
Pesticides	100	1000
Herbicides	100	1000
Metals	100	100

10.4.5 *LINE E.3* - Weigh at least 100g of the solid portion of the sample into an appropriate leach vessel. See Section 6.1 for appropriate leach vessels. Document the weight of the sample to the nearest 0.01g on Line E.3. A minimum sample size of 100g is required. If there is insufficient sample, a NCM is needed. If full suite TCLP is requested, use 150g to generate sufficient leachate.

10.5 WORKSHEET 3, SECTION F– DETERMINATION OF AMOUNT OF LEACH FLUID FOR BOTTLE LEACH PROCEDURE

10.5.1 *LINES F.1 through F.4 – Lot number of Leach fluid*. The worksheet will indicate the correct leach fluid to use as determined in Lines D.1 through D.5. Document the Lot number of the leach fluid used in Lines F.1 through F.4.

10.5.2 *LINE F.5 – pH of Leach Fluid*. Record the pH of the Leach fluid. Check to make sure the pH of the fluid is still within the specifications in Section 7.7 and Section 7.9. If the pH of the buffered TCLP fluids is not within specifications, ensure the pH meter is properly calibrated and re-check. If the re-check is also not within specifications, discard the fluid and make fresh fluid. If the pH of the un-buffered SPLP fluid is not within specifications, either discard the fluid or adjust the pH by adding more acid or more water. See Section 7.9.

10.5.3 *LINE F.6 – Volume of Leach Fluid.* The worksheet will calculate the volume of leach fluid to add to each sample based on the weight of the sample in Line E.3 using the formula in Section 11.7. Prepare method blanks by filling similar leach vessels with the same leach fluid used for the samples.

10.6 WORKSHEET 3, SECTION G– RECORD OF BOTTLE LEACH

10.6.1 LINE G.1 – Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and turn on the rotator. The rotator speed must be checked under load every day of use. Count the number of rotations in 15 seconds and multiply by 4 to obtain the rotations per minute (RPM). If the RPM is between 28 and 32, then mark line G.1 “YES”. If the RPM is not between 28 and 32, then tag out the rotator until it can be repaired and move the samples to a rotator that does rotate at the correct speed.

10.6.2 *LINE G.2 and G.3* – Rotate the sample end-over-end for 16-20 hours. Record the leach start date and time on Line G.2. As agitation continues, pressure may build up within the bottle for some types of samples. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented fume hood to relieve any built-up pressure. Due to the higher acidity of TCLP Leach Fluid #2, it is more common for these samples to generate excess pressure. Record the leach stop date and time on Line G.3.

10.6.3 *LINE G.4 – Temperature of Leach.* The temperature of the room should be 23 ± 2°C. A data-logging device (LogTag) records the room temperature. After the leach has been stopped, record the thermometer correction factor and the observed minimum temperature and in Line G.4.a and the observed maximum temperature in Line G.4.b. The worksheet will then calculate the actual minimum temp in Line 4.c and the actual maximum temp in Line 4.d.

Download LogTag data according to WI-DV-0067. Click on “Data” tab and print data to PDF. File should be stored with the EXCEL TCLP worksheet files. The LogTag PDF file will then be attached to each corresponding batch in the LIMS system “TALS” per instructions in Attachment 12, Line 13.

If the temperature of the room was not 23 ± 2°C, the solid fractions of the samples must be re-leached. If there is no volume to re-leach, the client must be contacted. The client must decide if the procedure should be canceled or if the laboratory should continue with a NCM.

10.6.4 Filter the leachate using vacuum or pressure filtration. This should be done the same day the 16-20 hour leaching was finished. For final filtration of the leachate, the glass fiber filter may be changed, if necessary to facilitate filtration. The entire leachate need not be filtered; however sufficient volume should be filtered to support the required analyses plus extra volume in case of re-extraction, re-digestion and MS/MSD. When an MS/MSD pair is not available, an LCS and LCSD are required. If needed, the leachate can be centrifuged to help facilitate filtration.

10.6.5 *LINE G.5 – pH of Leachate.* Record the pH of the leachate. If the leachate is especially oily, do not use the pH meter to measure the pH as this may damage the probe. Use narrow-range pH paper instead and write an NCM that the pH was measured using narrow-range pH paper instead of a pH meter.

10.6.6 If the sample contained no initial filtrate, (i.e the sample was 100% solids) the filtered leachate is defined as the final TCLP leachate. Proceed to Section 10.6.10

10.6.7 *LINE G.6 Volume of Leachate.* If the sample had an initial filtrate from Section 10.2, then measure the volume of leachate recovered so the leachate and the filtrate can be combined in the correct ratio. If the leachate contains an oil phase, it must be separated and its volume recorded on Line G.6.a. The oil and the filtered leachate must be analyzed separately. If requested, the results can be mathematically re-combined. See Section 11.11 and Section 11.12.

10.6.8 *LINE G.7 – Volume of initial filtrate for recombination*. The worksheet will use the equation in Section 11.8 to calculate how much of the initial filtrate should be combined with the volume of leachate in Line G.6. Consult Line D.6 to determine if the initial filtrate is compatible to the leachate. If they are compatible, they are to be combined in the correct proportions and mixed well. The combined solution is defined as the TCLP leachate. If the initial filtrate and the leachate are not compatible, they are to be prepared and analyzed separately and the results mathematically combined. See Section 11.11 and Section 11.12. The leachate and the filtrate will have to be logged as separate samples in LIMS.

10.6.9 *LINE G.8 – Volume of combined initial filtrate and leachate.* The worksheet will calculate the volume of the combined filtrate and leachate using the equation in Section 11.9.

10.6.10 Leachates for organic analyses should be stored in glass containers at 4°C ± 2°C. Refer to Table 2 to determine how much leachate is needed.

10.6.11 Leachates for metals analysis should be stored in poly bottles. A 250mL aliquot should be submitted for metals analysis.

10.6.12 Prepare a MS/MSD sub-sample for metals testing following the steps below. When an MS/MSD pair is not available, an LCS and LCSD are required.

10.6.12.1 Measure out 50mL of leachate into verified digestion tube. Add 0.5mL of the TCLP Spike described in Section 7.10.1. Add 0.5mL of the Prep Spike 2B described in Section 7.10.2. Add 0.5mL of the Prep Spike 3A described in Section 7.10.3. This 50mL aliquot will be split equally for the MS and the MSD after spiking.

10.6.12.2 If mercury is requested, measure out an additional 75mL aliquot. Pour two 30mL aliquots from the 75mL aliquot. Add 1.5mL of the mercury spike described in Section 7.10.4 to each of the 30mL aliquots. These two 30mL aliquots will now serve as the MS and the MSD.

10.6.13 Immediately preserve all leachates for metals by adding 1mL of nitric acid at a time until pH of 2 has been achieved. If after 5mL of acid has been added and the pH is still not 2, do not add more acid, but document final pH in an NCM. If a precipitate starts to form, immediately stop adding acid and document in an NCM.

10.7 WORKSHEET 4, ZHE PROCEDURE

Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g. metals, pesticides, etc.).

Due to the shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the

final leachate has been collected. Target compounds will volatilize very rapidly, therefore do not allow the waste, the initial liquid phase, or the leachate to be exposed to the atmosphere any longer than necessary. The sample should be kept cold and not allowed to come to room temperature until it is loaded into the ZHE and all headspace has been purged. Keep the sample in cold storage or in an ice bath.

The ZHE cannot accurately determine percent solids <5%. Go to Section 10.2 if it is apparent that the sample is less than 5% solids. If the sample is apparently greater than 5% solids, but less than 100% solids, go to Section 10.7.3. If the sample is 100% solids, go to Section 10.7.3. If the sample is 100% liquid, proceed to Section 10.7.1

10.7.1 ZHE Leaching of 100% Liquid Samples. – This procedure is to be used for samples determined to be 100% liquid per Section 10.2

10.7.1.1 Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with reagent water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the sample. At least 80mL of sample should be used. If the piston is 2cm below the top of the cylinder, this will be enough volume for 80mL. By seating the piston as high as possible, you will limit the headspace in the ZHE that will need to be purged later and the potential loss of volatiles.

10.7.1.2 Assemble the top flange and run water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing and will prevent the valve from leaking and reduce the frequency of o-ring replacement.

10.7.1.3 *LINE H.2.* - Place the sample in the ZHE body. Place the ZHE body on the ZHE base. Place the top flange on top of the ZHE body and secure tightly. Record the ZHE used on Line H.2

10.7.1.4 With the inlet/outlet valve closed, pressurize the ZHE until you hear the piston move upwards.

10.7.1.5 *LINE I.6* - Slowly open the inlet/outlet valve to release any headspace. Once liquid appears through the inlet/outlet valve, close the valve and attach a clean gas-tight syringe. Slowly open the valve and collect the filtrate. This filtrate is the final leachate. After all leachate has been collected, remove the syringe from the ZHE and document the filtration completion date and time on Line I.6

10.7.1.6 Transfer the leachate from the syringe to 20mL voa vials for TCLP leachates and 40mL voa vials for SPLP leachates. Care should be taken not to leave any headspace in the vials. The entire leachate need not be transferred, but three vials should be filled to allow for re-analysis and screening.

10.7.1.7 Generate a leach blank using reagent water in the same manner as above. Document in the ZHE spreadsheet which ZHEs were used for samples and which ZHEs were used for method blanks. A ZHE cannot be used for a sample if it has not been used as a method blank in the past 20 uses.

10.7.2 ZHE Leaching of 100% Solid Samples

10.7.2.1 Consult Worksheet 1 and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor. If the preliminary evaluations indicate the need for particle size reduction, crush, cut, or grind the sample so that all particles are less than 1 cm in size as measured with a ruler. (Do not sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If particle size reduction was necessary, document this on Worksheet 4 as an observation and write an NCM.

To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 ºC) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

10.7.2.2 Assemble the top flange and run T1 fluid through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing. It will also prevent the valve from leaking and reduce the frequency of o-ring replacement. Place the assembled top flange on top of the body, secure tightly. See Attachment 6.

10.7.2.3 Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the required sample. At least 25 grams of sample will be needed. Normally the piston should be seated approximately 2cm below the top of the cylinder, but if the sample is bulky, the piston might have to be seated lower. By seating the piston as high as possible, you will limit the headspace in the ZHE that will need to be purged later and the potential loss of volatiles.

10.7.2.4 *LINE H.1* – Calibration check the balance per DV-QA-0014 and record the balance ID.

10.7.2.5 *LINE H.2* – Record the ID of the ZHE

NOTE: To reduce the time the sample is exposed to the air, the steps described in Section 10.7.2.6 through 10.7.2.9 should be done in quick sequence, working with one sample at a time.

10.7.2.6 *LINE H.3* – Place the ZHE cylinder on the balance and tare. Transfer 25 to 25.5g of the sample into the ZHE cylinder. Record the mass on Line H.3. If less than 25 g is used an NCM should be written to document the deviation from the procedure.

10.7.2.7 Place the ZHE body on the ZHE base and secure the top flange.

10.7.2.8 Close the liquid inlet/outlet valve on the top flange. Pressurize the ZHE until you hear the seals set.

10.7.2.9 Slowly open the liquid inlet/outlet valve to release all headspace. Then depressurize the ZHE using the pressure release valve.

10.7.2.10 *LINE H.7* – The worksheet will calculate the required volume of leach fluid to add to the ZHE in Line H.7, which is 20 times the mass of the wet solids in the ZHE body (e.g. If 25 g of wet solids were used, then 500 mL of fluid would be required). See the formula in Section 11.7.

Load a clean ZHE that has been specifically designated for blank fluid with the correct volume of TCLP Fluid #1 or SPLP Fluid #3 depending on the analysis requested. Measure the amount of fluid from Line H.7 using a clean 500 mL or 1000 mL graduated cylinder, and pour it into the ZHE with the piston moved all the way to the bottom.

Place the assembled top flange (screens and filter paper aren't necessary) on top of the body holding the blank fluid and secure tightly. Attach the connective tubing to the inlet/outlet valve and with pressure flowing, slowly open the valve, while holding the tubing straight up until all the air has been removed from the line. At the first sign of liquid, immediately close the inlet/outlet valve to prevent the loss of any blank fluid.

Attach the other end of the tubing to the sample ZHE, making sure the pressure relief valve on the bottom of the sample ZHE is left open. Slowly open the inlet/outlet valves on both ZHEs and turn on the pressure, which is attached to the ZHE containing the blank fluid, to allow the fluid to flow into the ZHE containing the sample. When it is determined that all the fluid has been transferred, close the inlet/outlet valve on both ZHEs and remove the transfer line. Observe the valve opening for any leaks. If it is leaking, the valve o-rings will need to be replaced.

Pressurize the sample ZHE and slowly open the liquid inlet/outlet valve to release any air that was introduced into the ZHE with the fluid. Close the valve as soon as liquid appears.

10.7.2.11 *LINES H.7a, H.7.b, and H.7.c* – Record the lot number and the pH of the fluid used.

10.7.2.12 *LINE I.1.a and LINE I.1.b* – Making sure the pressure relief valve at the bottom is closed, pressurize the ZHE to at least 15 psi. Record the on I.1.a. Let the ZHE sit for at least 15 minutes. Check to make sure the gauge indicates no loss of pressure. Record this check on Line I.1.b. Check the inlet/outlet valve for signs of leakage. If the ZHE shows signs of leakage or the pressure gauge indicates leakage, then the ZHE will be removed from service and repaired. Start the procedure over using either a new ZHE or the repaired ZHE and a fresh aliquot of sample. All repairs and maintenance performed on ZHEs are documented in the ZHE log book. If the ZHE has held pressure and there is no sign of leakage from the inlet/outlet valve then proceed on.

10.7.2.13 If the pressure gauge indicates a leak, place the ZHE in a bucket of water and watch for air bubbles. If bubbles are coming from the o-ring at the bottom of the cylinder, clean or replace the o-ring and wipe any contamination from the o-ring grooves. If bubbles are coming from the base pressure relief valve, try seating the valve with your finger or mark the base as having a leaky valve and set aside for repair.

10.7.2.14 Generate a leach blank by assembling and loading a ZHE with the same leach fluid used for the samples. Record in the ZHE logbook which ZHEs were used for the leaching of samples and which ZHEs were used for the leaching of blanks. A ZHE cannot be used for the leaching of a sample if it has not been used for the leaching of a blank in the past 20 leaches.

10.7.2.15 *LINE I.2 through LINE I.4-* Secure the ZHE in a rotatary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. Record the start time and the end time on Lines I.3 and I.4. The rotator speed must be checked every day of use under load. Count the number of rotations in 15 seconds and multiply by 4 to obtain the rotations per minute (RPM). If the RPM is between 28 and 32, then mark line I.2 “YES”. If the RPM is not between 28 and 32, then tag out the rotator until it can be repaired and move the samples to a rotator that does rotate at the correct speed.

10.7.2.16 *LINE I.5.a and LINE I.5.b* – A data-logging device (Log Tag) records the room temperature. The maximum and minimum temperature during the leach is recorded.

If the temperature of the room was not 23 ± 2°C, the solid fractions of the samples must be re-leached. If there is no volume to re-leach, the client must be contacted. The client must decide if the procedure should be canceled or if the laboratory should continue with a NCM.

Download LogTag data according to WI-DV-0067. Click on “Data” tab and print data to PDF. File should be stored with the EXCEL TCLP

worksheet files. The LogTag PDF file will then be attached to each corresponding batch in the LIMS system "TALS" per instructions in Attachment 12, Line 14.

10.7.2.17 *LINE I.6* - Remove the ZHE from the rotary agitator and check that the ZHE is still under pressure. Do this by quickly opening and closing the pressure release valve and listening for the release of gas. If the ZHE is not under pressure, then the procedure must be repeated using a fresh aliquot of sample and the ZHE should be taken out of service for maintenance and repair.

10.7.2.18 *LINE I.7* – Attach a clean gas-tight syringe to the inlet/outlet valve. The plunger of the syringe should be completely compressed before being attached to the ZHE. Slowly open the inlet/outlet valve and allow the leachate to enter the syringe. If necessary the ZHE can be pressurized to facilitate the collection of the leachate, but care should be taken not to cause effervescence. After enough leachate has been collected to fill three 20 mL voa vials (about 75 mLs), remove the syringe from the ZHE. If the sample was multiphasic and the filtrate and leachate are to be recombined prior to analysis, the amount of leachate recovered needs to be entered in Line I.7. This step should be performed the same day the 16 to 20 hour leach is finished.

10.7.2.19 *LINE I.7.a* - If the leachate is bi-phasic record the volume of the non-aqueous phase on Line I.7.a. Document in an NCM. The oil phase may need to be analyzed separately and results mathematically re-combined.

10.7.2.20 Transfer the leachate from the syringe to three 20 mL voa vials for TCLP leachates or three 40 mL voa vials for SPLP leachates. Care should be taken not to leave any headspace in the vials. The entire leachate need not be transferred.

10.7.2.21 Label all leachates and deliver the leachates and associated blank to the GC/MS Volatiles department along with all completed documentation. The leachates should be stored at 4 ± 2 °C.

10.7.3 ZHE Leaching of Samples Less than 100%, but greater then 0.5% Solids

10.7.3.1 Consult Worksheet 1 and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor. If the preliminary evaluations indicate the need for particle size reduction, crush, cut, or grind the sample so that all particles are less than 1 cm in size as measured with a ruler. (Do not sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If particle size reduction was necessary, document this on Worksheet 4 as an observation.

NOTE: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 ºC) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

10.7.3.2 Assemble the top flange and run water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing. It will prevent the valve from leaking and reduce the frequency of o-ring replacement. Place the assembled top flange on top of the body, secure tightly. See Attachment 6.

10.7.3.3 *LINE H.4.b* - Weigh 1 to 2 empty gas-tight syringes. Record their combined weight as the tare weight on Line H.4.b. More syringes may be needed if the sample contains a low percent solids value. See Line B.5.

10.7.3.4 Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the required sample. By seating the piston as high as possible, you will limit the headspace in the ZHE that will need to be purged later and the potential loss of volatiles.

10.7.3.5 *LINE H.1* – Calibration check the balance per DV-QA-0014 and record the balance ID.

10.7.3.6 *LINE H.2* – Record the ID of the ZHE

NOTE: To reduce the time the sample is exposed to the air, the steps described in Section 10.7.3.7 through Section 10.7.3.15 should be done in quick sequence, working with one sample at a time.

10.7.3.7 *LINE H.3* - Place the ZHE cylinder on the balance and tare. Use the equation in Section 11.10 to estimate how much sample to place into the ZHE in order to ensure 25g of wet solids is included in the aliquot. Transfer the sample into the ZHE cylinder. Record the mass on Line H.3.

10.7.3.8 Place the ZHE body on the ZHE base and secure the top flange.

10.7.3.9 Close the liquid inlet/outlet valve. Pressurize the ZHE until you hear the seals set.

10.7.3.10 Slowly open the liquid inlet/outlet valve to release all headspace. Once liquid starts to come out of the valve, immediately close the valve and attach one of the tared syringes.

10.7.3.11 Open the valve again and collect the filtrate. Once the syringe is filled, close the valve and attach an additional tared syringe and repeat until no more filtrate is collected. Increase the pressure of the ZHE 10 psi at a time up to 50 psi until no more filtrate emerges from the ZHE after 2 minutes.

10.7.3.12 *LINE H.4.a* - Weigh the full syringes and record their combined weight as the gross weight.

10.7.3.13 *LINE H.4.c* – The worksheet will then calculate the net weight of the filtrate using the equation in Section 11.2

10.7.3.14 *LINE H.5* – Record the volume of the filtrate by reading the graduations on the syringe(s).

10.7.3.15 Transfer the filtrate into vials with no headspace. Label and store the filtrate refrigerated 4 ± 2°C.

10.7.3.16 *LINE H.6* – The worksheet will then calculate the total grams of wet solids remaining in the ZHE using the formula in Section 11.3. If less than 25g of wet solids remains in the ZHE, an NCM should be written to document the deviation from the procedure.

NOTE: The ZHE has a maximum capacity of 500mL. Therefore you cannot load more than 25g of solids into the ZHE or else you will not be able to add the appropriate volume of leach fluid.

10.7.3.17 *LINE H.8*- The worksheet will then calculate the percent wet solids using the formula in Section 11.4

10.7.3.18 Follow steps in Section 10.7.2.10 through 10.7.2.21

10.7.3.19 If the initial filtrate from Section 10.7.3.15 is miscible with the leachate (as determined in Section 10.3.3), the leachate and the initial filtrate are directly recombined in the correct proportions.

For samples containing greater than 5% wet solids, the percent wet solids value from the ZHE filtration process should be used to determine the volume of filtrate to re-combine with the leachate. Therefore use the value in Line I.8.b. This approach is required since the percent solids value determined using the pressure filter may differ from the percent solids value determined using the ZHE due to sample variability or differences in the filtration apparatus.

For samples containing less than 5% wet solids, the percent wet solids value from the pressure or vacuum filtration process should be used to determine the volume of the filtrate to re-combine with the leachate. Therefore use the value in Line I.8.a. This approach is required

because the ZHE is not appropriate to determine the percent solids of a sample if the percent solids are less than 5%.

Document the volume used in the comments section. For example, if the sample contained less than 5% wet solids and you are using the volume of initial filtrate calculated from Line I.8.a, Note "Sample "ABC" initial filtrate volume calculated from Line I.8.a"

10.7.3.20 If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately. If the individual phases are analyzed separately, the results can be mathematically recombined by using the recombination calculation in Section 11.12.

10.7.3.21 Label all leachates and deliver the leachates and associated blank to the GC/MS Volatiles department along with all completed documentation. The leachates should be stored at 4 ± 2 °C.

10.8 Maintenance

10.8.1 The pH probe should be replaced when it is noticed that the readings drift or are inconsistent. This should be documented in the pH logbook.

10.8.2 ZHE valve o-rings need to be replaced when worn. Scientific Instrument Service part numbers V010 and V012. This should be documented in the ZHE spreadsheet.

10.8.3 The ZHE inlet/outlet connector can become damaged and should be replaced with Millipore part number YT3009002. This should be documented in the ZHE spreadsheet.

10.8.4 The pressure gauges and pressure release valves on the ZHE base need to be replaced when worn or broken. The pressure release valves can be purchased from Millipore under part number XX6700024.

10.8.5 The quick connect on the ZHE base will need to be replaced when worn. Swaglok part number SS-QC4-B-2PM.

10.8.6 When working with especially oily samples, disposable ZHE screens are preferred to help prevent cross-contamination.

10.9 Troubleshooting

10.9.1 When leaching samples with TCLP Fluid #2, applying duct tape to the lids can prevent the samples from leaking.

10.9.2 It is advisable to monitor and the temperature of the tumble room throughout the day before samples are set to tumble so that the heaters and air conditioners can be adjusted to keep the temperature in range. If four or more rotators are running

at a time, this will generate heat in the room and the door might need to be propped open to keep the room in range. Normally heaters set at 75 °F and air conditioners set at 23°C keep the room in temperature range.

NOTE: The Air conditioners have filters, which are described in the users manual. These filters are akin to lint screens, as such the filters should be monitored, cleaned and replaced as deemed appropriate by the analysts to prevent overheating in the tumble room.

10.9.3 When working with a sample that appears to be 100% liquid, do not assume the sample is water miscible. Test the miscibility of the sample in water and methylene chloride.

10.9.4 When preparing the leach fluids, it is important to mix the fluids well. This is especially important when making large volumes of fluid.

10.9.5 When adjusting the pH of SPLP fluid, do not assume the pH of the reagent water is 7. Test the pH of the water and enter it into the adjustment spreadsheet. Also if only a very small amount of acid is needed to adjust the pH into range, the acid can be pre-diluted before adding it to the fluid to help in mixing and more accurate measurement.

11.0 Calculations and Data Reducation

11.1 Weight of Subsample (Line B.2.c)

(Net Weight, B.2.c) = (Gross Weight, B.2.a) – (Tare Weight, B.2.b)

11.2 Weight of Filtrate (Line B.3.c) or (Line H.4.c)

(Net Weight, B.3.c) = (Gross Weight, B.3.a) – (Tare Weight, B.3.b)

(Net Weight, H.4.c) = (Gross Weight, H.4.a) – (Tare Weight, H.4.b)

11.3 Total Weight of Wet Solids (Line B.4) or (Line H.6)

(Wet Solids, B.4) = (Weight of Subsample, B.2.c) – (Weight of Filtrate, B.3.c)

(Wet Solids, H.6) = (Weight of Subsample, H.3) – (Weight of Filtrate, H.4.c)

11.4 Weight Percent Wet Solids (Line B.5) or (Line H.8)

(% Wet Solids, B.5) = 100 x (Wet Solids, B.4) / (Weight of Subsample, B.2.c)

(% Wet Solids, H.8) = 100 x (Wet Solids, H.6) / (Weight of Subsample, H.3)

11.5 Weight Percent Dry Solids (Line B.6.c)

$$(\text{Weight percent dry solids, B.6.c}) = 100 \times \frac{(\text{Gross dry weight 2 or 1, B.6.b or B.6.a if B.6.a is blank}) - (\text{Weight of filter, B.1})}{(\text{Weight of subsample B.2.c})}$$

11.6 Volume of Aqueous Filtrate (Line B.7)

(Vol. of Filtrate B.7) = (Weight of Filtrate, B.3.c) / (Density of Filtrate, B.3.d)

11.7 Volume of Fluid for Bottle Leach (Line F.6) or ZHE Leach (H.7)

(Vol. Fluid, F.6) = (Weight of Wet Solids, E.3) x 20

(Vol. Fluid, H.7) = (Weight of Wet Solids, H.6) x 20

11.8 Volume of Initial Filtrate to recombine with Leachate (Line G.7), (Line I.7.a) or (Line I.7.b)

$$(\text{Vol. of Inital Filtrate for Recombination, G.7}) = \frac{(\text{Solids Leachated, E.3})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{Leachate Recovered, G.6})}{(\text{Fluid Added, F.6})} \times (\text{Initial Filtrat, B.7})$$

$$(\text{Vol. of Inital Filtrate for Recombination, I.7.a}) = \frac{(\text{Wet Solids in ZHE, H.6})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{Leachate Recovered, I.6})}{(\text{Fluid Added, H.7})} \times (\text{Initial Filtrat, B.7})$$

$$(\text{Vol. of Inital Filtrate for Recombination, I.7.b}) = \frac{(\text{Weight of Filtrate, H.4.c})}{(\text{Fluid Added, H.7})} \times (\text{Volume of Leachate Recovered, I.6})$$

11.9 Combined initial filtrate and leachate (Line G.8)

(Combined Filtrate & Leachate, G.8) = (Vol of Leachate, G.6)+(Vol of Filtrate, G.7)

11.10 Weight of Sample to Charge to ZHE

(Weight of Sample) = 100 X [20g/ (%wet solids, B.5)]

11.11 Reporting Conventions for Multi-phase Leachates:

11.11.1 If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

11.11.2 If both phases are “ND,” not detected, the recombined result is “ND,” and the reporting limit is calculated from the reporting limit for each phase.

11.11.3 If one phase is “ND” and the other phase has a positive result, use the reporting limit for the “ND” phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that “a positive result was detected below the calculated detection limit.”

11.11.4 Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

11.11.5 For limits and significant figures, consult the appropriate analytical methods

11.12 Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V_1 = total volume of the initial filtrate phase (L).

C_1 = analyte concentration in initial filtrate phase (mg/L).

V_2 = volume of the theoretical solid phase leachate (L).

C_2 = analyte concentration in solid phase leachate (mg/L).

12.0 Method Performance

12.1 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.1.1 Since a spiked aliquot is not appropriate for this procedure, initial and continuing demonstration of capability is documented by collecting data for a completed batch or a method specific test/quiz. An acceptable IDOC is determined by demonstrating that the method required batch QC was performed or the analyst "passed" the test/quiz.

12.1.2 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.1.3 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.1.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.2 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the

policies in Section 13, "Waste Management and Pollution Prevention", of the Enviornmental Health and Safety Manual, and DV-HS-001P, "Waste Management Plan."

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Solid waste (post extraction) – Excess Solid Samples - Waste Stream S See Note under Section 5.2.10

14.2.3 Aqueous waste (post extraction) - Aqueous Waste from TCLP - Waste Stream T

14.2.4 Buffer 4 - Aqueous Waste from TCLP - Waste Stream T

14.2.5 Buffers 7 and 10 - Aqueous Waste from TCLP - Waste Stream T

14.2.6 Methanol waste - Flammable Solvent - Waste Stream C

14.2.7 Methylene chloride waste - Waste Stream B

14.2.8 Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References

15.1 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

15.2 Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1992, SW-846 Proposed Update II.

15.3 Related Documents

15.3.1 Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

15.3.2 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

15.4 Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

16.0 Method Modifications

Item	Method	Modification
1	SW846 1311 & SW846 1312	Section 4.2.1 in both method 1311 and 1312 state "The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure." The laboratory pressurizes the ZHE and waits 15 minutes. This change in timing is supported by the laboratory's requirement to verify that pressure was maintained throughout the extraction process. If there is any loss of pressure at the end of the 16-20 hours the sample is discarded and the process repeated.

Item	Method	Modification
2	SW846 1311	Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). This SOP states that samples which have been subjected to the oven drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.
3	SW846 1311	Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, this SOP states that if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.
4	SW846 1311	Section 7.2.13 of the source method provides no guidance as to how to determine filtrate and leach fluid compatibility. Therefore, this SOP has incorporated a miscibility test into the Preliminary Determinations section.
5	SW846 1311	Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.
6	SW846 1311	Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The TestAmerica Laboratory Quality Manual is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.
7	SW846 1311	Section 6.4 of the source method states samples "may" be refrigerated unless refrigeration results in irreversible physical change to the waste. This procedures states the samples "should" be refrigerated unless refrigeration results in irreversible physical change to the waste.

17.0 Attachments

Attachment 1: Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)
Attachment 2: Metals TCLP Spike
Attachment 3: Metals ICP SPK 2A
Attachment 4: Metals ICP SPK 3A
Attachment 5: Metals Hg Daily Spk
Attachment 6: Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)
Attachment 7: Pressure Filtration Device
Attachment 8: TCLP Worksheet No. 1: Sample Description
Attachment 9: TCLP Worksheet No. 2: Selection of Leach Fluid
Attachment 10: TCLP/SPLP Worksheet No. 3: Bottle Leach Procedure

Attachment 11: TCLP Worksheet No. 4: ZHE Leach
Attachment 12: Instructions for Batching in LIMS
Attachment 13: Instruction Manual for pH meter.
Attachment 14: SPLP worksheet for correcting fluid pH.

18.0 Revision History

- Revision 12, dated 05 December 2018
  - Annual Review
  - Revised copyright information.
- Revision 11, dated 31 October 2017
  - Revised the note in section 7.9 and 10.3 to include that the PM will log all waste waters as 1312E.
  - Revised Sections 7.9 and 10.3 to reflect that the PMs will log all waste water samples under method 1321E.
  - Revised Section 10.3.2.5. as sample needs to be covered.
  - Revised Section 10.3.2.6 to indicate samples are not to be stirred while taking the pH
  - Revised Section 10.3.2.7 to notate the appropriate means of mixing the sample during pH evaluation.
  - Revised Section 10.3.2.9 to reflect sample should be brought to room temperature before final pH measurement is taken.
  - Revised Section 10.9.2 to update Air conditioner maintenance.
- Revision 10, dated 31, October 2016
  - Removed all references to AFCEE
  - Added current section 3.1 (all following sections re-numbered)
  - Added starting paragraph to section 6.0 to record all equipment IDs
  - Added note to section 6.0 to rotate glassware
  - Clarified RPM testing is to be performed “Before each use” in Section 6.6.
  - Added starting paragraph to section 7.0 regarding reagent grade chemicals
  - Grammatical changes in section 7.7.
  - Added “Note” to Sections 7.9 and 10.3 that all SPLP waste waters must be SPLP fluid 1.
  - Updated section 9.1 and subsections to reflect current practices and consistent verbiage
  - Revised sections 9.4, 10.7.1.7, 10.8.2, 10.8.3 to reflect that a spreadsheet is used for documentation in lieu of the logbook.
  - Added requirement for LCSD when no MS/MSD in sections 9.6, 10.4.45, 10.6.4 and 10.6.12.
  - Revised section 10.2.14 to include that a NCM should be generated to record the observation of a multi-phasic sample.
  - Revised section 10.3.2.8 to reflect the usage of a water bath and not hot plate.
  - Revised section 10.4.2 to state a “surface area to weight ratio” instead of surface area for size reduction.
  - Revised section 10.7.2.10 to reflect ZHEs must be pressurized before slowly opening the inlet and outlet valves.

- Revised section 10.8.2 to reflect O-ring part No. V011 is not being used. Instead V010 is being employed.
- Revised section 10.9.2 to reflect the temperature change from that of 30°C to 23°C.
- Added current section 12.1 regarding IDOCs
- Updated current section 12.2 to reflect current practices and consistent verbiage
- Added the current method modification #1 to section 16.

- Revision 9, dated 31, October 2015
  - Added detail to Section 4.5 and 10.7 stating that samples that are to be leached for volatiles should be kept cold until loaded into the ZHE and headspace is purged.
  - Revised Sections 10.6.1 and 10.7.2.15 to state that the rotation speed of the rotators must be checked under load each day of operation.
  - Revised Sections 10.6.4 and 10.7.2.18 to state that the leachate should be filtered the same day the 16-20 hour leach is completed.
  - Revised wording in Section 10.4.5 to match Section 10.4.2.
- Revision 8, dated 31, May 2015
  - Reformatted SOP.
  - Added comment to Section 1.4 to state that TestAmerica Denver cannot digest organic waste for metals analysis.
  - Section 4 was revised to discuss how organic waste samples will be treated. In the past organic liquids were assumed to be 100% liquid. The SOP was revised to state that organic liquids will be filtered to determine the percent solids.
  - Section 5.2.5 was revised to add details to the types of samples more likely to cause pressure to build up in the leach vessel.
  - Section 6 was revised to include the LogTag temperature recording device.
  - Section 9.2 was revised to clarify samples that are multi-phasic or solid per the procedure will be batched separately from samples that are liquid per the procedure.
  - Sections 3.4, 9.3 and 9.4 were revised to state that one leach blank will be prepared instead of “a minimum of one”.
  - Section 10.3 was revised to clarify that the steps to determine the leach fluid type are performed on the solid fraction of the sample.
  - Section 10.5.2 was revised to instruct the analyst on corrective action if the pH of the leach fluid is not within specifications.
  - Section 10.6.3 and Section 10.7.2.16 were revised to instruct the analyst on corrective action if the temperature of the room during the leach is outside of the control limits.
  - Sections 10.4.2 and 10.3.1 were revised to reference WI-DV-0058.
- Revision 7, dated 31, May 2014
  - Revised Section 6.9 to call for the pH meter to be calibrated at pH 2, pH 4, pH 7, and pH 10. Revised Attachment 13.
  - Added narrow range pH strips to Section 6 and revised Section 10 to allow their use as an alternative to the pH meter for oily samples.
  - Added the option to use disposable ZHE screens to Section 6.
  - The instructions in Section 7.7 on how to prepare TCLP fluids were revised to more closely match the source method.
  - Updated Section 7.10.2 for spike used to 2B

- Attachment 14 was added to aid in the preparation of SPLP fluids. Section 7.9 was revised to instruct the analysts to use the spreadsheets when adjusting the pH of the SPLP fluid.
- Section 9.1.2 was revised to state that this procedure meets all criteria for DoD QSM 5.0 unless otherwise stated.
- Section 9.3 was revised to clarify how samples are batched by matrix. A note was added to Section 9.6 to clarify the required frequency of MS/MSD samples.
- Revised Section 10.3.2 to remove the accuracy criteria for the bottle-top pump. Instead a reference was made to the SOP DV-QA-0008 which dictates the requirements for accuracy and precision of bottle-top pumps.
- Revised Section 10.3.2 to instruct the analyst to cover the sample with a watch glass and to not stir the sample during the 10 minute heating process or during the pH measurement after the 10 minute heating process. This was done to more closely match the source method.
- Revised Section 10.6.3.1 to instruct the analyst to attach the “Chart” data page from the temperature recording devise (LogTag) instead of the “Summary” data page.
- Added a comment to Section 10.6.10 to direct the analyst to Table 2 to ensure enough leachate is delivered.
- Updated Section 10.6.12 for Spike used to 2B
- Added Troubleshooting and Maintenance sections to Section 10.
- Updated Attachment 3 to include Sulfur and changed name to 2B
- Revised Attachment 6 to show the Cylinder o-ring and the wiper seal on the ZHE.
- Revised Attachment 7 to show the o-ring on the Pressure Filter.

- Revision 6, dated 31, May 2013
  - Section 5.2.10 was added and Section 14.2 were revised to address safety issues with waste handling.
  - Section 6.9 was revised and Attachment 13 was added because the lab acquired a new pH meter.
  - Section 8.9 was revised to show holding times are calculated from the beginning of the leaching procedure.
  - Section 9.1 and 10.0 were revised to reflect current practice.
  - Sections 10.2.19, 10.3.2, 10.6.3 and all Worksheets were revised to instruct the analysts to record the actual and observed temperatures and the thermometer correction factors.
  - Worksheet #3 was revised to indicate the correct leach fluid to use as determined in Lines D.1 through D.5. Section 10.5.1 was revised to reflect this change.
  - Section 10.7 was revised to instruct the analyst to be especially cautious to minimize the samples’ exposure to the atmosphere as much as possible to reduce the loss of volatiles.
  - Section 10.7.1.2, Section 10.7.2.3, and Section 10.7.3.4 were revised to instruct the analyst to seat the piston in the ZHE as high as possible when loading to limit the sample’s exposure to the atomosphere.
  - A note was added to Section 10.7.2.5 and Section 10.7.3.6 to instruct the analyst to work with one sample at a time to limit the samples’ exposure to the atmosphere.
  - Section 10.7.2.6 and Section 10.7.3.7 were revised to instruct the analyst to aliquot the sample directly into the ZHE instead of first aliquotting it into a weigh-boat and transferring it to the ZHE. This was done to limit the samples’ exposure

to the atmosphere and is now possible because the laboratory utilizes a 3kg mass in the daily balance calibration.

- Revision 5, dated 11, May 2012
  - This procedure was revised to require the use of a 25g aliquot in the zero headspace extractor.
  - This procedure was revised to instruct analyst to measure the mass of sample used in the ZHE procedure on a balance using a weigh boat instead of taring the ZHE body on the balance. This was done because the total mass of the ZHE body and sample exceeds all standard masses available for daily balance calibration checks.
  - This procedure was revised to more accurately reflect how the laboratory is preparing the TCLP Fluid #1 in large quantities.
  - This procedure was revised to remove the requirement that blank fluid be prepared using nitrogen-purged water when volatiles are requested. Water from the laboratory's ELGA purification systems were tested for volatiles, and no volatiles were detected, therefore water from the ELGA systems will be used for the preparation of all leach fluids.
  - Revised section 10.6.12 to properly reflect current practices regarding the MS/MSD spiking procedure for metals analysis.
- Revision 4, dated 25 May, 2011
  - Revised Section 4.1 to change instructions on how oily samples should be logged in LIMS.
  - Added detail to Section 6.1.1 on how to clean the ZHE apparatus.
  - Added a bottle-top pump to the equipment list in Section 6.
  - Revised Section 7.1 to state that the water from the ELGA purification system should be 18 to 18.2 Mohm-cm.
  - Added additional clarification to Section 7.7 and Section 7.9 on how the TCLP blank fluids are prepared with nitrogen purged water when volatile analyses are requested.
  - Section 9.2 was revised to remove the requirement that samples have to be batched separately if the bulk matrix is visibly different. Samples are only batched separately based on % solids determination or per client request.
  - Added more detail to Section 10.2.5 on aliquoting samples for percent wet solids determination.
  - Revised Section 10.3.2 to require the documentation of exactly how much water was added to the sample, exactly what time the sample was placed on the stir plate, exactly what time the sample was removed from the stir-plate, exactly what temperature the sample was on the hot plate and the exact times the sample was on the hot plate.
  - Added Section 10.6.3.1 and Section 10.7.2.16.1 to describe LogTag download and file retention procedure.
  - Revised Section 10.6.11 and 10.6.12 to make changes to how the leachates are spiked and preserved for metals analysis.
  - Revised Section 10.7.1.3 to state "Nitrogen-purged water" as opposed to "DI water".
  - Revised Section 10.7.2.7 to state "T1 fluid prepared with nitrogen-purged water" as opposed to "DI water".
  - Revised Section 10.7.2.18 to remove duplicated verbiage.

- Added Section 10.7.3.19.3 to instruct the need to document which calculated volume of initial filtrate was combined with the ZHE leachate.
- Updated Attachments 8 thru 11 to include the revisions made above.

Earlier revision histories have been archived and are available upon request.

Attachment 1.
Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

Attachment 2
Metals TCLP Spike

Component	Concentration (ug/mL)
Silver	100
Arsenic	300
Barium	1000
Cadmium	100
Chromium	500
Copper	200
Lead	500
Selenium	100
Zinc	200

Attachment 3.
Metals ICP SPK 2B

Component	Concentration (ug/mL)
Boron	100
Molybdenum	100
Antimony	50
Silicon	1000
Tin	200
Titanium	100
Zirconium	50
Sulfur	20

Attachment 4.
Metals ICP SPK 3A

Component	Concentration (ug/mL)
Silver	5
Aluminum	200
Arsenic	100
Barium	200
Beryllium	5
Calcium	5000
Cadmium	10
Cobalt	50
Chromium	20
Copper	25
Iron	100
Potassium	5000
Lithium	100
Magnesium	5000
Manganese	50
Sodium	5000
Nickel	50
Phosphorus	1000
Lead	50
Selenium	200
Strontium	100
Thorium	100
Thallium	200
Uranium	200
Vanadium	50
Zinc	50
Bismuth	200

Attachment 5.

Metals Hg Daily Spk

Component	Concentration (mg/L)
Mercury	0.1

Attachment 6.
Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

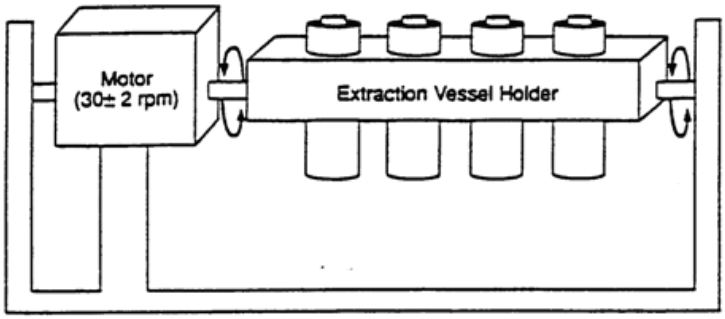
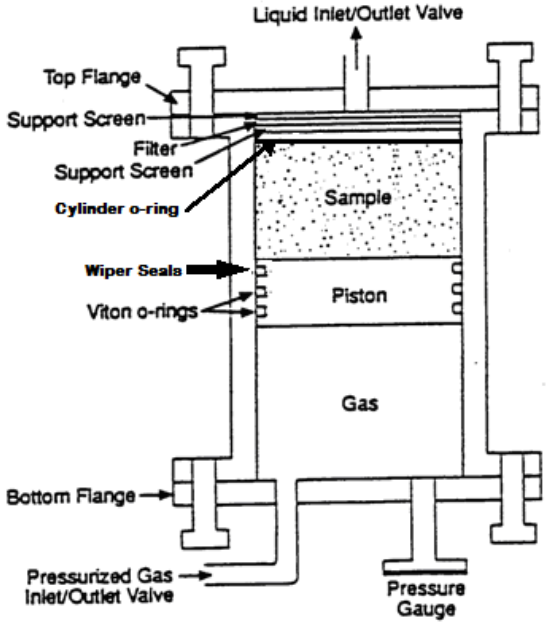
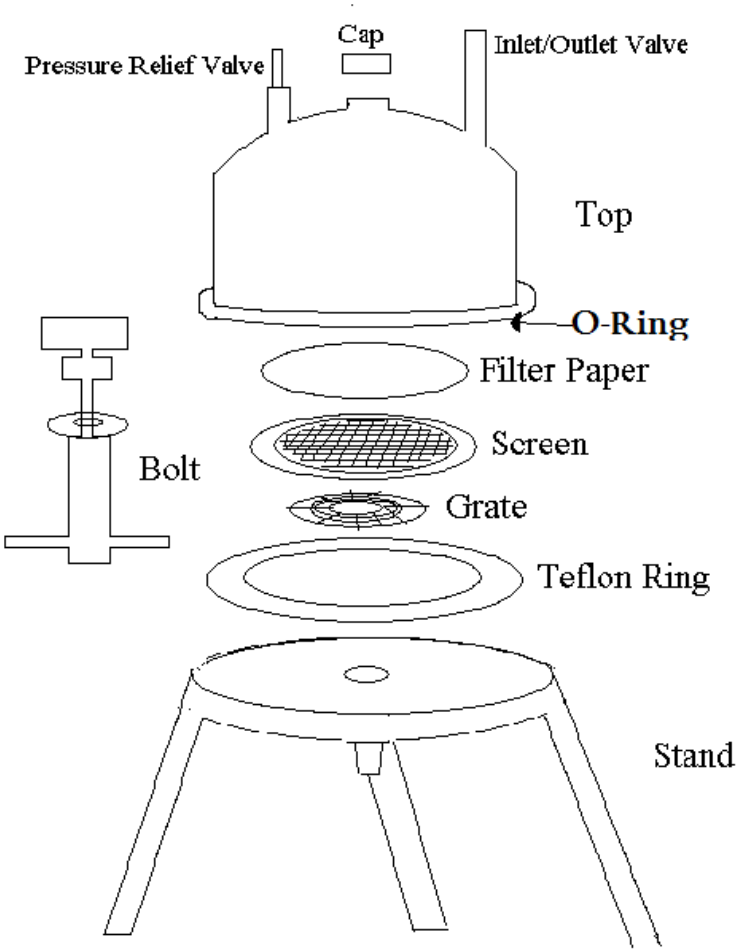


Figure 1. Rotary Agitation Apparatus



Attachment 7
Pressure Filtration Device



Attachment 8
Worksheet No. 1
TCLP

Analyst:	DV-IP-0012 TCLP/SPLP Worksheet No. 1 Sample Description									
Date:										
Login No.										
Sample No.										
A. Sample Description										
Number of phases										
1. Solid										
2. Liquid										
a. lighter than water										
b. water										
c. heavier than water										
B. Percent Solid Phase										
Balance ID										
1. Weight of filter (g)										
2. Weight of subsample										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3. Weight of filtrate										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
d. density of filtrate (g/mL)										
4. Total weight wet solids (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5. Weight percent solids (wet) (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
6. Weight percent solids (dry)										
a. gross dry weight 1 (g)										
b. gross dry weight 2 (g)										
c. percent dry solids (%)										
d. Oven Temp (observed) (°C)										
Thermometer Correction Factor										
Oven Temp Actual (°C)	0	0	0	0	0	0	0	0	0	0
7. Vol. of initial aqueous filtrate (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
8. Vol. of initial organic filtrate (mL)										

Comments:

$$(\text{Weight percent dry solids, B.6.c}) = 100 \times \frac{(\text{Gross dry weight 2 or 1, B.6.b or B.6.a if B.6.a is blank}) - (\text{Weight of filter, B.1})}{(\text{Weight of subsample B.2.c})}$$

(Net Weight of Subsample, B.2.c) = (gross weight, B.2.a) - (tare weight, B.2.b)

(Net Weight of Filtrate, B.3.c) = (gross weight, B.3.a) - (tare weight, B.3.b)

(Total weight wet solids, B.4) = (Weight subsample, B.2.c) - (Weight filtrate, B.3.c)

(Vol of initail filtrate, B.7) = (Weight of filtrate, B.3.c) / (Denisity of filtrate, B.3.d)

(Weight percent wet solids, B.5.) = 100 X (Total weight wet solids, B.4.) / (Weight of subsample, B.2.c.)

Attachment 9
TCLP Worksheet No. 2

Analyst:	0									
	DV-IP-0012 **TCLP Worksheet No. 2** **Selection of Leach Fluid**									
Login No.										
Sample No.										
C. Leach Fluid Determination- Does not apply to determination of volatile organic components or SPLP.										
1. Particle size reduction? (<1mm) Yes/No										
If yes, write NCM descibing how.										
2. Balance ID										
3. Sample weight, 5.0 +/- 0.1g										
4. Add 96.5 (+/- 2% or 94.57mL to 98.43mL)										
a. Pipette ID or Grad Cylinder ID										
5. Initial pH (after 5 min. mixing time)										
a. Start Time for Mixing										
b. Stop Time for Mixing										
c. pH reading after mixing										
6. If pH > 5.0, then add 3.5 mL 1N HCL & mark **"X"**										
a. HCL Lot# used										
b. Pipette ID										
7. Secondary pH (after 10min at 50C to 55C)										
a. Thermometer ID										
Termometer Correction Factor										
b. Start Time										
c. Start Temperature (Observed)										
Start Temperature (Actual)	0	0	0	0	0	0	0	0	0	0
d. Finish Time										
e. Finish Temperature (Observed)										
Finish Temperature (Actual)	0	0	0	0	0	0	0	0	0	0
f. pH reading after heating (temperature corrected)										
D. Selection of Leach Fluid										
1. If pH from C.5. or C.7.f. is <5.0 use Leach Fluid #1										
2. If pH from C.7.f is > 5.0, use Leach Fluid #2										
3. SPLP Fluid 1: Soils- East of the Mississippi River; Wastewates; or Wastewaters										
4. SPLP Fluid 2: Soils- West of Mississippi River										
5. SPLP Fluid 3: If VOCs or Cyanide containing wastes.										
6. **X** if filtrate and fluid are miscible										

Attachment 10
TCLP Worksheet No. 3

Analyst: 0

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

DV-IP-0012
TCLP/SPLP Worksheet No. 3
Bottle Leach Procedure for Metals and Semi-Volatile Organic Components

Login No.										
Sample No.										
E. Determination of Sample Size										
1. Particle size reduction? Yes/no										
If yes, write NCM descibing how.										
2. Balance ID										
3. Weight of wet solids after filtration (g)										
F. Determination of Amount of Leach Fluid										
Fluid Type from Wksht 2	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!
1. TCLP Fluid 1 Lot #										
2. TCLP Fluid 2 Lot #										
3. SPLP 1 (East) Lot #										
4. SPLP 2 (West) Lot #										
5. pH of leach fluid										
6. Vol of Fluid = wet solids x 20 (mL)										
G. Record of Leach - leach period is 16 to 20 hours										
1. Rotator checked to be rotating between 28 and 32 RPM?										
2. Leach start date and time										
3. Leach stop date and time										
4. Room temperature										
Thermometer Correction Factor										
a.Temp Min (Observed) (°C)										
b.Temp Max (Observed) (°C)										
c.Temp Min (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
d.Temp Max (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5. pH of leachate										
Was the sample multiphasic?	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
6. Volume of leachate (mL)										
a. Oil recovered from leachate (mL)										
7. Volume of initial filtrate for recombination (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
8. Combined initial filtrate + leachate (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
COMMENTS:										

(Volume of Fluid, F.4) = (Weight of wet solids, E.3) X 20

(Vol. of Combined Filtrate and Leachate, G.8) - (Vol of Leachate, G.6) + (Vol of Filtrate, G.7)

$$(\text{Vol. of Inital Filtrate for Recombination, G.7}) = \frac{(\text{Solids Leachated, E.3})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{Leachate Recovered, G.6})}{(\text{Fluid Added F.6})} \times (\text{Initial Filtrat, B.7})$$

Attachment 11
TCLP Worksheet No. 4

Analyst: 0

DV-IP-0012
TCLP/SPLP Worksheet No. 4
ZHE Leach

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Login No.										
Sample No.										
H.Determination of Amount of Leach Fluid										
1. Balance ID										
2. ZHE vessel number										
3. Weight of material added to ZHE (g)										
"X" if there was headspace in container.										
4. Weight of filtrate in syringe										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5. Volume of filtrate in syringe (mL)										
6. Wet solids in ZHE (g)										
7. Weight of fluid to add (g)	0	0	0	0	0	0	0	0	0	0
a. TCLP Fluid 1 Lot #										
b. SPLP Fluid 3 Lot #										
c. pH of Blank Fluid										
8. Percent Wet Solids (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
I. Record of ZHE Leach - the Leach period is 16 to 20 hours.										
1. Leak Check										
a. Reading #1 (psi)										
b. Reading #2 (psi)										
2. Rotator checked to be rotating between 28 and 32 RPM?										
3. Leach start date & time										
4. Leach stop date & time										
5. Room temperature										
Thermometer Correction Factor										
a.Temp Min (Observed) (°C)										
b.Temp Max (Observed) (°C)										
c.Temp Min (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
d. Temp Max (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6. **X** if still under positive pressure after leaching										
7. Volume of leachate recovered (mL)										
a. Volume of oil recovered after leaching										
8. Vol. of initial aqueous filtrate for recombination										
a. Calculated from Worksheet 1 (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
b. Calculated from Worksheet 4 (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

(Net Weight of Filtrate, H.4.c) = (Gross weight, H.4.a.) - (Tare weight, H.4.b.)
(Percent Wet Solids, H.8.) = 100 X **[** (Wet Solids in ZHE, H.6) / (Weight of material added to ZHE, H.3) **]**
(Wet Solids in ZHE, H.6) = (Weight of material added to ZHE, H.3.) - (net weight of filtrate, H.4.c.)
(Vol of Filtrate for recombination, I.7.b) = (Vol of Leachate Recvd, I.6) X (Weight of Filtrate, H.4.c) / (Vol Fluid Added, H.7.)
(Weight of Fluid to add, H.7.) = (Wet Solids in ZHE, H.6) X 20
(Vol of Filtrate for recombination, I.7.a) = **[** (Wet Solids in ZHE, H.6.) / (Tot Wet Solids, B.4) **]** X **[** (Vol Leachate Recvd, I.6.) / (Vol Fluid Add, H.7.) **]** X (Vol Filtrate, B.7.)

COMMENTS:

Attachment 12

How to Batch TCLP and SPLP:

1311_T (Organics) **1311T_Hg** (Mercury) **1311T_M** (Metals)	**1312_E** (Organics) **1312_E_Hg** (Mercury) **1312_E_M** (Metals)	**1312_W** (Organic) **1312_W_Hg** (Mercury) **1312_W_M** (Metals)	**1311_Z** (ZHE)

Overview

The pre-prep methods listed above are specific to the analytes requested, but it is not necessary to batch them all separately. Above, the methods are placed in boxes to indicate which methods can be batched together, with one exception: *SPLP 8260s will be logged with 1312_E or 1312_W, which is the same leach method used for organic bottle preps so not all 1312_E can be batched together and not all 1312_W can be batched together*.

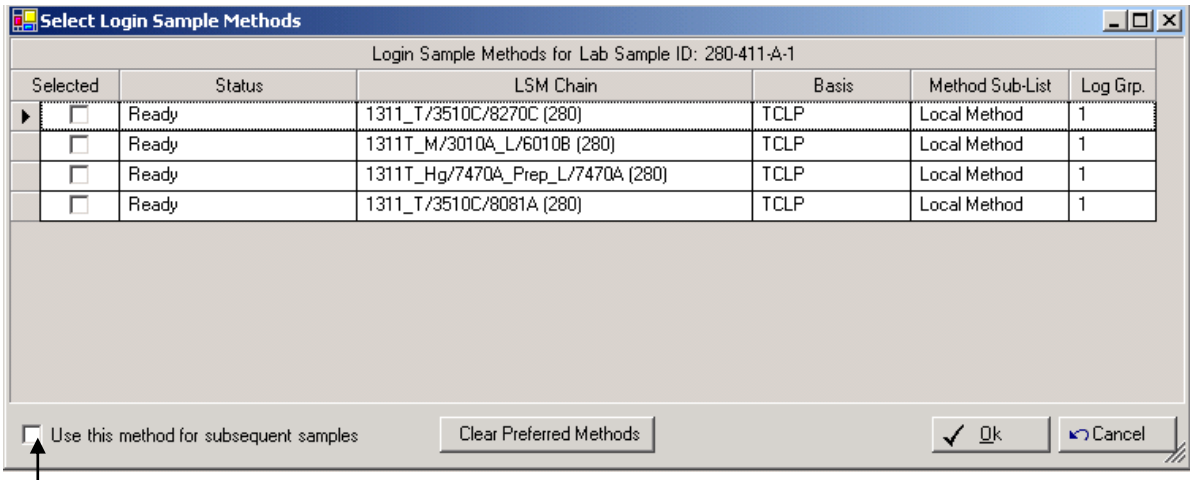
If one sample is logged in for TCLP 8270C, TCLP 8081B, TCLP 8260, TCLP 6010B, and TCLP 7470A, the sample will show up on the Organic Extractions backlog 5 times for TCLP, (once for each analytical method), and twice for 3510C.

Record Status	Status	A-Status	HT Expires	Rush	Method	A-Method	Job Number	Lab Sample ID	Container Matrix
Active	Ready	Active	1/23/2010 11:59	☐	1311_T	8081A	280-J411-1	280-411-1	Solid
Active	Ready	Active	1/23/2010 11:59 PM	☐	1311_T	8270C	280-J411-1	280-411-1	Solid
Active	Ready	Active	1/23/2010 11:59	☐	1311_Z	8260B	280-J411-1	280-411-1	Solid
Active	Ready	Active	2/6/2010 11:59	☐	1311T_Hg	7470A	280-J411-1	280-411-1	Solid
Active	Ready	Active	7/8/2010 11:59	☐	1311T_M	6010B	280-J411-1	280-411-1	Solid
Active	Wait	Active	1/23/2010 11:59	☐	3510C	8081A	280-J411-1	280-411-1	TCLP Leach
Active	Wait	Active	1/23/2010 11:59	☐	3510C	8270C	280-J411-1	280-411-1	TCLP Leach

For the sample above, we would leach the sample in a glass bottle for the organics and metals and we would also do a ZHE leach. Therefore there will be 2 leach batches.

Simple Steps

1. Run the OP - TCLP backlog. This backlog is sorted by sample ID so samples logged for multiple extraction and analytical methods will be grouped by sample. Pull the samples from the walk-in cooler and take custody of the samples.
2. Use the TCLP spreadsheets in EXCEL to determine blank fluid for each sample. Once the leach fluid has been determined, you will know what samples can be batched together. You can not put samples with different leach fluids in the same batch.
3. Open Analyst Desktop and select Create Batch from Scratch
4. Your Batch Notes will appear, but we are not going to use the Batch Notes. Instead all of our data will be recorded in the TCLP spreadsheets in EXCEL.
5. Scan your samples into the batch. If your samples are logged in for more than one of the leach methods, a window will appear called “Select Login Sample Methods”.



6. Select all LSM Chains that include the leach preps. If all of the samples that you want to batch together are logged in for the same methods, you can click the box in the lower left-hand corner that says “Use this method for subsequent samples”. Then click “OK”. **NOTE**: Be careful when clicking the “Use this method for subsequent samples” box. If you click this and the subsequent samples have more methods than the ones listed in the LSM box, they will not be included in the batch. You can check this in Step 10 below and fix it there if there is something wrong.
7. If your batch is for TCLP Fluid #1 or SPLP East Fluid, create a “LB” for the Leach Blank. If your batch is for TCLP Fluid #2 or SPLP_West Fluid, create a “LB2” for the Leach Blank. If your batch is for water TCLP samples, water SPLP samples, or SPLP ZHE samples, then you are using reagent water as your blank fluid and create a “LB3” for your Leach Blank There will be no other QC here at this point unless a client has requested MS/MSD on a sample. If that is the case, add the MS/MSD to the leach batch, but it does not get spiked before the leach.
8. Go to the Sample List tab. Here you will see that if the sample was logged in for more than one method chain, the sample will be listed here multiple times – one for each method chain. It is a good idea to check your backlog against the Sample List tab to make sure that all of your method chains that were listed on the backlog are in the batch. If they are not, right click on the sample and click on “Select LSM” to add the missing tests into the batch.
9. Go to the Worksheet tab. We won’t be using the fields here to record our data because the calculations are not locked. We will use the TCLP spreadsheet instead. **But we will have to enter the Leach Fluid type or else our spreadsheets will not get into the raw data. Scroll all the way over to the right and enter “T1”, “T2” “Milli-Q”, “SE”, “SW” or “S3”.**
10. We will use a different status to indicate where the samples are.
    a. A status of “Batched” or “1st Level Review”means the blank fluid determination is done, samples are tumbling.
    b. A status of “2nd Level Review” means that the samples have completed the leachate and have been filtered.
11. Once all steps in the procedure are complete, save the TCLP worksheet in EXCEL. Then print it to pdf and save it in the same directory as the EXCEL file. **When you print it to pdf, be sure to select “Entire Workbook” so that all worksheets will be in the pdf.**
12. Go into the TALS batch and click on the documents button. Right click and from the menu select “Change Document Type”, and then select “External Prep Worksheet”. Attach the pdf of the EXCEL spreadsheet and the pdf of the LogTag Summary to the TALS batch.

Attachment 13
Instruction Manual for pH Meter

pH Calibration

1. Prepare the electrode according to the electrode user guide.
2. In the setup mode, select the buffer set (USA or EUro) that will be used for the automatic buffer recognition feature.
3. In the measurement mode, press (☰) until the arrow icon points to the top line, press (△) until the **pH** icon is shown and press (↙) to begin the calibration.
4. Rinse the electrode, and ATC probe if being used, with distilled water and place into the buffer.
5. Wait for the **pH** icon to stop flashing.
   a. Automatic buffer recognition – When the **pH** icon stops flashing the meter will display the temperature-corrected pH value for the buffer.
   b. Manual calibration – When the **pH** icon stops flashing the meter will display the actual pH value read by the electrode. Press (⚙) until the first digit to be changed is flashing, press (△) / (▽) to change the value of the flashing digit and continue to change the digits until the meter displays the temperature-corrected pH value of the buffer. Once the pH buffer value is set, press (⚙) until the decimal point is in the correct location.
6. Press (↙) to proceed to the next calibration point and repeat steps 4 and 5 or press (🌡) to save and end the calibration.
7. The actual electrode slope, in percent, will be displayed in the main field and SLP will be displayed in the lower field.
   a. For a one point calibration, press (⚙) and (△) / (▽) to edit the slope and press (🌡) to return to the measurement mode.
   b. For a two or more point calibration, the meter will automatically proceed to the measurement mode after the slope is displayed.

Attachment 14
SPLP worksheet for correcting fluid pH

Dilution of Wrong SPLP pH with Water

	Inputs	Units	Note
pH of solution	1.29		Make sure is below Target pH
pH of Water	5.23		Elga pH is usually 5.25
Target pH	4.2		NOT pH 7
Target Volume (L)	45	**L**	
		Units	
Required Volume of Water	44.95	**L**	
Required Volume of Solution	0.05	**L**	

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Acid digestion of Aqueous Samples for Analysis by ICP- MS [SW-846 3005A, 3020A, and EPA Method 200.8.]

Approvals (Signature/Date):

Doug Gomer 10/31/18
Doug Gomer Date
Technical Specialist

Doug Gomer 10/31/18
Doug Gomer Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 10/31/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 10/31/18 Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This procedure describes the preparation of aqueous samples for the analysis of metals by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) using EPA Method 200.8, and SW-846 Methods 3005A and 3020A.

1.2 Aqueous samples also include TCLP & SPLP Leachates, aqueous equipment rinse blanks for soil sampling. In some cases, where the associated soil samples require the SW-846 Method 3050B, Section 7.5, optional treatment to improve solubility and recovery of Sb, Ag, and Sn, the client may require that the aqueous equipment blank receive the same treatment. Refer to section 10.14 for this prep.

1.3 The applicability of each of these preparation protocols to specific analytes is detailed in the TestAmerica LIMS System (TALS). Additional elements may be analyzed following digestion by these protocols, provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.4 This SOP provides procedures applicable to the preparation of dissolved, total recoverable, potentially dissolved, and total metallic elements in ground water, aqueous samples, aqueous sludges, aqueous wastes, aqueous air sampling media, and leachates/extracts. This SOP is not applicable to samples that contain or consist of oil or other immiscible organic solvents.

NOTE: Samples that are known to be immiscible with water, e.g., contain or consist of oil or other immiscible organic solvents should be logged with a waste matrix and subbed out to a different TestAmerica laboratory for digestion and analysis.

1.5 SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP-MS. Although digestion is not specifically required by the method (SW-846 3005A section 2.2) for dissolved samples, the standard operating procedure at TestAmerica Denver is for all matrices to be digested prior to analysis.

1.6 EPA Method 200.8 Section 11.2 is used to prepare surface water, drinking water, and domestic and industrial waste samples for total recoverable and dissolved metals.

1.7 SW-846 Method 3020A is used to prepare aqueous samples, TCLP leachates, SPLP leachates and aqueous wastes that contain suspended solids for total metals analysis by ICP-MS.

1.8 The following table lists the sample preparation methods that are covered in this SOP and the specific section of this SOP for each preparation method. Prepared samples are analyzed by inductively coupled plasma-mass spectrometry (ICP-MS).

PREPARATION METHOD	SOP SECTION	DETERMINATIVE METHOD	ANALYTICAL SOPS #
Method – 3020A Total	10.11	ICP-MS	DV-MT-0018 DV-MT-0022
Method – 3005A Total Rec./Dissolved	10.12	ICP-MS	DV-MT-0018 DV-MT-0022
Method 200.8 – Total Rec.	10.13	ICP-MS	DV-MT-0002
Method 200.8 – Dissolved	10.13	ICP-MS	DV-MT-0002
Method - 3050B Special Sb Prep	10.14	ICP-MS	DV-MT-0018 DV-MT-0022

2.0 Summary of Method

2.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals.

This preparation method is used for total recoverable and dissolved metals analysis by ICP-MS method 6020 and 6020A. A representative aliquot of sample is heated with nitric acid and substantially reduced in volume. The digestate is diluted to volume and then filtered (if necessary).

2.2 Method 3020A, Acid Digestion of Aqueous Samples and TCLP/SPLP Leachates for Total Metals.

This preparation method is used for total metals analysis by ICP-MS method 6020 and 6020A. A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. The digestate is diluted to volume and then filtered (if necessary).

2.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry.

This preparation method is used for metals analysis by ICP-MS method 200.8. A representative aliquot of sample is refluxed with nitric and hydrochloric acids. The digestate is diluted to volume and then filtered (if necessary).

3.0 Definitions

Additional definitions of terms used in this SOP may be found in the glossary of the QAM.

- Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45-μm membrane (Method 3005A). (The sample is acidified after filtration).

- Total Metals: The concentration of metals determined in an unfiltered sample following digestion (Method 3020A).

- Total Recoverable Metals: The concentration of metals determined in an unfiltered sample following treatment with hot, dilute mineral acid (Method 200.8 and Method 3005A).

- Potentially Dissolved Metals: An acidified sample is filtered between 8- 96 hours following acidification and the filtrate is digested using Method 3005A.

- Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include the following: metallic or metal-containing labware (e.g., latex gloves coated with talc, which contains high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.4 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.5 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared.

4.6 Specific analytical interferences are discussed in the ICP-MS determinative method SOPs, e.g., DV-MT-0002, DV-MT-0018, and DV-MT-0022.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns

5.1.1 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids

are added.

5.1.2 The digestion solution must be cooled sufficiently before adding hydrogen peroxide (H_2O_2) to avoid a reaction and possible violent effervescence, or boiling over of the digestion solution.

5.1.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the sample digestate.

5.1.4 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	**Hazards**	**Exposure Limit** [(2)]	**Signs and Symptoms of Exposure**
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm (TWA)	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Digestion block, with adjustable heating, capable of maintaining a sample temperature of 85 - 95 °C.

6.1.2 Thermometer that covers a temperature range of at least 80 - 110 °C.

6.1.3 Centrifugation equipment (if desired method of removing particulate material is centrifugation).

6.2 Supplies

6.2.1 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.2 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.3 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.4 Syringes or equivalent filtration apparatus.

6.2.5 Repipettors or suitable reagent dispensers.

6.2.6 Calibrated automatic pipettes with pipette tips or Class A glass volumetric pipettes.

6.2.7 Class A volumetric flasks.

6.2.8 pH indicator strips (pH range 0 - 6).

6.2.9 Plastic digestate storage bottles.

7.0 Reagents and Standards

7.1 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative method SOPs, e.g., DV-MT-0002, DV-MT-0018, and DV-MT-0022.

7.2 Laboratory control sample (LCS), and matrix spike and matrix spike duplicate (MS/MSD) spike solutions are purchased as custom TestAmerica Denver solutions. Standards are logged into the Reagents module of TALS and are assigned unique identification numbers that can be used to access traceability information. The TALS identification numbers are recorded on the metals prep bench sheet.

7.2.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. These plastic bottles may be stored in a glass jar.

7.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3 See TALS for the list of spiking levels. A volume of 0.1 mL of each working spike solution is added to the 50-mL final sample volume.

7.3 Nitric Acid (HNO_3), concentrated, trace-metal grade or better.

NOTE: When preparing diluted acids, always add acid to water. If the water is added to the acid, the sudden increase in temperature may cause splashing.

7.4 Nitric Acid, 1:1

Dilute concentrated HNO_3 with an equal volume of reagent water.

7.5 30% Hydrogen Peroxide (H_2O_2), ultra pure grade.

7.6 Hydrochloric Acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE Or Glass	500 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS login, sample or method comments and/or program QAS to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified appropriately. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Table 2 provides a summary of quality control requirements, including type, frequency, acceptance criteria, and corrective action. Detailed information regarding acceptance criteria and corrective action are found in each determinative method SOP.

9.3 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. Ongoing proficiency must be demonstrated by each analyst on an annual basis. See

Section 12.1 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.4 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). For samples logged in under Method 200.8, there must be two MS/MSD pairs for every batch containing more than ten samples. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

9.5 Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are usually not included in the sample count.

9.6 Method Blank (MB)

The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

9.7 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs)

may require the use of sample duplicates in place of or in addition to MS/MSDs. At least one MS/MSD pair must be processed for each preparation batch. Some client programs require a 10 % MS/MSD analysis frequency. If insufficient sample is available to process an MS/MSD pair, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede the SOP requirements. Quality Assurance Summaries (QASs) are developed to address these requirements.

10.0 Procedure

Sample Preparation

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervisors to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3 All samples are to be checked out of Sample Control with the Internal Chain of Custody in TALS properly completed.

10.4 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.5 Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be more like a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), then contact the project manager and the laboratory group leader for further instructions.

10.6 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab, data review, and reporting groups.

10.7 Guidelines are provided in Appendix A on procedures to minimize contamination of samples and standards.

10.8 All data shall be recorded directly on the described forms, logbooks, electronic forms, or directly in TALS at the time of data generation. It is not acceptable to record data on loose papers, scraps of paper, gloves, sample vials, or "Post-It" notes. Data may be recorded on paper bench sheets if the sheets are subsequently scanned and saved in a designated folder on the company server.

10.9 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure:

10.9.1 Sample pH is verified during sample receipt. When a sample is received with improper/insufficient preservation, the sample is delivered with notification of the deficiency.

10.9.1.1 Measure the sample pH with pH paper using a separate aliquot of sample. Do not put the pH paper directly into to bottle. Record the pH on a copy of the internal chain of custody (ICOC). When all of the samples have been tested, initial and date the copy of the ICOC, scan it, and save it to the Metals folder on the G: drive.

10.9.1.2 If the pH>2 for a sample requiring acidic preservation, add 1-2 mL of conc. HNO_3 to the sample. Replace the lid and mix.

10.9.1.3 Recheck the pH of the sample. If the pH<2, record the sample in the Metals preservation logbook. If the pH>2, repeat 10.9.1.2 until pH<2 or 5mls has been added. If the sample still has a pH greater than 2 do not add any additional acid and create an NCM. Add the sample to the Metals preservation logbook

10.9.1.4 Allow the sample to sit for 24 hours following acidification.

10.9.1.5 After 8-16 hours, recheck the pH of the sample. If the pH<2, proceed with the appropriate digestion procedure. Note the date/time of this pH recheck in the Metals preservation logbook.

10.9.1.6 If after 8-16 hours the pH>2, repeat steps 10.9.1.2 through 10.9.1.5 until the pH remains <2 following the 8-16 hour period or 5mls of HNO_3 has been added.

Note: Acid must be added at least 24 hours before analysis.

10.9.2 Select the unfiltered fraction for a total or total recoverable analysis or the

filtered fraction for a dissolved analysis. For SPLP select the proper sample leachates.

NOTE: Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number. Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples.

10.9.3 Mix the sample by shaking the container.

10.9.4 Measure and transfer 50 mL of the sample into a digestion tube. When using calibrated digestion tubes, pour the sample into the tube to the 50-mL mark. Record the lot number of the digestion tubes in TALS.

10.9.5 Measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot with 0.1 mL of each spiking solution (see TALS). Record the standards and pipette identifications in TALS.

10.9.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested, use filtered reagent water for the method blank.

10.9.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add 0.1 mL of spiking solution (see Table 2). Record the standards and pipette identifications in TALS. If determination of dissolved metals is requested (preparation method 3005A), and one or more samples were filtered in the laboratory, then filter the LCS and Method Blank using a filter of the same type that was used to filter the sample(s).

10.10 Proceed to the appropriate Section of this SOP for the desired preparation method as follows:

Preparation Method*	SOP Section	Analytical Method
3020A Total Metals	10.11	Method 6020
3005A Total Recoverable	10.12	Method 6020
3005A Dissolved Metals	10.12	Method 6020
200.8 Total Recoverable Metals	10.13	Method 200.8

200.8 Dissolved Metals	10.13	Method 200.8
3050B Special Sb prep	10.14	Method 6020

(See also WI-DV-017)

10.11 Method 3020A - Preparation for Total Metals Analysis by ICP-MS Method 6020 and 6020A

10.11.1 To the sample in a digestion tube, add 1.5 mL of concentrated HNO_3.

10.11.2 Heat at 90 - 95 °C until the volume is reduced to approximately 5 mL. Record the start time and the Hot Block temperature in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared and reanalyzed.

10.11.3 Allow the digestion tube to cool in a fume hood.

10.11.4 Add 1.5 mL of concentrated HNO_3. Cover and reflux gently.

10.11.5 Continue heating, adding additional acid as necessary in 1-2 mL increments to ensure a complete digestion. Record the start and stop times and the Hot Block temperature in TALS.

NOTE: Digestion is complete when the digestate is light in color and does not change in appearance with continued refluxing.

10.11.6 Evaporate to low volume, approximately 3 - 5 mL.

10.11.7 Allow the digestion tube to cool, then add about 10 mL of reagent water.

10.11.8 Replace the cover and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur. Record the stop time in TALS.

10.11.9 Allow the sample to cool and rinse the watch glass into the digestion tube with reagent water.

10.11.10 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.11.11 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018 or DV-MT-0022.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0018 or DV-MT-0022 for additional details.

10.12 Method 3005A - Preparation for Total Recoverable and Dissolved Metals Analysis by ICP-MS Method 6020 and 6020A

10.12.1 To the sample in a digestion tube, add 2.0 mL of concentrated HNO_3.

10.12.2 Heat the sample to 90 - 95 °C and cautiously evaporate to approximately 10 mL, while ensuring that no portion of the sample container is allowed to go dry. Record the start and stop times and the Hot Block temperature in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.12.3 Allow the sample to cool in a fume hood.

10.12.4 Rinse the digestion tube with reagent water.

10.12.5 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.12.6 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0018 or DV-MT-0022 for additional details.

10.13 Method 200.8 - Preparation for Total Recoverable/Potentially Dissolved/Dissolved Metals Analysis by ICP-MS

10.13.1 To the sample, add 0.5 mL of concentrated HNO_3 and 0.25 mL of concentrated HCl.

10.13.2 Adjust the digestion block temperature so the solution in a covered container rises to approximately 90 - 95 ºC. Record temperature on bench sheet.

10.13.3 Heat the sample until it evaporates to approximately 10 mL, while ensuring that no portion of the bottom of the digestion tube is allowed to go dry.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.13.4 Cover the sample and gently reflux for an additional 30 minutes. Avoid vigorous boiling to prevent the loss of the HCl-H_2O azeotrope. Record the start and stop times and the Hot Block temperature in TALS.

10.13.5 Allow the sample to cool in a fume hood.

10.13.6 Rinse the watch glass or cover into the container and re-volume to 25 mL with reagent water. Cap and mix thoroughly.

NOTE: If the samples are being prepared to satisfy drinking water compliance requirements, bring the samples to a final volume of 50 mL before capping and mixing. Samples logged for this purpose will have attached TALS Method Comments to that effect.

10.13.7 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0002.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0002 for additional details.

10.14 Method 3050B – Special Prep for Sb, Sn and Ag for Analysis by ICP-MS Method 6020

10.14.1 To 25 mL of sample in a digestion tube, add 2.5 mL of HNO_3 and 2.5 mL of HCl.

10.14.2 Heat at 90-95 °C until the sample has reduced to a volume of 10-15 mL ensuring that no portion of the sample container is allowed to go dry.

Record the start and stop times and the Hot Block temperature in TALS.

10.14.3 Remove the sample from the Hot Block and allow it to cool in a fume hood.

10.14.4 Add 1.0 mL of HCl to the digestion tube and cover with a ribbed watch glass.

10.14.5 Replace the watch glass and heat the sample for 15 minutes.

Record the start and stop times and the Hot Block temperature in TALS.

10.14.6 Remove the sample from the Hot Block and allow it to cool in a fume hood.

10.14.7 Re-volume to 100 mL with reagent water, cap and mix thoroughly.

10.15 Calibration

The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded on the metals preparation bench sheet. The temperature must be monitored by measuring the temperature of reagent water contained in a digestion tube that is placed in each digestion block. The thermometer used and the start and end time temperatures are recorded in TALS. The thermometer is calibrated in accordance

with SOP DV-QA-0001, *Thermometer Calibration Procedures*.

11.0 Calculations / Data Reduction

11.1 Not applicable. See the determinative method SOPs, DV-MT-0002, DV-MT-0018 and DV-MT-0022 for data analysis and applicable calculations.

11.2 Documentation

11.2.1 All of the preparation information is recorded and stored in TALS.

11.2.2 The preparation information includes:

11.2.2.1 Batch number, job and sample numbers, preparation date, and analyst name;

11.2.2.2 Matrix and prep type;

11.2.2.3 Initial sample pH, Initial sample volume and final volume;

11.2.2.4 Reagent manufacturer and lot number for each reagent used;

11.2.2.5 Digestion tube lot information;

11.2.2.6 Standard identification number for each standard used;

11.2.2.7 Start and stop times for digestions;

11.2.2.8 Observed and corrected temperature readings during digestion;

11.2.2.9 Identification numbers of calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.3 Method 6020, Inductively Coupled Plasma - Mass Spectrometry, Revision 0, September 1994.

15.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

15.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectroscopy, Revision 5.4, May 1994.

16.0 Method Modifications:

16.1 Modifications and Interpretations Applicable to SW-846 Reference Methods

16.1.1 Chapter 1 of SW-846 states that the method blanks should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above one-half of the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

16.1.2 The referenced methods, as well as Table 3-1 of SW-846, refer to the use of a 100-mL aliquot for digestion. This SOP requires the use of a 50-mL sample size to reduce waste generation. The use of reduced sample volumes is supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition", dated November 3, 1994. This document stated, "...flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..."

EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a

significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated. " As a 'representative sample' can be assured, scaling causes no loss of precision and accuracy in the analysis."

16.2 Modifications Specific to Method 3005A

An additional 1.0 mL of HNO_3 was included to replace the 5.0 mL of HCl. HCl was eliminated to reduce interferences from chloride.

16.3 Modifications and Interpretations Specific to Method 3020A

16.3.1 Section 10.11.6 of this SOP requires that the sample be reduced to a volume of 3-5 mL. Section 7.2 of Method 3020A states that the volume should be reduced to 3 mL, but also states that no portion of the bottom of the digestion tube should go dry. The volume required by this SOP is a closer approximation of the volume required to provide an adequate covering of the bottom of the digestion tube so as to prevent the loss of critical analytes through volatilization.

16.3.2 The scope of 3020A has been expanded to include silver, based on comparison studies with 7760A. Method 3020A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water, and TCLP leachate) up to a concentration of 1 ppm silver.

17.0 Attachments

Table 1. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels
Table 2. Summary of Quality Control Requirements
Appendix A. Contamination Control Guidelines

18.0 Revision History

- Revision 11, Dated 31 October 2018
  - Annual Review
  - Updated Section 10.12.2 volume to approximately 10 mL.
  - Updated Copyright section
  -
- Revision 10, Dated 31 October 2017
  - Annual Review
  - Removed Note about sample count for AFCEE

- Revision 9, Dated 30 September 2016
  - Annual Review
  - Removed reference to Method 3050 from title page and Sections 1.1 and 15.
  - Removed Acceptance Criteria and Corrective Actions for QC samples from Section 9. These are addressed in each determinative SOP.
  - Updated Section 10.9.1.4 preservation time to 24 hours
  - Added method blank to section for filtration if samples are filtered. All QC must be treated the same.
  - Revised Section 12 to reflect current practice
  - Revised Table 2 to include all determinative method SOPs

- Revision 8, Dated 30 September 2015
  - Added drinking water to Section 1.6.
  - Corrected Section reference tables.
  - Added note to Section 10.13.6 describing changed final volume requirement.

- Revision 7, Dated 30 June 2015
  - Updated Section 10.13.2 for temperature requirement of 90-95 for method 200.8
  - Updated Section 10.9.1.1 for proper technique when using pH paper.
  - Added new Section 10.8 reminding analysts to enter data directly at time of acquisition
  - Added Section 11.2 describing required data to be recorded
  - Removed Section 11.4

- Revision 6, Dated 29 September 2014
  - Annual review
  - Removed references to microwave procedure
  - Removed direct-shoot for dissolved analysis
  - Corrected section references
  - Replaced LIMS and Standards Log references with TALS
  - Clarified the number of MS/MSDs per prep batch based on method
  - Removed workflow diagrams Figures 1-4
  - Removed Tables 1 and 2
  - Minor spelling and language corrections throughout

- Revision 5, Dated 30 September 2013
  - Reference to SOP DV-IP-0017 for preparation of organic waste
  - Formatting updates
  - Updated section 9, 12 & 14 to include more detail
  - Annual review
  - Updated sections 10.8.1.2 - 10.8.1.4 to removed reference to amount of HNO_3 acid added.
  - Added to Section 10.8.1.6 that a maximum of 5mls of HNO_3 can be added.

- Revision 4, Dated 28 September 2012
  - Annual review
  - Section 9.6 Updated method blank control limits to ½ the reporting limit.
  - Updated appendix B with revised Work Instruction.

- Revision 3.5, dated 23 September 2011
  - Annual Technical Review
  - Removed reference to Supplemental Metals Prep Sheets in Sections 10.10.8 and 10.13.5.1
  - Removed reference to Clouseau in Section 10.8.1.2
  - Removed references to LIMS codes in Appendix B

Earlier revision histories have been archived and are available upon request.

Table 1.

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

Table 2.

Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-digest and reanalyze samples associated with the method blank
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-digest and reanalyze samples associated with the method blank
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-prep not required unless preparation error suspected.
Matrix Spike Duplicate (MSD)	See Matrix Spike frequency	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	See Corrective Action for Matrix Spike

Appendix A.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: ACID DIGESTION OF SOLIDS [Method EPA 3050B]

Approvals (Signature/Date):

Doug Gomer 10/31/18
Doug Gomer Date
Technical Specialist

Doug Gomer 10/31/18
Doug Gomer Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 10/31/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 10/31/18
Richard Clinkscales Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This is a strong acid digestion procedure for the preparation of sediments, sludge, soils, and other types of solid materials by EPA Method 3050B for analysis by inductively coupled plasma atomic emission spectroscopy (ICP) or inductively coupled plasma-mass spectrometry (ICP/MS).

1.2 Method 3050B is designed to determine the concentration of "environmentally available" metals, and is not a true "total metals" digestion (see discussion below). The procedure is used primarily for hazardous waste characterization and other Resource Conservation and Recovery Act (RCRA) compliance testing.

1.3 The elements approved for Method 3050B are shown in Table I. The source method also mentions that other elements may be prepared by the method if the quality control requirements are met. The complete list of elements routinely included in this procedure by TestAmerica Denver is shown in Table II.

1.4 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

2.0 Summary of Method

A representative 1 to 2 gram portion of sample is digested with two cycles of nitric acid additions, followed by hydrogen peroxide digestion. For ICP analysis, the sample is also refluxed with hydrochloric acid. The resulting solution is filtered and diluted to 100 mL with reagent water. For the Incremental Sampling Method, 10 g of sample is used and brought to a final volume of 500 ml.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.2 **Total Metals -** Although Method 3050B is often referred to as a "total metals" digestion, it is important to understand that there are many compounds formed from these elements that are not efficiently dissolved using this digestion procedure. It is more accurately termed a strong acid digestion procedure. The limitations are discussed further in Section 4 (Interferences) below. The method itself states, "This method is not a total digestion technique for most samples." There are a variety of total digestion procedures used for metal assay, geochemical analysis, etc., that involve more vigorous digestions than 3050B.

3.3 **Preparation Batch -** A group of up to 20 samples that are of the same matrix and are processed together using the same lots of reagents and standards. The minimum QC elements in a batch are outlined in Section 9.

3.4 **Reagent Water –** Water that is free of the analytes of interest. In the Metals group, reagent water is obtained from a Barnstead E-Pure water purification system.

3.5 Other quality control terminology used in this procedure is based on SW-846, and is defined in the glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*.

4.0 Interferences

4.1 There are common compounds formed by the elements of interest (e.g., barium sulfate, beryllium oxide, silicon dioxide, crystalline silicates, titanium dioxide, etc.) that are not efficiently dissolved using this EPA approved procedure.

4.2 Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will include only acid-soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, but the final result is sometimes expressed as silica rather than silicon.

4.3 Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP-MS procedure that includes HCl as the final digestion acid. See Section 11.12 of this SOP.

4.4 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. See Attachment 1 for more information regarding contaminant control.

4.5 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.6 For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.7 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrix materials may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.8 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals or conversion of metals to insoluble forms. For example, antimony is easily lost by volatilization from hydrochloric media. If this occurs the sample must be re-prepared.

4.9 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.10 Samples Requiring Additional Digestion Reagents

A few examples of types of samples that might require additional digestion reagents follow. It is very important to note situations where samples are not

behaving normally. However, do not assume that adding additional reagents will be acceptable for the project, even if it is obvious that the digestion will be incomplete without it. The situation must be discussed with the project manager and documented in a Nonconformance Memo (NCM), whether or not the variations suggested in the following examples are approved.

4.10.1 Samples with high organic content may require additional nitric acid and/or hydrogen peroxide for a thorough digestion, but these oxidizing reagents should be added very carefully to avoid violent reactions.

4.10.2 Samples with high concentrations of metal in the elemental form or refractory oxides may require additional hydrochloric acid for a thorough digestion. As an example, blasting sand used to remove paint from the hull of ships typically consists of 30% cupric oxide. Following 3050B exactly will produce results as low as 0.1% without additional hydrochloric acid. Increasing the volume of hydrochloric acid can produce results approaching the true copper concentration. Samples that appear to have nonstandard matrices or have visible metal particles should be documented in an NCM.

4.10.3 Highly alkaline materials may require larger volumes of acid than specified in this procedure.

4.10.4 If the use of extra digestion reagents is approved, the same volume of reagents must be added to all field samples and QC samples in the batch. Usually the method blank results will not be elevated. To ensure that the QC sample results accurately reflect sample results, the QC samples must be treated exactly like the samples.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic materials or samples that are at elevated pH can react violently when acids are added. If any solid sample appears to be a chemical substance rather than an environmental sample, consult with the group supervisor or the Project Manager (PM) before adding acid.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and

reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit(2)	Signs and Symptoms of Exposure
Hydrogen Peroxide, H_2O_2	Oxidizer Corrosive Poison	1 ppm TWA 1.4 mg/m^3 TWA 75 ppm IDLH	Contact with other materials may cause fire. Eye contact may result in permanent eye damage. Causes eye and skin burns. Corrosive: May cause severe respiratory tract irritation. Harmful if swallowed, may cause digestive tract irritation or burns.
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid, HCl	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.

(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Top-loading balance capable of accurately weighing to the nearest 0.01 grams.

Note: Balances are serviced annually and the accuracy checked daily using three standard masses. See SOP DV-QA-0014 for details.

6.1.2 Digestion "Hot Block" or equivalent heating device capable of maintaining a temperature of 90-95 °C. The Hot Block temperature must be monitored separately for each unit. The temperature of each Hot Block is checked by placing a calibrated thermometer through a cap on a digestion tube that is partially filled with water. The water in the tube must be high enough to cover the thermometer past the minimum immersion line. The temperature is directly recorded in the Batch Information area in the TestAmerica LIMS (TALS).

6.2 Supplies

6.2.1 Thermometers (non-mercury liquid filled or digital) that cover a temperature range including 80-110 °C with clearly visible 1 °C increments.

Note: Thermometers are calibrated before use and periodically as described in SOP DV-QA-0001.

6.2.2 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.3 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.4 Whatman 541 (acid washed) filter paper, or equivalent.

6.2.5 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.6 Syringes or equivalent filtration apparatus.

6.2.7 Disposable plastic funnels.

6.2.8 Disposable wooden spatulas for subsampling.

6.2.9 Centrifuge, capable of at least 2,000 rpm.

6.2.10 Graduated cylinders, 100 mL and 500 mL, capable of ± 3% accuracy.

6.2.11 Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

Note: Mechanical pipettes are calibrated before use as described in SOP DV-QA-0008.

6.2.12 Class A volumetric flasks.

6.2.13 pH indicator strips (pH range 0 – 6).

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent water – Millipore DI system or equivalent, 10-18.2 megohm-cm. See SOP DV-QA-0026 for daily water monitoring procedure.

7.2 Nitric acid (HNO_3), concentrated - Trace metal grade or better.

7.3 Nitric acid (HNO_3), 5% - Add 50 mL of concentrated HNO_3 to approximately 900 mL of reagent water and dilute to 1 liter.

7.4 Hydrochloric acid (HCl), concentrated - Trace metal grade or better.

7.5 30% Hydrogen peroxide (H_2O_2) - Reagent grade used for ICP analysis.

7.6 30% Hydrogen peroxide (H_2O_2) – Ultra pure used for ICP-MS analysis.

7.7 Glass beads, ≤ 1 mm diameter, washed with aqua regia (for DoD projects).

7.8 Standards

7.8.1 All standards must be NIST traceable. Unless purchased directly from NIST, the accuracy of each standard is verified before the initial use, as described in SOP DV-QA-0015.

7.8.2 Storage and Shelf Life of Metal Standards

7.8.2.1 Standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. They are stored at room temperature.

7.8.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.8.3 LCS and MS Spike Solutions

7.8.3.1 ICP and ICP/MS spike solutions are purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed.

7.8.3.2 If a non-routine element is required that is not contained in the custom-made solution, single-element solutions from a commercial vendor may also be used.

7.8.3.3 Intermediate standards prepared in the laboratory may be used for spiking as long as the procedures for standard recording and verification outlined in SOP DV-QA-0015 are followed.

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	ICP SPK 3A	Ag, Be, Cd Cr Cu Co, Mn, Ni, Pb, V, Zn As, Fe, Li, Sr, Th Al, Ba, Bi, Se, Tl, U P Ca, K, Mg, Na	5 10 20 25 50 100 200 1,000 5,000
ICP	ICP SPK 2B	Sb, Zr B, Mo, Ti Sn, S Si (SiO_2)	50 100 200 1,000 (2,140)

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP-MS	MS CALSTD-1	Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Th, Tl, U, V, Zn	20
ICP-MS	MS spike 2	Mo, Sb, Sn, W, Zr Al, Fe	20 200

Note: ICP or ICP-MS digestions may select different combinations of spikes in order to satisfy client requests. All spikes used for sample digestion will be recorded in the Reagent module in TALS.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2 Soil samples do not require chemical preservation, but are stored at ≤6 °C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation[1]	Holding Time[2]	Reference
Soils	Glass	3 grams	Cool ≤ 6 °C	180 Days	N/A

[1] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

[2] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0

unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation batches may consist of up to 20 field samples. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.3 Minimum QC Requirements

Each preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. Note that some programs require an unspiked duplicate sample in place of or in addition to the duplicate matrix spike. Be sure to check special instructions in TALS. If clients specify specific samples for the MS and MSD, the batch may contain multiple MS/MSD pairs.

9.3.1 Method Blank (MB)

One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. Soil method blanks are prepared by taking 5 mL or 5 g of reagent water through the procedure described in Section 11. Add 1.0 g of prewashed glass beads to the blank if required by the client to better simulate a solid matrix.

The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: Criteria for the acceptance of blanks are contained within the individual analytical method SOPs.

Corrective Action: If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.3.2 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS contains reagent water that is spiked with all the analytes of interest and is carried through the entire analytical procedure. A duplicate LCS (LCSD) must be prepared when there is insufficient sample volume to perform an MS/MSD. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Add 1.0 g of prewashed glass beads to the LCS if required by the client to better simulate a solid matrix.

The spike solutions described in Section 7.8.3 are used to prepare LCSs as follows:

- Routine ICP: Add 1.0 mL of spike
- DoD ICP: Add 1.0 mL of spike to 1.0 g of glass beads
- Routine ICP-MS: Add 1.0 mL of spike
- DoD ICP-MS: Add 1.0 mL of spike to 1.0 g of glass beads

The resulting spike concentrations for each element are given in Table 2 and Table 3.

Incremental Sampling Method LCSs are spiked with 5 ml of spike.

Acceptance Criteria: Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

Corrective Action: When LCS results fail to meet control limits, the LCS and all associated samples in the batch must be re-prepared and reanalyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a second aliquot of a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Samples identified as field blanks cannot be used for MS/MSD analysis. The MS/MSD results are used to determine the effect of a

matrix on the precision and accuracy of the analytical process.

The spike solution described in Section 7.7.3 is also used to prepare matrix spikes, as follows:

- ICP: Add 1.0 mL of spike
- ICP-MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Tables II through IV. Incremental Sampling Method MS/MSD pairs are spiked with 5 ml of spike.

NOTE 1: The spike must be added after the sample aliquot but before any reagents.

NOTE 2: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD pair is not available, an LCS and LCSD are used to measure precision.

10.0 Calibration

Not applicable. This SOP addresses sample preparation only for subsequent ICP or ICP/MS analysis. Calibration of the measurement system is covered in the SOPs for the determinative methods.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Custody

11.3.1 Samples are transferred from the Sample Control group to the Metals group and the transfer is documented using the Sample Transfer function of the Internal Chain of Custody in TALS (see SOP DV-QA-0003 for details).

11.3.2 Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample.

11.4 Subsampling

11.4.1 It is not acceptable to simply collect 1.0 g off of the top of the sample. Samples must be mixed and incrementally subsampled to obtain a representative portion. At a minimum, mix by stirring with a disposable wooden spatula. If there is insufficient room in the sample container to allow for proper mixing, refer to SOP DV-QA-0023, *Subsampling*, for directions.

11.4.2 Select at least three incremental subsamples from different locations in the original sample and place them in a tared 50 mL digestion tube. The final sample weight should be between 1.0 and 1.5 g. Record the weight to the nearest 0.01 g.

11.4.3 Measure additional aliquots for QC samples required in the batch and spike as required (see Section 9 for details).

NOTE: When adding glass beads to the Method Blank and LCS digestion tubes, the nominal weight must be entered into the Initial Amount field in TALS. The true weight of glass beads should be recorded in the Notes field on the Worksheet tab in the preparation batch.

11.5 Incremental Sampling Method Digestion

For the Incremental Sampling Method approximately 10 g of sample is weighed out by the Organic Prep group following the procedure described in SOP DV-OP-0013. This pre-weighed sample is then delivered to the Metals group for digestion and analysis. The sample weight is recorded on the ISM Worksheet and attached to the incremental sampling batch in TALS. The pre-weighed aliquots are delivered in 125 mL digestion tubes which are ready for spike standards and reagents to be added. The Method 3050B digestion reagents are increased 5x to maintain the same proportions as are used for a 1-2 gram sample. When required, 10 g of glass beads are added to the Method Blank and LCS prior to digestion.

11.6 Initial Digestion Cycle with 1:1 Nitric Acid

11.6.1 Add approximately 5 mL of reagent water to each digestion tube.

11.6.2 Add 5 mL of concentrated HNO_3.

11.6.3 After all of the acid has been added to the preparation batch, gently swirl the samples to mix and then place the sample rack on the Hot Block.

11.6.4 Place a ribbed cover on each tube.

11.6.5 Heat samples to 90-95 °C, and reflux for 15 minutes without boiling.

NOTE: DO NOT ALLOW SAMPLES TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.6.6 Remove the samples from the Hot Block and allow them to cool before proceeding with the next step.

11.6.7 Record the start time, starting temperature, end time, and ending temperature in TALS.

11.7 Second Digestion Cycle Using Concentrated Nitric Acid

11.7.1 Add 5 mL of concentrated HNO_3, and replace the ribbed cover.

11.7.2 Place samples back on the Hot Block and reflux at 90-95 °C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.7.3 If brown fumes are observed, this means that material in the sample is actively being oxidized. There may not be enough HNO_3 acid to complete the oxidation, and there could be violent reaction of the sample with peroxide in the third digestion step. For that reason, it is necessary to repeat the previous two steps until no more fumes are evolved.

11.7.4 Heat the samples at 90-95 °C for 2 hours.

11.7.5 Allow the samples to thoroughly cool before proceeding.

11.8 Third Digestion Cycle Using Hydrogen Peroxide

11.8.1 Add 2 mL of reagent water to each tube.

11.8.2 Add 3 mL of 30% H_2O_2 a few drops at a time. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.8.3 Replace the ribbed cover and heat samples until effervescence subsides.

11.8.4 Allow the samples to cool.

11.8.5 Continue adding 30% H_2O_2 in 1 mL increments with warming until

effervescence is minimal or sample appearance is unchanged. If additional peroxide is added to a sample then it must also be added to the method blank and LCS.

NOTE: Do not add more than a total of 10 mL of 30% H_2O_2. If 10 mL have been added and the samples are still vigorously effervescing, document the situation with an NCM and continue with the digestion.

11.8.6 Heat the samples at 90-95 °C for 2 hours.

11.8.7 Allow the samples to cool.

11.8.8 If samples will be analyzed by ICP, continue on with the fourth digestion step using HCl in Section 11.8. If the samples will be analyzed by ICP-MS, skip the HCl digestion step and go to step 11.10.

11.9 Fourth Digestion Cycle for ICP Using Concentrated Hydrochloric Acid

11.9.1 If the samples are being prepared for ICP analysis, add 10 mL of concentrated HCl to the samples in the digestion tubes and cover with ribbed covers.

11.9.2 Reflux for an additional 15 minutes without boiling.

11.9.3 Allow the samples to cool.

11.10 Separating Undigested Solids from the Digestion Solution

11.10.1 Filter samples through Whatman 541 or equivalent fiber filters into a graduated 125 mL digestion tube whose accuracy is documented to be better than ± 3%.

NOTE: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.10.2 For samples digested by the Incremental Sampling Method use a 500 mL poly bottle that has been measured after measuring out 500 mL of DI water from a graduated cylinder.

11.10.3 Wash the original digestion tube and ribbed cover with reagent water to ensure quantitative transfer of all of the digestion solution into the new digestion tube.

11.10.4 Rinse the funnel and filter paper with reagent water to ensure complete sample transfer into the new digestion tube.

11.10.5 Re-volume sample to 100 mL with reagent water. This must be done volumetrically, rather than by weight. Record the final volume in TALS. For Multi-Incremental samples the final volume is 500 mL.

11.11 Documentation and Record Management

The following information must be recorded for each preparation batch. This information is directly entered into TALS.

- Initial sample weight and final digestion volume
- Preparation analyst and date
- Identification of all reagents and standards
- Identification of all measuring and test equipment used (e.g., balances, thermometers, pipettes)
- Glass beads lot number
- Filter paper lot number
- Digestion tube lot number
- Hot Block ID number
- Fume Hood ID number

11.12 Alternate Antimony Preparation for Analysis by ICP-MS

11.12.1 Weigh out 1.0-1.5 g soil samples according to the procedure in Section 11.3.

11.12.2 Add approximately 5 mL of reagent water to each digestion tube.

11.12.3 Spike the LCS, LCSD, MS, and MSD with 1.0 mL of the MS spike 2 standard.

11.12.4 Add 2.5 mL concentrated HNO_3 and 2.5 mL concentrated HCl to each sample and batch QC.

11.12.5 Cover each tube with a watch glass and reflux on hot block at 90-95 °C for 15 minutes.

11.12.6 Filter through Whatman 541 or equivalent filter paper into a new 125 mL digestion tube while still hot.

11.12.7 Rinse the filter and funnel with 1.25 ml of hot (~95 °C) concentrated HCl.

11.12.8 Rinse three times with hot (~95 °C) reagent water (5 mL rinses.)

11.12.9 Place the filter paper and soil residue back into the original sample digestion vessel. Add 2.5 mL concentrated HCl, cover and reflux on the hot block for 20 minutes or until paper dissolves.

11.12.10 Filter through a fresh filter into the original filtrate. Rinse three times with reagent water (5 mL rinses).

11.12.11 Bring to final volume of 100 mL with reagent water.

12.0 Calculations / Data Reduction

Not applicable. Calculations of final results are described in the determinative analytical SOPs.

13.0 Method Performance

13.1 Method Detection Limit (MDL)

An MDL must be determined for each analyte/matrix prior to the analysis of any samples. See the SOPs for the determinative analysis methods for details.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.3 Training Requirements

The group leader or supervisor is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered in the batch record (View Batch Information)until that analyst's IDOC has been approved by the QA Manager (or designee). See

requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Aqueous Acidic (Metals) - Corrosive – Waste Stream J

15.2.2 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996; Method 3050B.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

17.0 Method Modifications:

Item	Method	Modification
1	3050B	Method 3050B Section 7.1 states that a 1-2 g aliquot is to be used. The amount specified by TestAmerica Denver in this procedure is limited to 1-1.5 g in order to prevent increased instrument maintenance and sample reruns due to dilutions.

18.0 Figures, Tables, and Attachments

Table 1: Method 3050B Approved Analyte List for ICP/ICP-MS

Table 2: Soil LCS and MS/MSD Spikes for ICP

Table 3: Soil LCS and MS/MSD Spikes for ICP-MS

Attachment 1: Contamination Control Guidelines

19.0 Revision History

- Revision 11 dated 31 October 2018
  - Annual Review
  - Updated copyright information
- Revision 10 dated 31 October 2017
  - Annual Review
- Revision 9 dated 31 October 2016
  - Annual Review
  - Update the temperature heating range to 90-95°C where stated in the SOP
  - Removed the reference to AFCEE throughout SOP
  - Added current section 3.1 – reference to QAM for general definitions
  - Restructured and renumbered section 6.0
  - Added initial paragraph to section 6.0 regarding the documentation of equipment IDs
  - Revised the current sections 6.1 and 6.2 to reflect consistent verbiage and formatting as other SOPs
  - Added current section 7.6 Ultra Pure Peroxide reference
  - Added current footnote 1 to the section 8 table regarding soil preservation
  - Re-numbered previous footnote 1 to be footnote 2 to the section 8 table
  - Updated section 9.1 and subsections to reflect current practices and verbiage
  - Re-numbered Notes in section 9.3.3 to be Note 1 and Note 2
  - Added LCSD required when an MS/MSD is not available to sections 9.3.2 and 9.3.3 Note 2
  - Renumbered and updated section 11.1 and 11.2 to reflect current practices and verbiage
  - Added current section 11.2
  - Updated section 13.2 to reflect current practices and verbiage
  - Added Strontium to Table 3
  - Removed Titanium and Zirconium from Table 3
- Revision 8 dated 31 October 2015
  - Annual Review
  - Edited Sections 9.5.1 and 9.5.2 to clarify glass bead requirement
  - Added definition of reagent water
  - Updated Section 11.6.4 and 11.7.6 to reflect current practice
  - Removed Method exception 1 regarding method blank limits as it no longer

applies
- Added detail to training requirements for new analysts Section 13.3
- Added note to Section 9.5.3 regarding precision requirements
- Added note to Section 11.3 regarding recording of glass bead weights

- Revision 7 dated 31 March 2015
  - Annual Review
  - In Section 11.7.8 the section referenced was updated to 11.8
  - Updated spike standard name to MS spike 2 in Section 11.11.3
  - Formatting and grammar corrections throughout
  - Section 6.4 removed reference to calibrating digestion tubes
  - Section 6.6 changed name of filter paper to match current practice
  - Section 6.14 added to define computer systems used
  - Sections 7.7.3.1 and 7.7.3.2 combined
  - New Sections 7.7.3.2 - 7.7.3.4 added to define spikes used
  - Table of spike names and concentrations added to Section 7.7.3.4
  - Changed LIMS to TALS throughout
  - Section 8.2 changed storage temperature to ≤6 °C
  - Deleted Section 9.3, duplicated in 13.2
  - Added new Section 9.3 to address federal requirements
  - Rewrote Section 9.5
  - Changed Sections 9.6 – 9.8 to be subsections of the new 9.5
  - Rewrote Section 11.2.1
  - Removed method modification 2 because it referred to the analytical SOP
  - Created new method modification 2 explaining the 1-1.5 g sample aliquot
  - Section 11.3.2 changed required sample aliquot to 1-1.5 g to help avoid targeting
  - Rewrote Section 11.4 to define and explain the Incremental Sampling Method
  - Added new Section 11.5.3 to explain sample mixing
  - Section 11.7.5 added language to note regarding samples that require more than 10 mL of H2O2
  - Added detail into Sections 11.9.1 – 11.9.5
  - Folded Section 11.10.1 into 11.10
  - Rewrote list of data to be entered into TALS in Section 11.10
  - Rewrote Section 13.2 to match boilerplate
  - Deleted flowcharts Figures 1 and 2
  - Corrected element list in Table 2

- Revision 6 dated 31 March 2014
  - Annual Review
  - Formatting changes throughout document
  - Added to Section 11.7.5 to add additional peroxide to QC if added to samples
  - Updated section number in text to 11.8 in section 11.7.8
  - Added references for DoD QSM
  - Removed Attachment 2

- Revision 5 dated 04 March 2013
  - Section 7.7.3.1 Added DoD to the glass beads requirement
  - Section 11.11.2 Added that 5ml of water is added to the samples
  - Section 11.11.3 Changed spike name to 200.8 Cal-2
  - Updated spike level to 1.0ml in Table 3

- Updated work instructions to current revision.
- Formatting changes throughout document

- Revision 4 dated 3 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling Method throughout document
  - Section 2.0 Added reference to Incremental Sampling Method
  - Section 6.4 Added 50 mL digestion tubes
  - Added introductory statement to section 7.0 regarding reagent purity
  - Section 7.1 Updated acceptable criteria for the reagent water
  - Section 9.7.2 Added LCS Incremental Sampling Method spike amounts
  - Section 9.8.2 Added MS/MSD Incremental Sampling Method spike amounts
  - Section 11.4 Updated sample amount for Incremental Sampling Method to 1 10g aliquot
  - Section 11.9 Added Incremental Sampling Method final volume

- Revision 3.5, dated 24 August 2011
  - A note has been added to section 9.8.3 for the addition of the LCS/MS spike before reagents.

Earlier revision histories have been archived and are available upon request.

Table 1.

Method 3050B Approved Analyte List for ICP/ICP-MS

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

Table 2.

Soil LCS and MS/MSD Spikes for ICP

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (mg/L)
Aluminum	200	200	2.0
Antimony	50	50	0.5
Arsenic	100	100	1.0
Barium	200	200	2.0
Beryllium	5	5	0.050
Bismuth	200	200	2
Boron	100	100	1.0
Cadmium	10	10	0.1
Calcium	5,000	5,000	50.
Chromium	20	20	0.20
Cobalt	50	50	0.50
Copper	25	25	0.25
Iron	100	100	1.0
Lead	50	50	0.50
Lithium	100	100	1.0
Magnesium	5,000	5,000	50.
Manganese	50	50	0.50
Molybdenum	100	100	1.0
Nickel	50	50	0.50
Phosphorous	1,000	1,000	10.
Potassium	5,000	5,000	50.
Selenium	200	200	2.0
Silicon	1,000	1,000	10.
Silver	5	5	0.050
Sodium	5,000	5,000	50
Strontium	100	100	1.0
Sulfur	200	200	2.0
Thallium	200	200	2.0
Thorium	100	100	1.0
Tin	200	200	2.0
Titanium	100	100	1.0
Uranium	200	200	2.0
Vanadium	50	50	0.50
Zinc	50	50	0.50
Zirconium	50	50	0.5

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Table 3.

Soil LCS and MS/MSD Spikes for ICP-MS

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (μg/L)
Aluminum	200	200	2,000
Antimony	20	20	200
Arsenic	20	20	200
Barium	20	20	200
Beryllium	20	20	200
Cadmium	20	20	200
Chromium	20	20	200
Cobalt	20	20	200
Copper	20	20	200
Iron	200	200	2,000
Lead	20	20	200
Manganese	20	20	200
Molybdenum	20	20	200
Nickel	20	20	200
Selenium	20	20	200
Silver	20	20	200
Strontium	20	20	200
Thallium	20	20	200
Thorium	20	20	200
Tin	20	20	200
Tungsten	20	20	200
Uranium	20	20	200
Vanadium	20	20	200
Zinc	20	20	200

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Attachment 1

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with 5% HNO_3 according to the procedure described in SOP DV-IP-0005.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample cups have been found to contain lead or cobalt.
- New glassware can be a source of silica and boron.
- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Latex gloves contain over 500 ppb of zinc.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Nitroaromatic and Nitroamine Explosive Compounds by High Performance Liquid Chromatography (HPLC) [SW-846 8330A & 8330B]

Approvals (Signature/Date):

Heather Fiedler — 6/19/19 — Date
Technical Specialist

Doug Gomer — 6/19/19 — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan — 6/19/19 — Date
Quality Assurance Manager

for Roxanne Sullivan — 6/19/19 — Date
Charles Newton
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of nitroaromatic and nitroamine explosive residues by high performance liquid chromatography (HPLC) using dual columns and dual UV wavelengths. This includes analysis in water, soil, and sediment matrices. The instrumental analysis is based on EPA Methods 8330A and 8330B.

1.2 This SOP does not include the extraction procedures. For those details, please refer to DV-OP-0017, *Solid Phase Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from water samples by SW-846 3535A*, and DV-OP-0018, *The Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Soil Samples by SW-846 8330A and 8330B.*

1.3 On occasion, clients may request modifications to this SOP. Requests for modifications must be received in writing and will be communicated to the laboratory through method comments in the TestAmerica LIMS (TALS). Significant method changes require a work instruction signed by both the client and TestAmerica Denver management and the Quality Assurance Manager. (See SOPs DV-QA-001P and DV-QA-0010.)

1.4 Application of 8330A versus 8330B

1.4.1 This procedure is for analysis by either Method 8330A or 8330B. The most important differences in the two source methods are the more rigorous sample collection and preparation measures in 8330B, which are designed to produce more representative results. The more rigorous 8330B process is specifically intended to complement the multi-increment field sampling process described in Appendix A of 8330B. If multi-increment or equivalent systematic sampling processes are not employed in the field, then the extra laboratory homogenization and subsampling effort 8330B requires (see details in DV-OP-0018) may add little or no improvement in the overall precision of results.

1.4.2 For soil analysis a sample size of 2 g is used for 8330A and a sample size of 10 grams is used for 8330B.

1.4.3 8330A only describes the cyano (CN) column for confirmation. 8330B gives the option of either cyano (CN) or phenyl-hexyl columns for confirmation. Because it provides better sensitivity and resolution, TestAmerica Denver routinely confirms using the Phenomenex Luna Phenyl-Hexyl column for both methods.

1.4.4 8330B also added compounds to the potential analyte list. TestAmerica Denver offers all of the compounds shown in Appendix 1 of this SOP by both methods, except 3,5-dinitroaniline, which is only analyzed by 8330B.

1.5 Analytes, Matrix(s), and Reporting Limits

1.5.1 The list of analytes, CAS numbers, abbreviations and TA-Denver's standard reporting limits can be found in Appendix 1.

1.5.2 The working ranges of this method are as follows:

Analytes	8330A Soil (2 g prep)	8330B Soil (10 g prep)	Water
All Analytes except nitroglycerin and PETN	0.2 µg/g – 25 µg/g	0.08 µg/g – 10 µg/g	0.2 µg/L - 25 µg/L
Nitroglycerin and PETN	2.0 µg/g – 250 µg/g	0.80 µg/g – 100 µg/g	2.0 µg/L - 250 µg/L

2.0 Summary of Method

2.1 Instrument calibration is performed by external standardization using a minimum of five concentration levels.

2.2 An acetic acid and phosphate buffer in water / methanol gradient program is used for HPLC separation (see details in Section 7.8.10 and Appendix 4). Compounds are tentatively identified based on retention time and detection by the UV detector using the primary Agilent Poroshell 120 EC-C18 column. Confirmation is performed by the UV detector using a Phenomenex Luna Phenyl-Hexyl column (see Appendix 4 for instrument conditions).

3.0 Definitions

3.1 Explosives: As used in this SOP, the term "explosives" refers specifically to the analytes listed in Appendix 1. These include compounds that can be readily detonated with heat, shock, or ignition, such as nitroglycerin, RDX, and TNT. It also includes production by-products and degradation products of true explosives.

3.2 Definition of terms used in this SOP may be found in the Glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) and in Policy QA-DV-003P.

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2 Contamination by carryover can occur when a low-concentration sample is analyzed immediately following a high-concentration sample. This potential is minimized by continuously flushing the needle with solvent. If contamination is suspected, the sample should be re-aliquoted and re-analyzed.

4.3 Co-elution of target analytes with non-target analytes can occur, resulting in false positives or biased high results.

4.4 Co-elution between target analytes can occur when high concentrations of individual compounds are present in samples, see Section 12.2.3.5 for details.

4.5 The inclusion of vegetation is not recommended given the nature of the detector and different uses the data will potentially support (USACE comment – Issue #306 TA Denver Audit Database; DOD/DOE QSM 5.0 and 5.1 both state that vegetation should be excluded).

4.6 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All samples expected to contain tetryl should not be exposed to temperatures above room temperature. (Reference: EPA Method 8330A & 8330B, Section 4.3) Elution solvent for the SPE cartridges is also acidified to help preserve tetryl in sample extracts.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual (RSM) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 If a sample is expected to have an explosive concentration ≥ 2% (but less than 10%), the EH&S Coordinator and Group Leader shall be notified before any work is performed. Additional safety precautions may be implemented as required due to high concentrations of explosives.

WARNING: Soil samples with explosive concentrations greater than 2% cannot be accepted by the laboratory unless they have a moisture content of 25% or greater. Under no circumstances shall a soil sample with an explosive concentration greater than 10% be accepted by the laboratory.

5.1.3 Soil samples with high concentrations (between 2 and 10%) of explosives should not be ground using a mortar and pestle. Visual observation of a soil samples is important prior to grinding samples. Any samples containing metal fragments, powders, waxy appearing pieces, or other suspicious material should be brought to the attention of the Group Leader and the EH&S Coordinator before proceeding with the procedure.

Bypassing the grinding step and proceeding to solvent dilution is an alternative for samples that are determined to be unsafe to grind.

5.2 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not contain all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
Acetonitrile	Flammable Poison	40 ppm – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Glacial Acetic Acid	Corrosive Poison Flammable Liquid and Vapor	10 ppm - TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
Methanol	Flammable Poison Irritant	200 ppm - TWA	A slight irritant to the mucous membranes. Toxic effects are exerted upon the nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause the skin to be become dry and cracked. Skin absorption can occur, symptoms may parallel inhalation exposure. Irritant to the eyes.
Phosphoric Acid	Corrosive	1 ppm - TWA	Ingestion can cause severe burns to the throat, mouth, and stomach, abdominal pain and nausea. Severe exposures by ingestion can lead to shock, circulatory collapse, and death. Inhalation is not an expected hazard unless misted. Corrosive, contact with skin or eyes can cause redness, pain, severe burns, blurred vision, and permanent eye damage.
Sodium hydroxide	Corrosive Poison	2 mg/m^3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.

MATERIAL (1)	HAZARDS	EXPOSURE LIMIT (2)	SIGNS AND SYMPTOMS OF EXPOSURE
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 HPLC System

HPLC, equipped with a pump capable of achieving 6,000 psi, a 100 µL loop injector, and a Diode Array Detector (DAD) or Multi-Wavelength Detector (MWD), Hewlett Packard Model 1100, or equivalent.

6.1.2 Primary Column: Reverse phase HPLC column, Agilent Poroshell 120, EC-C-18, 4.6 mm x 150 mm (2.7 µm) or equivalent.

6.1.3 Confirmation Column: Phenomenex Luna Phenyl-Hexyl reverse phase HPLC column, 15 cm x 4.6 mm (3 µm) or equivalent.

6.1.4 Hewlett Packard HPLC Chem Station for instrument control.

6.2 Supplies

6.2.1 Glass vials, various sizes.

6.2.1.1 Amber glass, 8.0 mL and 12.0 mL, with Teflon-lined screw caps, for the storage of standards.

6.2.1.2 Crimp-top vial with caps for analysis, 1.8 mL.

6.2.2 Disposable pipettes, used for non-quantitative transfers only.

6.2.3 Volumetric flasks, various sizes.

6.2.4 Hamilton syringes, various sizes.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Stock Standards

7.1.1 Stock standards are purchased as certified solutions or prepared from 100%, neat materials. Stock standard solutions are stored at -10 °C to -20

°C, or per vendor instructions. All stock standards must be protected from light and should be brought to room temperature before using.

7.1.2 Stock standard solutions must be replaced after 1 year or sooner if comparison with check standards prepared from an independent source indicates a problem. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule.

7.1.3 3,5-Dinitroaniline is purchased at a concentration of 100 µg/mL in acetonitrile, equivalent to the High-Level Calibration Mix. This standard is added directly to the Intermediate Level Calibration Standard. (Section 7.4).

7.1.4 PETN and Nitroglycerin are purchased as separate individual standards at a concentration of 1,000 µg/mL in acetonitrile. These standards are added directly to the Intermediate Level Calibration Standard (Section 7.4).

7.1.5 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene are purchased as separate individual standards at a concentration of 100 µg/mL in acetonitrile, equivalent to the High-Level Calibration Mix. These standards are added directly to the Intermediate Level Calibration Standard (Section 7.4).

7.1.6 MNX, DNX, TNX are purchased from Ultra Scientific as a custom standard at 100 µg/mL. These compounds are in a separate standard called 8330 DMT in TALS. This is the only known source of MNX, DNX, and TNX and we do not currently have a second-source standard for these compounds.

7.2 Volume Measurements for all Standards Preparation

The volume of stock and intermediate standard solutions used in subsequent dilutions is measured using Hamilton syringes appropriate for the volume being measured and accurate to ± 2%. Standards are prepared either by (1) using a syringe to measure the standard solution and bringing to volume with the appropriate solvent in a Class A volumetric flask, or by (2) measuring the volumes of both the standard solution and the solvent using a calibrated syringe or Class A pipette and combining them in a vial.

7.3 High-Level Calibration Mix, Prepared from Stock Standards

This high level standard must be replaced every 1 year or sooner if comparison with check standards prepared from independent sources indicate a problem.

A solution is prepared to contain most standard analytes at a concentration of 100 µg/mL each in acetonitrile (see details in standards database instructions). Nitroglycerin, PETN, 3,5-Dinitroaniline, 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene are purchased as separate stock standards, and are not included in this mixture. They are added to the intermediate level standards in Section 7.4.

7.4 Intermediate Level Calibration Standards

8330IntermStk: Prepare a 20 µg/mL solution (nitroglycerin and PETN at 200 µg/mL) from the high-level calibration mix (see Section 7.3) by diluting 1.0 ± 0.02 mL of the High-Level Calibration mix along with 1.0 ± 0.02 mL each of stock standards of nitroglycerin and PETN to a final volume of 5.0 mL in acetonitrile. The shelf life of this material is 6 months.

8330_ADDs: Prepare a 20 µg/mL solution from stock standards by diluting 1.0 ± 0.02 mL each of stock standards of 3,5 -dinitroaniline, 2,4-diamino-6-nitrotoluene, and 2,6-diamino-4-nitrotoluene to a final volume of 5.0 mL in acetonitrile. The shelf life of this material is 6 months.

8330 DMT: Prepare a 20 µg/mL solution from stock standard by diluting 1.0 ± 0.02 mL of MNX, TNX, DNX stock to a final volume of 5 mL. The shelf life of this material is 6 months.

7.5 Working Level Standards for Calibration Curve

Prepare calibration standards by diluting the intermediate standard solutions as shown in the table below using the 75%:25% (v/v) acidic water:ACN solution (described in Section 7.8.7). These standards must be prepared fresh on the day of calibration and refrigerated if not used immediately. All volumes are measured using the appropriately sized Hamilton syringe.

7.5.1 On the primary (C18) column, the 8330IntermStk, 8330_ADDs and 8330 DMT standards must be calibrated using separate sets of calibration standards due to co-elutions with other target compounds. The intermediate standards are prepared at the same concentration; therefore, the calibrations are made with the same volumes of intermediate stock and solvent in the following table.

7.5.2 On the confirmation (phenyl-hexyl) column, the intermediate full-list and intermediate 3,5-dinitroaniline stocks can be combined into the same calibration standards and follow the “Confirmation (Phenyl-Hexyl) Column Calibration” recipes in the following table. 8330 DMT standards must be calibrated using separate standards due to co-elutions with other target compounds.

Recommended Calibration Levels

Calibration Level	Final Concentration (µg/mL)		Primary (C18) Column Calibration		Confirmation (Phenyl-Hexyl) Column Calibration	
	Standard Analytes	NG & PETN	Vol. Intermediate (µL)	Vol. Solvent (µL)	Vol. EACH Intermediate (µL)	Vol. Solvent (µL)
8	2.5	25.0	125 ± 1	875 ± 9	125 ± 1	750 ± 9
7	1.0	10.0	50 ± 0.5	950 ± 10	50 ± 0.5	900 ± 10
6	0.7	7.0	35 ± 0.4	965 ± 10	35 ± 0.4	930 ± 10
5	0.4	4.0	20 ± 0.2	980 ± 10	20 ± 0.2	960 ± 10
4*	0.25	2.5	12.5 ± 0.1	988 ± 10	12.5 ± 0.1	975 ± 10
3	0.1	1.0	5 ± 0.05	995 ± 10	5 ± 0.05	990 ± 10
2	0.05	0.5	2.5 ± 0.02	998 ± 10	2.5 ± 0.02	995 ± 10
1	0.02	0.2	20 ± 0.1 µL of Level 7	980 ± 10	20 ± 0.1 µL of Level 7	980 ± 10

- Level 4 concentration is used for the daily and continuing calibrations.
- Nitroglycerine and PETN are 10X higher than the other analytes in all calibration levels.

7.6 Extractions Standards

7.6.1 LCS Spike Solution

The LCS spike solution is prepared at a working level concentration of 10 µg/mL (nitroglycerin and PETN at 100 µg/mL) in acetonitrile. This standard is stored in a freezer at –20 °C to –10 °C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible. This standard contains all explosives target compounds except 3,5-dinitroaniline, 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene.

7.6.2 3,5-Dinitroaniline LCS Solution

The 3,5-DNA LCS solution is prepared at a working level concentration of 10 µg/mL in acetonitrile. This standard is stored in a freezer at –20 °C to –10 °C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible. This standard is used only for method 8330B and is spiked into separate LCS and MS/MSD samples. This compound cannot be completely resolved from tetryl and nitrobenzene on the primary column.

7.6.3 MNX, DNX, TNX LCS solution

The 8330 OP DMT LCS solution is prepared at a working level concentration of 10 µg/mL in acetonitrile. This standard is stored in a freezer at –20 °C to –10 °C and given a six-month expiration date.

7.6.4 Diamino LCS Solution

The Diamino LCS solution is prepared at a working level concentration of 10 µg/mL in acetonitrile of 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene. This standard is stored in a freezer at –20 °C to –10 °C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible. This standard is only added when these compounds are specifically requested by the client. They are added to a separate LCS/MS/MSD. They are spiked into the same LCS/MS/MSD as 3,5 DNA if the client requests all three compounds.

7.6.5 Working Level Surrogate (1,2-Dinitrobenzene) Solution

The 8330 surrogate solution is prepared at a working level concentration of 10 µg/mL in acetonitrile. This standard is stored in a freezer at –20 °C to –10 °C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible.

7.7 Second Source Initial Calibration Verification Solution

The second source standard must be obtained from a different source than the standards used for initial calibration. This standard is used to verify the accuracy of the calibration standards.

NOTE: There is currently no second source available for MNX, DNX, and TNX.

7.7.1 Working-Level Second Source Mix

Prepare a solution containing all compounds except 3,5-dinitroaniline, 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene at a concentration of 0.40 µg/mL (nitroglycerin and PETN at 4.0 µg/mL). A separate solution containing 3,5-dinitroaniline, 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene is prepared at a concentration of 0.40 µg/mL when analyzing on the primary (C18) column; the standards can be combined for analysis on the confirmation (phenyl-hexyl) column. These solutions are prepared using the 75%:25% (v/v) acidic water:ACN solution (described in Section 7.8.7). These standards must be prepared fresh on the day of calibration. All volumes are measured using the appropriately sized Hamilton syringe.

7.8 Reagents

7.8.1 Reagent Water

For method blanks and laboratory control samples reagent water is generated by an ELGA water purification system. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.8.2 HPLC Grade Water

7.8.3 Acetonitrile, CH_3CN (ACN) - HPLC grade

7.8.4 Methanol - HPLC grade

7.8.5 Glacial Acetic Acid – Reagent Grade

7.8.6 85% Phosphoric acid, H_3PO_4 – Reagent Grade.

7.8.7 Acidified Water (75%): Acetonitrile Solution (25%)

TALS Reagent: 8330AcidH2O

Take 250 mL of acetonitrile (ACN), and bring to 1.0 L with Elga or HPLC-grade water. Acidify the solution to a pH of approximately 3 by adding 20 drops (1 mL) of 85% phosphoric acid (H_3PO_4).

7.8.8 Acidified Calcium Chloride, $CaCl_2$ Solution, 5 g/L

TALS Reagent: CaCL2 Sol

Place 5 ± 0.05 g of reagent grade $CaCl_2$ into a one-liter volumetric flask containing approximately 500 mL of reagent water. Swirl the solution until the $CaCl_2$ is dissolved. Add approximately 1 mL of 85% H_3PO_4 to acidify the solution and bring to volume with reagent water.

7.8.9 Sodium Phosphate Buffer Stock

TALS Reagent: 8330bufferstk

Slowly add 115.29 grams of 85% phosphoric acid (molecular weight = 97.9924 g/mol) to approximately 500 mL of water in a 1 L beaker. Place a stir bar in the beaker and the beaker in an ice bath on top of a magnetic stirrer. Slowly add 150 mL of 10 M (10 M = 10 N) sodium hydroxide, allowing time for the mixture to cool down between additions. Transfer to a 1 L volumetric flask and bring to volume with Elga or HPLC-grade water. The final pH of this solution should be 7.2.

7.8.10 Buffer Eluents for Analysis:

Make up the HPLC eluents for each column as described in Sections 7.8.10.1 and 7.8.10.2. The pH of the working eluents *must* be modified by the analyst by changing the volume of glacial acetic acid added to ensure compound resolution. This is particularly necessary when the column is replaced, to ensure that picric acid does not co-elute with any other target compound. Increasing the concentration (or volume added) of glacial acetic acid will result in greater retention of picric acid. The eluent can only be adjusted at the start of an initial calibration, not with the CCVs. The calculated retention time windows should account for the drift of any specific analyte.

7.8.10.1 Working Eluent for Primary (C18) Column:

Combine 1 L of water, 1 mL of Sodium Phosphate Buffer Stock solution (Section 7.8.9), and 50 µL of Glacial Acetic acid to adjust the pH of the buffer to approximately 6.5. Make fresh at least weekly or more often as needed.

7.8.10.2 Working Eluent for Confirmation (Phenyl-Hexyl) Column:

Combine 1 L of water, 2 mL of Sodium Phosphate Buffer Stock solution (Section 7.8.9), and enough glacial acetic acid (approximately 65 - 95 µL) so that picric acid does not co-elute with MNX or RDX. The pH of the buffer should be adjusted to approximately 6.5. Make fresh before each run.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous Samples

Water samples should be collected in duplicate 500 mL amber glass bottles with Teflon-lined caps.

8.2 Soil and Sediment Samples

8.2.1 For method 8330A, soil samples should be collected in eight-ounce wide mouth jars with Teflon-lined caps.

8.2.2 For method 8330B, it is not uncommon to receive samples of 1 kg or more. Samples may be shipped in wide mouth jars or clean plastic bags.

8.3 Samples and sample extracts must be stored in amber glass containers at ≤ 6 °C from the time of collection through analysis, except during drying.

8.4 Soil and sediment samples should be air dried at ambient temperature until dry enough to sieve. See DV-OP-0018 for details. Once the sample is air dried, the sample can be stored at room temperature.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Amber glass	1 Liter (2 x 500 mL)	Cool; ≤ 6 °C	7 Days to Extraction 40 Days to Analysis	SW846 8330A
Soil	Glass / plastic	4 grams (8330A) / up to 1 kg (8330B)	Cool; ≤ 6 °C	14 Days to Extraction 40 Days to Analysis	SW846 8330A/B
Note: If DNX, MNX, or TNX are requested analytes, additional volume will be required for MS/MSD analysis.					

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for the DoD/DOE QSM (5.0 – 5.3) unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each column on which the associated samples are analyzed. See QC Policy DV-QA-003P for further details.

9.3 Method Blank (MB)

A method blank (MB) must be prepared and analyzed with each batch of samples. The MB consists of reagent water for aqueous samples, and Ottawa sand for soil samples, with surrogates added. The MB is created at the time of extraction after the samples have been dried, sieved, and ground and is then carried through all extraction and analysis steps. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data. See Section 9.9 for the use of the grinding blank for Method 8330B solids samples.

Acceptance Criteria: The MB must not contain any analyte of interest at or above one-half the RL or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

MBs are evaluated on each column on which associated samples are analyzed, when confirmation data are required. If there is a detection on either column in the MB, then detections for that target compound are suspect in associated samples. See Appendix 4 for guidance on interpretation of confirmation data to assess acceptance of the MB.

Corrective Action: If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize, or eliminate the problem. Reanalyze and/or reprepare all samples associated with a failed method blank.

If the MB acceptance criteria are not met and re-preparation and reanalysis are not possible, then the sample data associated with the unacceptable MB must be qualified. This nonconformance must be addressed in the project or case narrative and the client must be notified.

9.4 Laboratory Control Sample (LCS)

One LCS must be analyzed with each batch of samples (up to 20 samples). The LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. For water samples, the LCS is prepared by spiking the analytes of interest into reagent water. For soil samples, the LCS is prepared by spiking the analytes of interest into Ottawa sand. The LCS is created at the time of sample extraction after the samples have been dried, sieved and ground. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be analyzed on the confirmation column. If there is insufficient volume to prepare an MS/MSD, and LCSD must also be prepared and analyzed.

Acceptance Criteria: The LCS recovery for each spiked analyte must be within established control limits. Laboratory default control limits are calculated as ± 3 standard deviations around the mean of historical data, as described in SOP DV-QA-003P. For DOD/DOE work, QSM limits are applied unless project specific limits are requested by the client. When no QSM limits are available, laboratory historical limits are applied. Control limits are maintained in TALS.

In accordance with the TNI 2009 Standard a marginal exceedance within ± 4 standard deviations is allowed for one of the analytes. This is based on the number of analytes typically spiked for this method, which is between 11 and 30. These acceptance criteria may be superseded by project-specific limits, as applicable.

Corrective Action: If recoveries for all spiked analytes are not within the acceptance limits, including the one allowed marginal exceedance, the analytical system is out of control and corrective action must occur. Generally this requires re-extraction and reanalysis of all associated samples. If the LCS is biased high and all associated samples are ND (not detected), it may be possible to report results with an NCM (see requirements for individual programs and clients).

9.5 Matrix Spike Sample (MS) and Matrix Spike Duplicate (MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The soil matrix spikes are created at the time of extraction. Spikes and surrogate compounds are added after the sample has been dried, sieved, and ground. One MS/MSD pair must be processed for each preparation batch (up to 20 samples). The MS/MSD results are used to determine

the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

Acceptance Criteria: The spike recoveries must fall within established control limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. LCS limits are used for MS/MSD evaluation. Control limits are maintained in the LIMS. For DoD/DOE work, QSM limits are applied if available unless project specific limits are requested by the client. The RPD limit for DoD/DOE QSM work is 30% for Method 8330A and 20% for Method 8330B.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the

reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.6 Surrogates

Each calibration standard, field sample, and QC sample is spiked with the surrogate compound 1,2-dinitrobenzene. The surrogate is added to field samples and QC samples before the first extraction step for all matrices.

Acceptance Criteria: The recovery of the surrogate must fall within established statistical limits, which are based on historical data.

Corrective Action: If recoveries for surrogates in blanks or LCSs are outside of the control limits, check for calculation or instrument problems and reprepare and reanalyze the associated samples.

For samples with failing surrogate recoveries the decision to reanalyze or flag the data should be made as required by the project.

If matrix interference is obvious from observation of chromatograms or other objective evidence, reanalysis is unlikely to produce new or more useful information. If the matrix interference is not obvious from the initial analysis, it is only necessary to reprepare and reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, so long as the extraction/instrument system is proven to be working properly.

9.7 Sample Duplicate

NOTE: Method 8330B requires the preparation of both a soil duplicate and a soil triplicate. See Section 9.8.

Although not typically required for organic analyses, a duplicate sample may be required for project-specific quality control. In this case, a sample duplicate is a second aliquot of one of the samples in the batch. Field blanks cannot be used for duplicate testing. The results for duplicates are reported separately, and cannot be averaged when reporting results. Sample duplicate results are used to evaluate the precision of the method. As such, results should be greater than or equal to the RL for a valid statistical comparison.

Acceptance Criteria: The RPD between the sample and the sample duplicate results must be less than the established limit.

Corrective Action: Results for samples that do not meet acceptance limits, particularly if due to difficulties in subsampling, shall be discussed in the final report case narrative, after client notification and agreement.

9.8 Sample Replicates

Replicate analyses are not part of the laboratories standard quality control samples. Method 8330B requires the preparation and analysis of sample duplicate and triplicate for soil samples ground by the ring and puck or ball mill. The lab will extract triplicate aliquots after grinding on the client designated sample. If a sample is not designated by the client, the lab will select the sample. The lab will determine the %RSD as defined below. Results for the %RSD as well as the individual replicate results will be reported to the client. The %RSD for results above the LOQ must be ≤ 20%, including DoD/DOE samples.

The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{s}{\bar{C}} \times 100\% \qquad \textit{Equation 1}$$

Where s is the standard deviation of the average concentration ($\overline{C}$) and is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(C_i - \overline{C})^2}{n-1}} \qquad \textit{Equation 2}$$

In the event that the laboratory is requested to perform the evaluation of field replicate precision, three field replicates designated by the client will be processed through the entire homogenization and extraction steps. The %RSD for these replicates will be calculated as indicated above and reported to the client.

9.9 Grinding Blank (GB)

Refer to SOP DV-OP-0018 for details on how the grinding blanks for soils by method 8330B are prepared. The laboratory composites the grinding blanks to prepare and analyze one grinding blank per batch. The DOD/DOE QSM requires only one grinding blank per batch of samples, processed after the LCS (if ground) or after a client identified sample with known contamination, or at the end of the batch.

Acceptance Criteria: The grinding blank must not contain any analyte of interest at or above one-half of the RL or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

Corrective Action: Per the method, the entire client sample is ground initially. Therefore, it is not possible to re-grind client samples if the grinding blank fails. Sample results will be reported with an NCM (see requirements for individual programs).

If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. The potential carry-over between samples associated with a contaminated grinding blank producing positive results for the same contaminant must be described in a non-conformance memo and discussed in the final report case narrative.

9.10 Grinding LCS (LCSSRM)

Refer to SOP DV-OP-0018 for details on how the grinding LCS for soils by method 8330B is prepared. The grinding LCS is spiked by an outside vendor, and then ground with the associated samples. One grinding LCS per batch is required.

Acceptance Criteria: The grinding LCS recovery for each spiked analyte must be within established control limits. Control limits are

maintained in TALS.

Corrective Action: Per the method, the entire client sample is ground initially. Therefore, it is not possible to re-grind client samples if the grinding LCS fails. Sample results will be reported with an NCM (see requirements for individual programs).

If the surrogate compound recovers below control limits, or every compound recovers outside of control limits, but at approximately the same percentage, this could be an indication of a bad extraction (post-grinding). Associated samples will be sent for re-extraction and re-analysis.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Extraction of Water Samples – *Please reference DV-OP-0017 for details*

Samples are extracted using a 500 mL initial volume and are concentrated to a 5 mL final volume.

10.4 Extraction of Soil Samples – *Please reference DV-OP-0018 for details*

10.4.1 Samples for method 8330A are extracted using a 2 g initial weight and delivered to the analytical group with a 10 mL final volume. Samples for method 8330B are extracted using a 10 g initial weight and delivered to the analytical group with a 20 mL final volume.

10.4.2 TALS prep batches for soils will show a final volume of 20 mL for 8330A extracts, and 40 mL for 8330B extracts. These final volumes include the 1:1 dilutions with the acidified calcium chloride done by the analyst prior to instrument analysis. (See Section 10.5.2.)

10.5 HPLC Analysis

10.5.1 HPLC Startup: All electronic equipment should be allowed to warm up for 30 minutes. During this period, at least 15 void volumes of mobile phase are passed through the column. Continue until the detector's baseline has

stabilized.

10.5.2 Prior to analysis, soil extracts (or a portion of the extract) must be diluted exactly 1:1 with the acidified calcium chloride solution that is described in Section 7.8.8.

10.5.3 Analyze the samples using the chromatographic conditions given in Appendix 5. All positive measurements above the method detection limit observed on the primary C18 column are confirmed by injection of the sample extract onto the confirmation Phenyl-Hexyl column. The MB and LCS must be analyzed on the confirmation column if samples are analyzed on that column. The MS and MSD must be analyzed on the confirmation column if the parent sample is analyzed on the confirmation column. Many EDDs require the parent and MS/MSD results be from the same analytical batch. For DoD/DOE work, calibration and QC criteria are the same as for primary column analysis. QC must pass on both columns when confirmation analysis is performed for DoD or DOE.

10.5.4 Analytes are introduced by direct injection of the extract. Samples, standards, and QC samples must be introduced using the same procedure.

10.5.5 It has been demonstrated that water samples with total concentrations of 16,000 µg/L can yield low recoveries due to saturation of the extraction cartridge. The client should be contacted to determine if re-extraction using a smaller sample aliquot size is required for samples with concentrations in this range. The low extraction recovery may meet the client's action limit, such that a re-extraction may not be necessary.

10.5.6 Analytical Sequence

10.5.6.1 The analytical sequence starts with either an initial calibration or a Continuing Calibration Verification (CCV). If the sequence begins with a CCV, the center of the retention time window is set based on the initial CCV in the sequence. Do not reset the retention times with the bracketing CCV.

10.5.6.2 The CCV includes analyzing standards that contain all target analytes. If 3,5-dinitroaniline, 2,4-diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene are not target compounds, it is not required to analyze a CCV for these analytes.

10.5.6.3 If there is a break in the analytical sequence greater than 12 hours since the analysis of a CCV standard, a new CCV standard must be analyzed before proceeding with the sequence.

10.5.7 Retention Time Windows

10.5.7.1 Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day for a three-day period. Calculate the standard deviation of the three

retention times for each analyte (relative retention times may also be used). The width of the retention time window for each analyte is defined as ± three times the standard deviation.

NOTE: Determination of retention time windows using the 72-hour study is required for DoD/DOE work. A retention time window report can be generated in the Control Chart Module in TALS.

10.5.7.2 The chromatograms in Appendices 5 and 6 summarize the estimated retention times on both the C18 and Phenyl-Hexyl columns for many of the compounds analyzed using this method.

10.5.7.3 The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid-point of the initial calibration, and each subsequent initial CCV (i.e., the CCV that begins the analytical sequence). The widths of the windows will remain the same until new windows are generated following the installation of a new column.

10.5.7.4 If the retention time window, as calculated above, is less than ± 0.035 minutes for the C18 column or less than ± 0.07 minutes for the phenyl hexyl column, use ± 0.035 or ± 0.07 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

10.5.7.5 The laboratory must calculate new retention time windows each time a new column is installed or at least annually. Until these standards have been run on the new column, the retention time windows from the old column may be used, but updated with the retention times from the new initial calibration.

10.5.8 Daily Retention Time Windows

The center of the retention time window is adjusted to the retention time of each analyte, as determined in each initial calibration or each initial CCV.

Note: Chromatographic conditions, including the exact makeup of the eluent are determined at the time of the initial calibration and shall not be changed until the next initial calibration.

Corrective Action:

If there are shifts in retention times for target compounds between CCVs that are outside the established retention time window (see Section 10.5.7), all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

- The retention time of that compound in the standard must be within the retention time range equal to twice the original window, as determined by the opening CCV of a bracket, and
- The retention time of the compound must be shifted in the same direction as the surrogate and by approximately the same amount.

If these two conditions are met, reset the window and reprocess the data.

10.6 Calibration Range and Dilutions

10.6.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (between the CCV and highest standard) of the calibration range.

10.6.2 Samples that are analyzed immediately following a sample with an unusually high concentration of explosives must be evaluated for carryover. The potential for carryover is minimized in the analytical system by continuously flushing the HPLC needle with solvent. If contamination is suspected, the sample should be re-aliquoted and re-analyzed.

10.6.3 Guidance for Dilutions Due to Matrix Interference:

It may also be necessary to dilute samples because of matrix interferences. If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

10.6.4 Reporting Dilutions

Some projects require reporting of multiple dilutions (check method comments in TALS). In other cases, the lowest dilution with no target compounds above the calibration range will be reported. In general, a maximum of two dilutions will be reported; one at the lowest dilution and one in which the most concentrated target analyte is in the upper half of the calibration range.

10.7 Instrument Maintenance and Troubleshooting

10.7.1 Minor instrument maintenance may include back flushing the column, changing the guard cartridge, and changing the frit on the front end of the column.

10.7.2 The solvent channel on which the buffer is run should be rinsed weekly with pure water followed by pure methanol and finally pure water again before reloading the buffer in order to prevent the buildup of salts and prevent bacterial growth in the system.

10.7.3 A cleanup method is provided in the instrument software to remove the buffer from the column. It should be run as the last injection of any run sequence. The buffer solution should not be left on the column for extended periods when the instrument is not in use or decreased column lifetime will be observed.

10.7.4 Noisy baseline, particularly noticeable in RL level standards and MDLV samples, is normally due to a noisy UV Lamp. If the noise is sufficient to interfere with the quantitation of these samples, the lamp should be replaced and the instrument recalibrated. Less commonly, noisy baselines are the result of dirty flow cell windows, which should be cleaned or replaced according the manufacturer's instructions.

10.7.5 Unstable retention times are normally due to a malfunction somewhere in the flow path of the instrument. Likely sources are the active inlet valve, outlet ball valve, multichannel gradient valve or purge valve. Dirty solvent inlet filters can starve the pump and may also result in unstable retention times.

11.0 Instrument Calibration

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

11.2 Instrument QC

11.2.1 External calibration is used for this analysis. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Appendix 2.

11.2.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include a new column and any changes in instrument operating parameters (including solvent flows, replacement of a detector lamp, replacement of the flow cell windows, etc.).

11.2.3 With the exception noted in Section 11.2.4 below, it is NOT acceptable to remove points from the calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. At least 5 points must be included in a linear calibration curve and at least 6 points for a second order calibration curve.

11.2.4 A mid-level point may be removed from the calibration only if the reason can be clearly documented (i.e., a broken vial or an injection error). Due to the difficulty involved in properly documenting these errors, removing mid-points should be avoided if at all possible. The curve must also still satisfy the requirements for total numbers of points, five levels must remain in the

calibration for a linear model and six for a second order model. The documentation must be retained with the initial calibration.

11.3 Initial Calibration

Calibrations are modeled either as average response factors or as calibration curves, using a systematic approach to selecting the optimum calibration function in order as follows. When calibration acceptance criteria cannot be met for a model, appropriate corrective action must be taken. This may include processing the data using another model, instrument maintenance and or re-preparation of standards followed by recalibration.

11.3.1 The following requirements must be met for any calibration to be used:

11.3.1.1 Response must increase with increasing concentration.

11.3.1.2 Calibration curves will not be forced through the origin.

11.3.1.3 The absolute value of the intercept of the curve at zero response should ideally be less than the MDL for the analyte. At a minimum the intercept must be less than ½ the on-column equivalent of the reporting limit.

11.3.2 Linear Calibration Using Average Calibration Factors

11.3.2.1 External standard calibration using average calibration factors involves the comparison of instrument response (e.g., peak area or peak height) from the target compounds in the sample to the responses of the target compounds in the calibration standards. The ratio of the detector response to the concentration of target analyte in the calibration standard is defined as the calibration factor (CF), as follows:

$$CF = \frac{R_x}{C_s} \qquad \textit{Equation 3}$$

Where: R_x = Response for analyte
C_s = Concentration in calibration standard, μg/mL

11.3.2.2 For each target analyte, calculate the average calibration factor ($\overline{CF}$) as follows:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 4}$$

Where: n = Number of calibration levels
CF_i = Calibration factor for the i^{th} level

11.3.2.3 The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor.

11.3.2.4 The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} x100\% \qquad \textit{Equation 5}$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \textit{Equation 6}$$

11.3.2.5 To calculate the concentration in an unknown sample extract, the equation is solved for concentration, resulting in the following equation:

$$C_{ex} = \frac{R_x}{\overline{CF}}$$ *Equation 7*

Where: C_{ex} = Extract analyte concentration, µg/mL
R_x = Response for analyte
$\overline{CF}$ = Average Calibration Factor

11.3.3 Average Calibration Factor Evaluation

Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD of the average response factor must be < 20%. (15% for Method 8330B for DOD/DOE QSM projects). Also examine the residuals, especially for the high points versus the fitted function. If the residual values are large, a linear regression should be considered.

Corrective Action: If the RSD is > 20% (or > 15% for Method 8330B for DOD/DOE projects), average response factor cannot be used and least-squares linear regression should be attempted.

11.3.4 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x).

The weighting used is the reciprocal of the concentration or the reciprocal of the square of the concentration. The regression produces the slope and intercept terms for a linear equation in the following:

$$R_x = m_1(C_s) + b$$ *Equation 8*

Where: C_s = Concentration in calibration standard, µg/mL
R_x = Response for analyte
b = y - Intercept
m_1 = Slope

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equations, where C_{ex} is the concentration of the target analyte in the unknown sample:

$$C_{ex} = \frac{[R_x - b]}{m_1}$$ *Equation 9*

Where: C_{ex} = Extract analyte concentration, µg/mL
R_x = Response for analyte
b = y - Intercept
m_1 = Slope

11.3.5 Evaluation of the Linear Least-Squares Regression Calibration Function:

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations.

Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria:

11.3.5.1 Examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

11.3.5.2 The linear regression must have a correlation coefficient (r) ≥ 0.995 (r^2 ≥ 0.990).

11.3.6 Non-linear Calibration Using a Second-Order Equation

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$R_x = m_2(C_s)^2 + m_1(C_s) + b$$ *Equation 10*

Where: C_s = Analyte concentration in calibration standard, µg/mL
R_x = Response for analyte
m_2 = Curvature
m_1 = Slope
b = y - Intercept

To calculate the concentration in an unknown sample extract, the roots of the quadratic equation are solved for:

$$C_{ex} = \frac{-m_1 \pm \sqrt{(m_1)^2 - 4(m_2)(b - R_x)}}{2m_2}$$ *Equation 11*

Where: C_{ex} = Extract analyte concentration, µg/mL
R_x = Response for analyte
m_2 = Curvature
m_1 = Slope
b = y – Intercept

11.3.7 Evaluation of Second-Order Regression Calibration:

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria:

11.3.7.1 Second-order regressions should be the last option, and note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

11.3.7.2 The coefficient of determination (COD, r^2) must be ≥ 0.990.

11.3.7.3 The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).

11.3.7.4 The distribution of concentrations is adequate to characterize the curvature.

11.4 Calibration Verification

11.4.1 Initial Calibration Verification (ICV)

A second-source verification standard must be analyzed with each initial calibration. The calculated concentration of the analytes in this standard may not be greater than 20% different from the calibration standard (15% for DoD/DOE QSM 5.0 and 5.1 for Method 8330A).

11.4.2 Continuing Calibration Verification (CCV)

The working calibration curve or RF must be verified by the analysis of a mid-point continuing calibration standard at the beginning of the analysis sequence, after every 10 samples, and at the end of the analysis sequence.

Acceptance Criteria:

Results are acceptable for any individual compound if the %D (percent difference between the standard and measured values of the CCV standard) is ≤ 20%. (≤ 15% for DoD/DOE QSM 5.0 and 5.1 for Method 8330A). TestAmerica discourages the use of grand mean for method 8000B. The use of grand mean is not acceptable for Method 8000C (required by Arizona) or 8000D (required by North Carolina, South Carolina and West Virginia).

NOTE: In order to comply with DoD/DOE QSM requirements, the use of the grand mean is not acceptable (refer to policy DV-QA-024P). Results are acceptable for individual compounds if the %D is:

8330A		8330B	
QSM 4.2	%D ≤ 15%	QSM 4.2	%D ≤ 20%
QSM 5.0	%D ≤ 15%	QSM 5.0	%D ≤ 20%
QSM 5.1	%D ≤ 15%	QSM 5.1	%D ≤ 20%
QSM 5.2	%D ≤ 15%	QSM 5.2	%D ≤ 20%
QSM 5.3	%D ≤ 15%	QSM 5.3	%D ≤ 20%

Corrective Action:

If the percent difference for any analyte falls outside of ± 20% (or program specific limit such as DoD or DOE), corrective action must be taken. This may include back flushing the column, changing the guard cartridge, changing the frit on the front end of the column, or other minor instrument adjustments, followed by reanalyzing the standard. If the response for any analyte still varies by more than 20% (or program specific limit such as DoD or DOE), a new calibration curve must be prepared and analyzed. The column may also need to be replaced based on the chromatography.

Reported sample results must be bracketed by successful CCVs. When a CCV fails, all samples run since the last successful calibration verification must be reanalyzed. If the CCV recovery is > 20% D (or program specific limit such as DoD or DOE) and the associated samples are ND, the samples may be reported without reanalysis. Flag the data and document the decision in an NCM. For DoD or DOE, this must be accepted by the client and documented in the project records.

12.0 Calculations / Data Reduction

12.1 Detailed calibration equations can be found in the corporate SOP CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

12.2 Qualitative Identification

12.2.1 Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the method detection limit. The required quantitation level is defined as the TALS reporting limit for standard reports, adjusted for initial weight and volume and any dilutions. A UV detector wavelength of 254 nm is used to quantify and report all analytes except PETN and Nitroglycerin that are quantified and reported using a UV detector wavelength of 215 nm.

12.2.2 Identification is confirmed if a peak is also detected within the retention time window on a dissimilar column (Section 12.3).

12.2.3 Sample Evaluation:

12.2.3.1 Analyst judgment weighs heavily in the evaluation of retention time shifts for client samples. The evaluation may be based on RT shifts of the surrogate standard. The chromatograms must be examined closely to ensure that false positive / negative results are not reported. In the absence of significant shifts of the surrogate, peaks within a ± 0.035 minutes (C18 column) or ± 0.07 minutes (phenyl hexyl column) window must be considered positive results.

12.2.3.2 If the sample required significant dilution due to high levels of target peaks or interfering compounds, the surrogate peak may not be obvious. In this case, an adjustment of RTs due to matrix for target compounds cannot be done reliably, and ± 0.035 minutes (C18 column) or ± 0.07 minutes (phenyl hexyl column) (or the established RT window described in Section 10.5.7) from the most recent CCV will be used for all compounds.

12.2.3.3 The expected retention time for target analytes is updated with the retention times of each CCV. If sample matrix is causing significant retention time shifts between CCVs, samples may require dilution and reanalysis to minimize the matrix effects.

12.2.3.4 Method Blank Detections:

When a detection is observed in the method blank on either column, the result must be confirmed on the other dissimilar column to be considered a true hit. See Section 9.3 and Appendix 4 for additional information for evaluation of method blanks.

12.2.3.5 Interferences:

2,4,6-Trinitrotoluene elutes closely with 4-amino-2,6-dinitrotoluene on the primary (C18) column. Because of this close elution, high levels of 2,4,6-trinitrotoluene can overlap the retention time window for, and thus mask the presence of, low levels of 4-amino-2,6-dinitrotoluene. Therefore, 4-amino-2,6-dinitrotoluene may be reported as a detection from the confirmation (phenyl-hexyl) column, even though no peak could be detected on the primary column. In this event, an NCM is necessary.

Other target compound interferences may be observed in samples. Analyst judgment will be necessary to evaluate sample chromatograms and potentially report additional detections without confirmation due to interferences and/or analyze samples at dilutions. In this event, an NCM is required, documenting the decision.

12.3 Second-Column Confirmation

Detection of compounds on the primary column is confirmed using a second, dissimilar column. This column is calibrated using the same calibration levels as the primary column. The analysis on the second column must meet all of the instrument QC described in Section 9.0 and 11.0. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column.

The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 12}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

Acceptance Criteria:

The RPD between confirmed results should agree within 40%; results will then be reported from the primary column.

Corrective Action:

If the RPD is > 40% and there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

If the RPD is > 40% and there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

Special project reporting requirements may supersede these reporting schemes. Verify in the method comments or project Quality Assurance Summary.

12.4 Manual Integrations

Raw instrument data is automatically transferred to Chrom at the completion of each run for further processing. Review the chromatograms to ensure correct assigning of peaks and correct integration of each peak. If manual data manipulations are necessary, they must be justified and documented. See Policy DV-QA-011P for requirements for manual integration.

12.5 % Difference Calculation for ICV / CCV Evaluation

The percent difference for the analysis of a CCV standard is calculated as follows:

$$\%\,\text{Difference} = \left(\frac{\text{Expected Value} - \text{Measured Value}}{\text{Expected Value}} \right) \times 100\% \qquad \textit{Equation 13}$$

12.6 Concentration in Aqueous Samples

The concentration of analyte in the original aqueous sample is calculated as follows:

$$\text{Concentration}, \mu\text{g/L} = \frac{C_{ex} V_t}{V_o} xDF \qquad \textit{Equation 14}$$

Where:
- C_{ex} = Extract analyte concentration, µg/mL
- V_t = Volume of total extract in mL (normally 5 mL)
- V_o = Volume of water extracted in liters (normally 0.5 L)
- DF = Dilution factor, as appropriate

12.7 Concentration in Soil Samples

The concentration of analyte in the original non-aqueous sample is calculated as follows:

$$\text{Concentration}, \mu\text{g/kg} = \frac{C_{ex} V_t}{WD} \times DF \qquad \textit{Equation 15}$$

Where:
- C_{ex} = Concentration of analyte in the extract (µg/mL)

V_t	=	Total volume of original extract in mL (normally 20 mL for 8330A or 40 mL for 8330B); this volume includes the extracted volume in ACN and the 1:1 dilution with $CaCl_2$
W	=	Weight (mass) of sample extracted in kg (normally 0.002 kg for 8330A or 0.010 kg for 8330B)
D	=	(100-% moisture in sample)/100 for dry weight basis or 1 for wet-weight basis
DF	=	Dilution factor, as appropriate

12.8 Concentration of Ammonium Picrate

Ammonium picrate is requested by some clients as a target compound. This is the ammonium salt of picric acid. Chromatographically, there is no difference between these two compounds, therefore, upon request, the result for ammonium picrate (C_{AP}) is calculated based on the measured concentration of picric acid, as follows:

$$C_{AP} = Picric\,Acid\,Result \times \left(\frac{246.13(Molar Mass\,of\ Ammonium Picrate)}{229.11(Molar Mass\,of\ Picric\,Acid)} \right) \quad \textit{Equation 16}$$

In TALS, this calculation is accomplished by opening the batch information in the analytical batch, and setting the calculation line to 1 (Yes).

12.9 LCS and CCV Percent Recovery

$$\text{Control Spike Recovery} = \frac{S_{SR}}{S_A} \times 100\% \quad \textit{Equation 17}$$

Where	S_{SR}	=	Calculated analyte concentration of spiked sample
	S_A	=	Concentration of standard added

12.10 MS / MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\% \quad \textit{Equation 18}$$

Where	S_{SR}	=	Calculated analyte concentration of spiked sample
	S_R	=	Calculated analyte concentration of parent sample
	S_A	=	Concentration of standard added

12.11 Relative Percent Difference Calculation for the MS/MSD

$$RPD = \frac{\left|MS_R - MSD_R\right|}{1/2(MS_R + MSD_R)} \times 100 \qquad \textit{Equation 19}$$

Where RPD = Relative percent difference
MS_R = Matrix spike result of analyte
MSD_R = Matrix spike duplicate result of analyte

12.12 Reporting limits are shown in Table 1. If samples require dilutions or smaller volumes than normally used, the MDLs and RLs will be correct based on the actual volume used and/or the dilution factor. Reporting limits for soil samples are adjusted for the actual weight of sample extracted. As samples are dried prior to subsampling for analysis percent moisture is not determined.

12.13 All results are subject to two levels of technical review. See SOP DV-QA-0020 for a more detailed description for data review and an example of this checklist.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-P-003. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. When reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Flammable solvent waste – Waste Stream C

15.2.3 Flammable vial waste – Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 3535A, Solid Phase Extraction (SPE), Revision 0, December 1996.

16.1.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.1.3 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.1.4 Method 8000D, Determinative Chromatographic Separations, Revision 4, July 2014.

16.1.5 Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 0, September 1994.

16.1.6 Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 1, January 1998.

16.1.7 Method 8330B, Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography, Revision 2, October 2006.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

16.4 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.1, 2017.

16.5 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.2, 2018.

16.6 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.3, 2019.

17.0 Method Modifications:

17.1 Deviations from Method Source and Rationale

Method 8330 prescribes the shelf life for standards as follows:

Standards	Concentration	Shelf Life per 8330A	Shelf Life per 8330B
Stock standards	1,000,000 µg/L (1,000 ppm)	Six Months	One year
Intermediate standards	2.5 to 10,000 µg/L	Thirty days	One year
Working standards	1 to 500 µg/L	Daily	Daily

This SOP describes the use of 100,000 µg/L high-level standards, which are assigned a six month shelf life based on TestAmerica's experience with these materials. Further, a 25 - 1,000 µg/L standard mix is characterized as an intermediate-level, and assigned a 30 day shelf life.

17.2 Acidic water (pH < 3) is added to the concentrated extract, dilutions, and all calibration and check standards in place of reagent water. This is to preserve any Tetryl present in the extract.

17.3 Method 8330B suggests that the %RSD for triplicate analysis for soil should be ≤ 10%. The laboratory uses a criterion of ≤ 20% consistent with DoD/DOE QSM requirements.

18.0 Attachments

Appendix 1. Analyte List
Appendix 2. Suggested Calibration Levels (µg/mL)
Appendix 3. Spike Levels
Appendix 4. Assessment of Method Blank Results
Appendix 5. Suggested Instrument Conditions
Appendix 6. Example Chromatogram from Primary Column – Ultracarb ODS (20)
Appendix 7. Example Chromatogram from Confirmation Column – Luna Phenyl-Hexyl

19.0 Revision History

- Revision 21, dated 19 June 2019
  - Annual Review
  - Minor formatting and language corrections throughout
  - Added extra volume requirement for DNX, MNX, TNX, to Section 8.4

- Revision 20, dated 31 July 2018
  - Annual Review

- Revision 19, dated 30 June 2017
  - Updated text and references to DOD QSM to DOD/DOE QSM 5.0 and/or 5.1 as appropriate throughout SOP.
  - Revised low point of curve in Sections 1.5.2 and 7.5.2 and Appendix 2.
  - Added statement to Section 4.3 that both DOD/DOE QSM 5.0 and 5.1 exclude vegetation.
  - Updated reference from 2003 NELAP Standard to 2009 TNI Standard in Section 9.4.
  - Removed language in Section 9.5 regarding dilution of MS/MSD and parent sample as how to handle over-range MS/MSD is discussed later in the paragraph.
  - Added DOD/DOE QSM 5.1 requirements for grinding blank to Section 9.9.
  - Added notes regarding states requiring 8000D (North Carolina, South Carolina, West Virginia).
  - Added reference to 8000D in Section 16.
  - Updated Section 17.1 to reflect current shelf life for standards
  - Added 3,5-Dinitroaniline (Method 8330B only) to Appendix 2.

- Revision 18, dated 6 July 2016
  - Added clarification to Section 1.3 to comply with document control policy
  - Revised Section 7.8.10 to require no changes to eluent composition between initial calibrations.
  - Removed reference to DoD QSM 3 in Section 8.2.1. The laboratory no longer performs work in compliance with this outdated version of the QSM.

- Added reference to grinding blank in Method Blank description in Section 9.3
- Clarified corrective action for failed method blank in Section 9.3
- Added statement to Section 9.4 to analyze LCS on confirmation column if samples analyzed on confirmation column (positive control).
- Revised corrective action for Section 9.5 to include requirements for dilution of MS/MSD when required
- Added clarification to replicate analyses in Section 9.8
- Removed section 9.11, redundant with Section 9 subsections.
- Added clarification for confirmation analyses in Section 10.5.3
- Added clarification in Sections 10.5.6.2 and 10.5.7.3 for updating RT windows only at start of each 12-hour sequence.
- Removed statement in Section 10.5.7.4 that RT can be adjusted based on each calibration verification standard.
- Added “at least annually” to section 10.5.7.5 for consistency with requirements in the QA Manual.
- Removed statements regarding changes to eluent between initial calibrations.
- Revised corrective action in Section 10.5.8.
- Added method reference to DoD QSM criteria stated in Section 11.3.3
- Removed paragraph referencing the grand mean; TestAmerica discourages the use of the grand mean.
- Clarified Section 12.2.3.4 on Method Blank Detections referencing Section 9.3 and new Appendix 4.
- Clarified sentence in Section 12.2.3.5 regarding interferences.
- Removed references in Section 12.2.3.5 to using MS confirmation as the laboratory uses HPLC analysis on a second distinct column for confirmation rather than MS analysis.
- Added sentence to Section 12.3 corrective action regarding potential for project specific reporting requirements pending results for dual column RPDs.
- Added section 15.2.3 for proper disposal of vial waste.
- Added Section 17.3 to the Method Modifications section.
- Added new Appendix 4 to provide MB interpretation guidance and renumbered remaining appendices.

- Revision 17, dated 31 March 2016
  - Revised Section 4.6 to clarify procedural steps to minimize decomposition of tetryl.
  - Added 12.0 mL vial size to section 6.2.1.1
  - Added TALS reagent IDs in Section 7
  - Revised preparation frequency of working eluent for primary column in Section 7.8.10.1
  - Revised amount of glacial acetic acid to add for working eluent for confirmation column, section 7.8.10.2
  - Added new section 9.6 to describe surrogate, acceptance criteria and corrective actions; renumbered remainder of section 9
  - Revised new sections 9.7 and 9.8 to clarify when duplicate and triplicate are required
  - Updated reference to corporate SOP for calibration curves from CA-Q-S-005 to current version CA-Q-P-003.
  - Revised Section 11.4.2 to clarify use of grand mean cannot be used in conjunction with Method 8000C such as required by Arizona and South Carolina
  - Revised equation 15 to include dry weight correction
  - Updated Section 13 to reflect current practice

- Clarified DoD specific criteria throughout
- Removed all references to AFCEE
- Formatting and grammatical changes throughout.

- Revision 16, dated 31 March 2015
  - Annual Review
  - Updated section 1.4.4 to reflect current analyte exceptions
  - Expanded and clarified section 7.1
  - Made minor corrections to sections 7.3 and 7.4.
  - Moved Instrument Maintenance and Troubleshooting section 13.5 to section 10.7
  - Expanded the Instrument Maintenance and Troubleshooting section 10.7

- Revision 15, dated 31 March 2014
  - Updated Sections 2.2, 6.1.2, 7.8.10, Appendix 4 and Appendix 5 with a new primary column (Agilent Poroshell 120, EC-C18)
  - Added Section 13.5 as a DoD QSM 5.0 requirement
  - Added QSM reference information
  - Annual Review

- Revision 14, dated 30 April 2013
  - Updated Sections 9.1, 10.1 and 10.2
  - Annual Review

- Revision 13, dated 04 April 2012
  - Updated calibration section.
  - Updated standards section.
  - Replaced chromatograms in Appendices 5 and 6 with current chromatograms from Chrom.
  - Source method review.

- Revision 12.2, dated 15 February 2011
  - Added a comment to section 10.3 regarding saturation concentrations for the solid phase extraction cartridges.
  - Added section 11.1 referencing corporate SOP CA-Q-S005, “Calibration Curves”.

Earlier revision histories have been archived and are available upon request.

Appendix 1. Analyte List

Compound	Peak #		CAS #	Symbol	Standard Reporting Limits		
	Col A	Col B			Water (µg/L)	Soil, 2 g (mg/kg)	Soil, 10 g (mg/kg)
2,6-Diamino-4-nitrotoluene**	1	1	59229-75-3	2,6-DA-4-NT	1.0	2.0	1.0
Hexahydro-1,3,5-trinitroso-1,3,5-triazine	2	3	13980-04-6	TNX	0.50	0.25	0.20
Octahydro-1,3,5,7-tetranitro-1,3,5,7,-tetrazocine	3	6	2691-41-0	HMX	0.40	0.25	0.10
2,4-Diamino-6-nitrotoluene**	4	2	6629-29-4	2,4-DA-6-NT	1.0	2.0	1.0
1,3-dinitroso-5-nitro-1,3,5-triazacyclohexane	5	5	80251-29-2	DNX	0.50	0.25	0.20
Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine	6	7	5755-27-1	MNX	0.50	0.25	0.20
Hexahydro-1,3,5-trinitro-1,3,5-triazine	7	8	121-82-4	RDX	0.30	0.26	0.20
Picric Acid (2,4,6-Trinitrophenol)	8	4	88-89-1	PA	0.4	0.25	0.10
1,3,5-Trinitrobenzene	9	19	99-35-4	1,3,5-TNB	1.0	0.25	0.10
1,2-Dinitrobenzene (surrogate)	10	10	528-29-0	1,2-DNB	NA	NA	NA
1,3-Dinitrobenzene	11	13	99-65-0	1,3-DNB	0.40	0.25	0.10
Methyl-2,4,6-trinitrophenyl nitramine	12	22	479-45-8	Tetryl	0.24	0.50	0.20
3,5-Dinitroaniline** (8330B only)	13	11	618-87-1	3,5-DNA	0.40	NA	0.10
Nitrobenzene	14	9	98-95-3	NB	0.40	0.25	0.30
Nitroglycerin	15	12	55-63-0	NG	3.0	5.1	2.0
2,4,6-Trinitrotoluene	16	23	118-96-7	2,4,6-TNT	0.40	0.25	0.10
4-Amino-2,6-dinitrotoluene	17	16	19406-51-0	4-Am-DNT	0.20	0.25	0.10
2-Amino-4,6-dinitrotoluene	18	18	35572-78-2	2-Am-DNT	0.20	0.25	0.10
2,6-Dinitrotoluene	19	20	606-20-2	2,6-DNT	0.20	0.25	0.10
2,4-Dinitrotoluene	20	21	121-14-2	2,4-DNT	0.40	0.25	0.10
2-Nitrotoluene (o-Nitrotoluene)	21	14	88-72-2	2-NT	0.40	0.25	0.20
4-Nitrotoluene (p-Nitrotoluene)	22	15	99-99-0	4-NT	1.0	0.40	0.20
PETN	23	24	78-11-5	PETN	2.0	4.0	2.0
3-Nitrotoluene (m-Nitrotoluene)	24	17	99-08-1	3-NT	0.40	0.50	0.20

**Non-standard spike analytes, only spiked when specifically requested.
A: - UltraCarb5uDODS
B: - Lina-Phenyl Hexyl

Appendix 2. Suggested Calibration Levels (µg/mL)

Compound	Level 1	Level 2	Level 3	Level 4*	Level 5	Level 6	Level 7	Level 8
MNX	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
DNX	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
TNX	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
HMX	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
RDX	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
1,3,5-Trinitrobenzene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
1,3-Dinitrobenzene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
Tetryl	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
Nitrobenzene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
2,4,6-Trinitrobenzene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
4-Amino-2,6-dinitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
2-Amino-4,6-dinitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
2,4-Dinitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
2,6-Dinitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
2-Nitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
3,5-Dinitroaniline (8330B only)	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
3-Nitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
4-Nitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
Nitroglycerin	0.2	0.5	1.0	2.5	4	7	10.	25
PETN	0.2	0.5	1.0	2.5	4	7	10.	25
2,4-Diamino-6-ditrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
2,6-Diamino-4-nitrotoluene	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
Picric Acid	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5
1,2-Dinitrobenzene (surrogate)	0.02	0.05	0.10	0.25	0.4	0.7	1.0	2.5

* This level is used for the daily and continuing calibration standards.

Appendix 3. Spike Levels

LCS / MS / MSD Spike Levels					
Method and Matrix	Working Solution		Spike Amount	Final Concentrations	
	Standard Analytes	Nitroglycerin & PETN		Standard Analytes	Nitroglycerin & PETN
Water, Methods 8330A and 8330B	10 µg/mL	100 µg/mL	0.1 mL	2 µg/L	20 µg/L
Soil Method 8330A (2 g prep)	10 µg/mL	100 µg/mL	0.5 mL	2.5 mg/kg	25 mg/kg
Soil Method 8330B (10 g prep)	10 µg/mL	100 µg/mL	1.0 mL	1.0 mg/kg	10 mg/kg

Surrogate Spike Levels			
Method and Matrix	Working Solution 1,2-DNB	Spike Amount	Final Concentration
Water, Methods 8330A and 8330B	10 µg/mL	0.1 mL	2 µg/L
Soil Method 8330A (2 g prep)	10 µg/mL	0.5 mL	2.5 mg/kg
Soil Method 8330B (10 g prep)	10 µg/mL	1.0 mL	1.0 mg/kg

Appendix 4. Assessment of Method Blank Results

Primary Column	Confirmation Column	Corrective Action
NO	NO	Proceed with analysis; MB is ND
YES	NO	Proceed with analysis; MB is ND
NO	YES	Proceed with analysis; MB is ND
J FLAG	NO	Proceed with analysis; MB is ND
NO	J FLAG	Proceed with analysis; MB is ND
J FLAG	J FLAG	Detection confirms; however, no reworks needed if primary result < ½ RL If primary result > ½ RL, re-extract if possible unless the sample-samples are ND or > 10x the MB for the analyte detected in the MB. Report with NCM
YES	YES	Samples must be ND or > 10x the MB; otherwise re-extract. Report with NCM
J FLAG	YES	If primary result > ½ RL, re-extract if possible unless the sample analytes are ND or > 10x the MB for the analyte detected in the MB. The relative percent difference between the primary and confirmation result determines which result to report. See Section 12.3. Flags applied by formatter. Report with NCM.
YES	J FLAG	

YES = Analyte was detected at a concentration above the RL
NO = Analyte was not detected above the MDL
J FLAG = Analyte was detected at a concentration less than the RL but at or above the MDL

NOTE: The formatter assigned in TALS will apply B flags according to program rules. Do NOT remove B flags. If a client requests such removal, QA staff and PM must be involved in the decision making process and all such removals must be documented. (See Policy P15-001.)

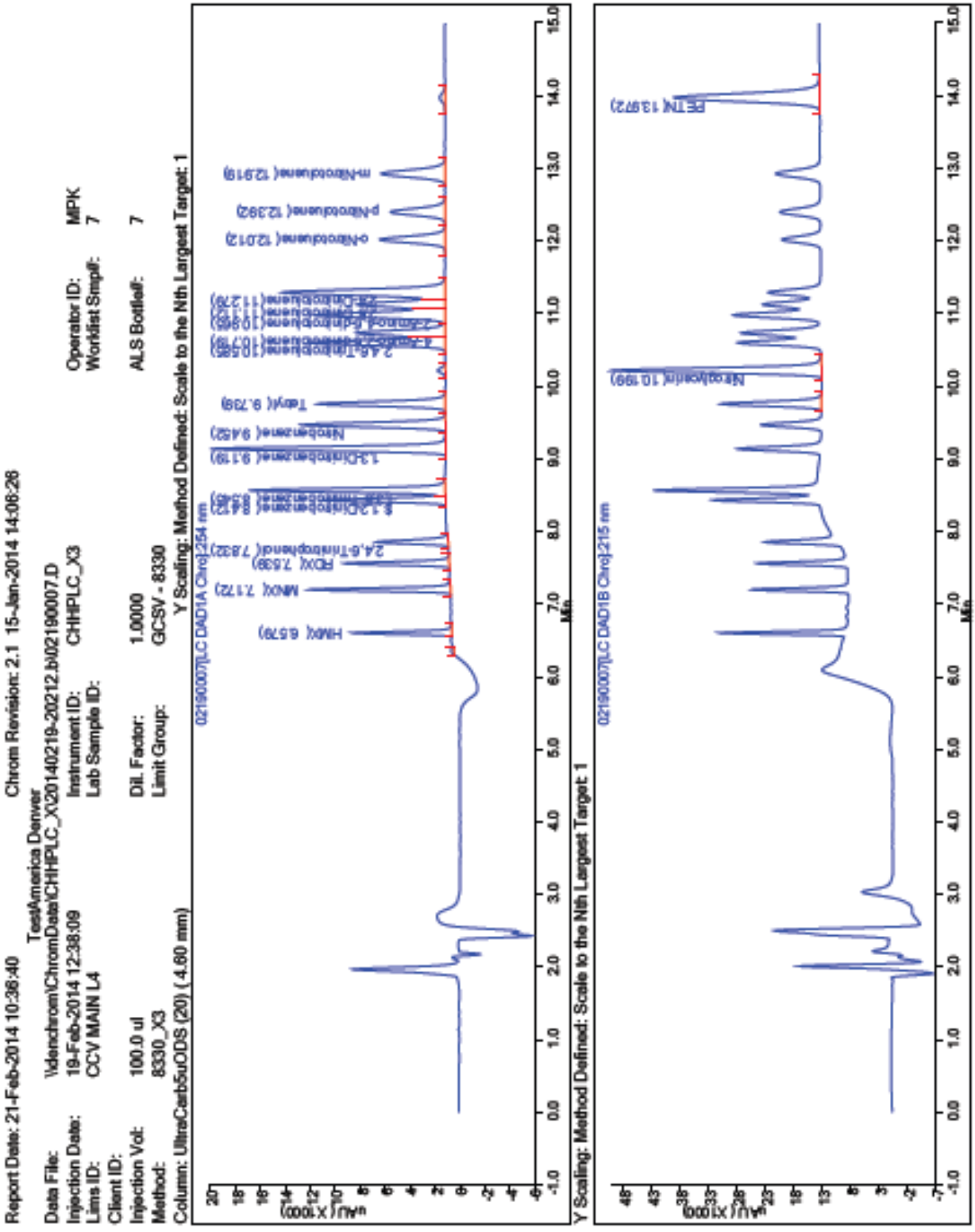
Appendix 5. Suggested Instrument Conditions

Instrument Conditions			
Column Types	Primary Column: Agilent Poroshell 120, EC-C18, 4.6 mm x 150 mm (2.7 µm)		
	Confirmation Column: Phenomenex Luna Phenyl-Hexyl, 4.6mm x 150 mm (3.0 µm)		
Detector - 1st Channel	UV 254 nm, 40 R 550 nm		
Detector - 2nd Channel	UV 215 nm, 40 R 450 nm		
General Parameters		Primary Column	Confirmation Column
	Injection Volume:	50 µL	100 µL
	Column Temperature:	26.9 °C	24.3 °C

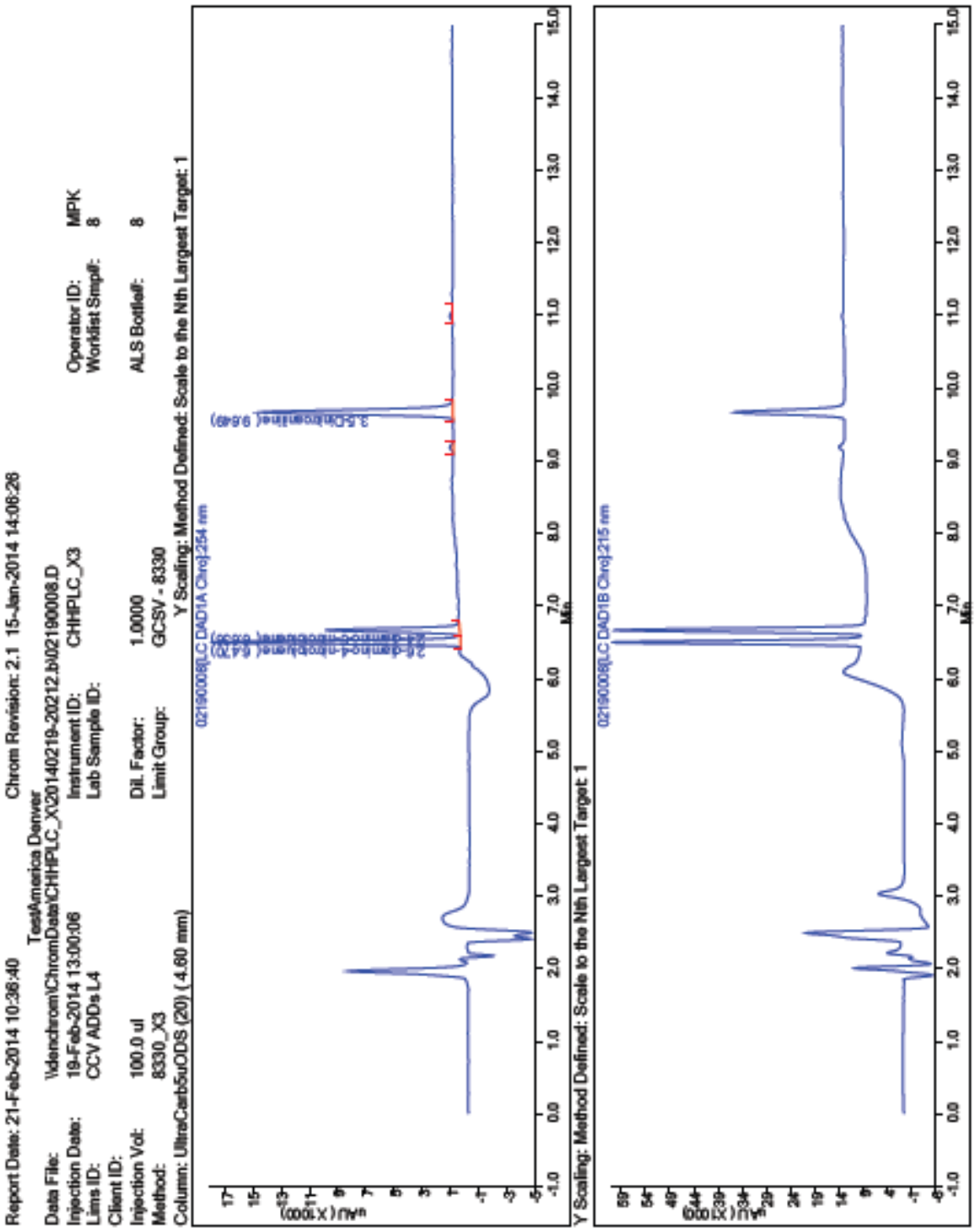
Suggested Column Parameters						
	Stop Time (min.)	**Post Time (min.)**	**Flow Rate (mL/min.)**	**Time (minutes)**	**% H_2O**	**% Methanol**
Gradient: Primary (C18) Column	15.5	5.0	0.75	0.0, 2.0, 2.98, 13.0, 13.5, 15.0, 15.5	90, 90, 41, 40,10, 10, 90	10, 10, 59, 60, 90, 90, 10
Gradient: Confirmation (Phenyl-Hexyl) Column	26.0	4.0	0.8	0.0, 26.0, 30.0	50, 25, 50	50, 75, 50

Appendix 6. Example Chromatograms from Primary (C18) Column

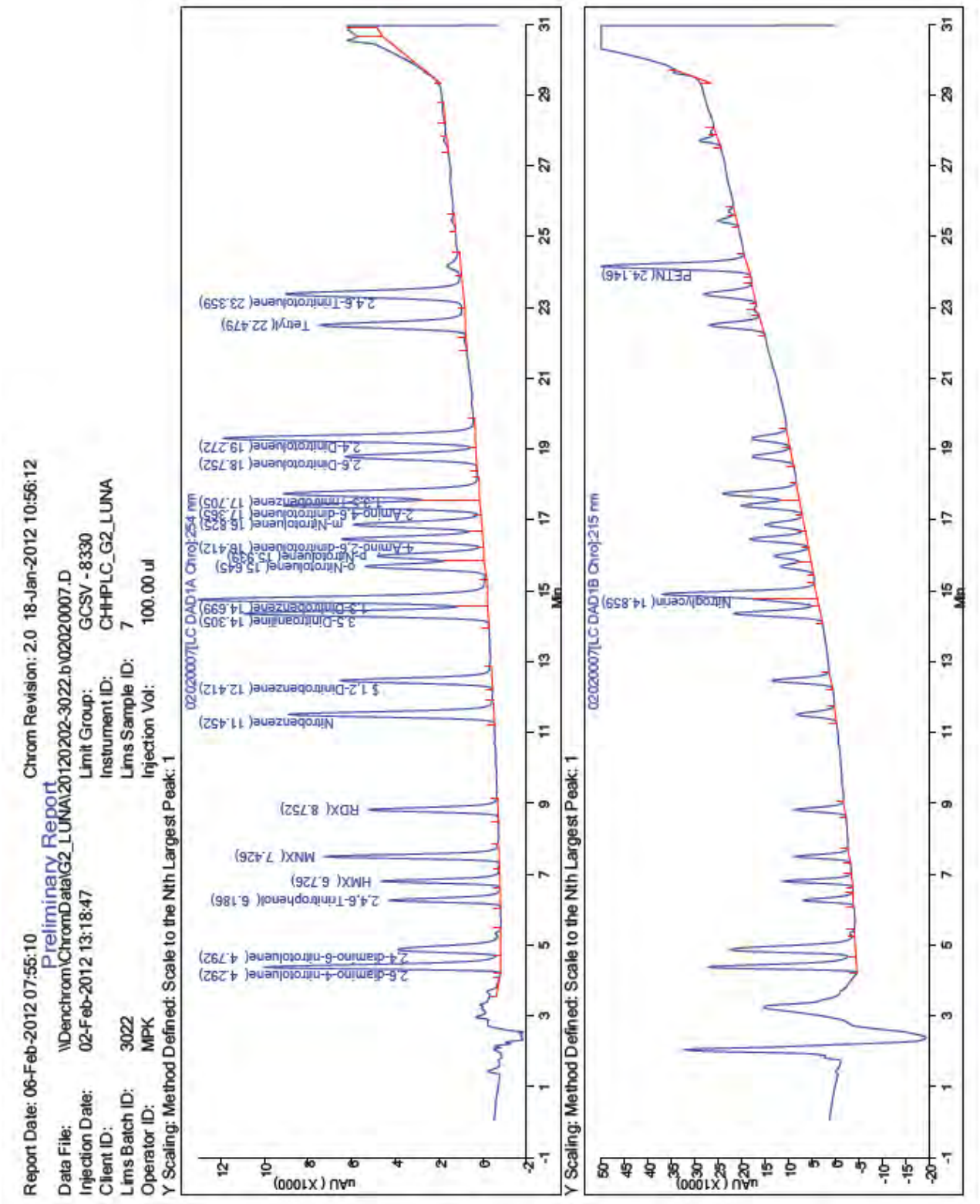
8330IntermStk:



Additional Compounds (8330_ADDs):



Appendix 7. Example Chromatogram from Confirmation (Phenyl-Hexyl) Column



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Polynuclear Aromatic Hydrocarbons by GC/MS Selected Ion Monitoring (SIM) [SW 846 Method 8270C and 8270D]

Approvals (Signature/Date):

Tegan Moore 2/4/19
Technical Specialist — Date

Doug Gomer 2/4/19
Health & Safety Manager / Coordinator — Date

Roxanne Sullivan 2/4/19
Quality Assurance Manager — Date

Richard Clinkscales 2/4/19
Laboratory Director — Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure is a Gas Chromatography/Mass Spectrometry (GC/MS) technique for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion (ng/L or ng/kg) level in waters or solids. This procedure follows the general guidelines of EPA Methods 8270C and 8270D for Selected Ion Monitoring (SIM) analysis.

1.2 The SIM technique optimizes quantitative information at the expense of qualitative information gained from other methods of analysis. It is important to note that this procedure is intended for the analysis of samples previously characterized by another method such as open-scan 8270C/D. The initial characterization is necessary to avoid misidentification of the parent compounds producing the ions used for this analysis.

1.3 In addition, this procedure is appropriate only for sample analytes of interest at less than 10,000 ng/L or 330,000 ng/kg. Samples containing semivolatile organics at concentrations greater than 10,000 ng/L and 330,000 ng/kg should be analyzed by a method designed to detect at higher (part per billion) levels. Samples at these levels may still be analyzed by this procedure; however, extra measurement uncertainty would be introduced because of the sample dilutions that would be required.

1.4 This procedure is applicable to water and soil samples. For water samples, 1 liter of water is extracted. It is also possible to extract 250 mL of water and analyze by an LVI (large volume injection) method designed to maintain reporting limits while reducing the initial volume of sample required for extraction. For soil samples, a sample aliquot of 30 g is extracted.

1.5 Analytes, Matrix(s), and Reporting Limits

The standard list of compounds that can be analyzed by this procedure is shown in Table IV. Typical reporting limits are 100 ng/L for aqueous samples and 5.0 μg/kg for soil samples for the PAH compounds.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Aqueous Samples

Analytes of interest are extracted from water samples using separatory funnel extraction (EPA 3510C or 3510C_LVI) described in SOP DV-OP-0006. The PAH compounds are extracted from the sample without any adjustment to pH. The concentration of organic extracts is covered in SOP DV-OP-0007.

2.1.2 Solid Samples

Solid samples are extracted by sonication (EPA 3550C), which is

covered in SOP DV-OP-0016 or by microwave extraction (EPA 3546) described in SOP DV-OP-0015. The extraction solvent is a 1:1 mixture of methylene chloride and acetone. The concentration of organic extracts is covered in SOP DV-OP-0007.

2.2 Instrumental Analysis

2.2.1 Quantitation of the extracted compounds is performed by gas chromatography - mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). Routine instrument conditions and the ions used for analysis are shown in Tables I and IV, respectively.

2.2.2 Development of a successful SIM method requires identifying the ions to be monitored, the ion dwell times, the ions in each group, and the timing for switching between groups. A quantitation ion is selected with confirmation ions being monitored for identification purposes (see Table IV). Switching times are set where there is adequate resolution (a gap of 1-2 minutes) between peaks. If there is inadequate time between eluting peaks, small retention time shifts may cause peaks to partially or completely disappear as there are changes in the ions monitored. Dwell times will be set by default once the ions per group and the switching times are identified in the data acquisition method. These can be adjusted manually in order to optimize sensitivity as needed.

3.0 Definitions

3.1 Refer to TestAmerica Denver's Quality Assurance Manual (QAM) and SOP DV-QA-003P for definitions of the quality control terms used in this document.

3.2 Selected Ion Monitoring - A mass spectrometry technique that provides lower detection level capability by monitoring fewer mass scans for longer periods of time than is done in open-scan methods.

3.3 Primary Ion Area - The signal chosen for quantitation purposes.

3.4 Secondary Ion Area - The signal chosen for identification and confirmation purposes.

3.5 LVI – Large Volume Injection – An analysis method designed to maintain reporting limits while reducing the initial volume of sample required for extraction by increasing the volume of sample extract introduced onto the GC column.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high purity reagents and solvents helps to minimize interference problems.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

4.3 An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which produces the same ion used for quantitation of one of the PAHs. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present. Open scan analysis to identify compounds throughout the mass range is the most reliable assurance against reporting false positives.

4.4 Contamination by carryover can occur whenever high–level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, typically with compound concentrations well in excess of the high calibration standard, the sample analysis that immediately follows the high level sample should be evaluated for carryover. If detections are observed for the compounds that were over the calibration range in the prior sample this sample should be reanalyzed to rule out carryover unless some other objective evidence indicates that carryover is not an issue.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.3.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect the instrument from its source of power.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm - TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm - TWA 125 ppm - STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
PAH standards can contain all or some of the following: benzo(a)anthracene benzo(b)fluoranthene benzo(k)fluoranthene benzo(a)pyrene chrysene dibenz(a,h)anthracene indeno(1,2,3-cd)pyrene naphthalene	 Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen 	 0.2 mg/m^3 - PEL 10 ppm - PEL	Standards contain low concentrations of compounds known to be or suspected to be carcinogens. All PAH compounds are considered to be hazardous, toxic, and irritants. Some or all are reported human carcinogens, mutagens, and/or teratogens.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Gas Chromatograph (See Table I for operating conditions)

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for split or splitless injection when using capillary columns. Instruments F (Agilent 6890 with a 5973 MSD), G5 (Agilent 6890 with a 5975 MSD), and X4 (Agilent 6890 with a 5973 MSD) may be used for this analysis. Equivalent instruments may be used.

6.1.2 Mass Spectrometer (See Table I for operating conditions)

A mass spectrometer operating at 70 eV (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

6.1.3 A computer system interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

6.1.4 Please refer to the Master List of Documents, Software, and Hardware (or current revision) located on R:\QA\Read\Master List of Documents for the current software and hardware to be used for data processing.

6.2 Supplies

6.2.1 All glassware used, both within the scope of this SOP and for the initial sample extraction (see SOPs DV-OP-0006, DV-OP-0008, DV-OP-0007, DV-OP-0015, and DV-OP-0016), must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with acetone.

6.2.2 Glassware should not be oven dried or heated in a muffle furnace. Successive solvent rinses of the CLLE, separatory funnel, sonication, and Kuderna-Danish glassware are required to minimize low level contamination of samples.

6.2.3 Store glassware inverted or in sealed containers capped with aluminum foil.

6.2.4 Gas-tight syringes, various sizes, and SMI pipettors.

6.2.5 Serological pipettes are used for final extract volume measurement.

6.2.6 Micro reaction vessels, 1.8 mL vials with Teflon caps, for storing concentrated extracts.

6.2.7 Column – A Varian VF-5MS 30-meter fused silica capillary column, 0.5 μm film thickness, 0.25 mm ID, plus 10-meter EZguard, or equivalent.

6.2.8 Agilent Ultra Inert splitless single taper liners.

6.2.9 Amber crimp cap vials with Sil/PTFE aluminum seals.

6.2.10 Hamilton 10 μL autosampler syringes.

7.0 Reagents and Standards

7.1 Reagents

All solvents are reagent grade or higher unless specified otherwise. See SOPs CA-Q-S-001 and CA-Q-S-001 DV-1 for a description of the program for testing solvents prior to use. The manufacturer expiration applies to all solvents and when not specified by the manufacturer the expiration will be recorded as one year after opening the solvent for use.

7.1.1 Methanol, reagent grade.

7.1.2 Methylene chloride, reagent grade.

7.1.3 Helium gas, 99% + purity.

7.2 Standards

Commercial standards are received in flame-sealed ampoules or as neat, 100% concentration solutions. Standards are verified before use. Details concerning verification of standards are given in SOP DV-QA-0015. Stock standards are stored refrigerated at ≤ 6 °C. All stock standards must be protected from light. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier, if the vendor indicates an earlier date. Dilutions or working level standards that are prepared from stock standards are assigned an expiration date according to the earliest expiring stock or one year from the date of preparation, whichever date is earlier.

For the PAH compounds and the additional compounds that are mentioned in this SOP the following stock standards are currently used: **MS-48925** Supelco cat. # 48925 at 1,000 µg/mL (surrogates), **MS-31009** Restek cat. # 31009 SV Calibration Mix #3 at 2,000 µg/mL, **MS-31010** Restek cat. # 31010 SV Calibration Mix #4 at 2,000 µg/mL (has 2-methyl naphthalene), **MS-31853** Restek cat. # 31853 at 2,000 µg/mL (1,4-Dioxane), **MS-31995** Restek cat. #31995 8270 Calibration Mix #5 at 2,000 µg/mL (has all PAH compounds including 2 methyl naphthalene), **MS-APP914820X** Accustandard cat. #APP-9-148-20x at 2,000 µg/mL (n-

nitrosodiethylamine), and **MS-47643-U** Supelco cat. # CRM47643 8270 Ether/Phthalate mix at 2,000 µg/mL. Other vendors and mixes may be substituted for these stocks but an NCM must be written for the SOP deviation.

7.2.1 GC/MS Tuning Standard

A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) at a concentration of 50 µg/mL (25 µg/mL for LVI) is prepared by diluting 0.5 mL of the stock to a final volume of 10ml. The current vendor for the tuning standard is Supelco cat. # 47548-U at a concentration of 1,000 µg/mL.

7.2.2 Calibration Standards

Calibration standards for the initial calibration (ICAL) are prepared at 7 concentrations to cover the calibration range by diluting vendor stock standard solutions using methylene chloride. The standards are prepared directly in autosampler vials by using syringes to deliver the appropriate volumes of stock standard solution, internal standard solution, and methylene chloride. The following tables summarize a typical set of calibration standards:

MS-SIM SL_Stk is a 200 µg/mL calibration substock that is prepared by diluting 0.5 mL of **MS-31009, MS-31010, MS-31853, MS-31995, (and MS-APP914820X)** to a final volume of 5 mL.

Standard Method: Prepared using a PAH SIM stock standard **MS-SIMSL** with a concentration of 20 µg/mL for levels 4 through 7. The **MS-SIMSL** standard is prepared by diluting 0.2 mL of **MS-48925** (surrogates) and 1 mL of **MS-SIM SL_Stk** and 0.1 mL of **MS-47643-U** to a final volume of 10 mL. A secondary PAH SIM stock standard **MS-SIMSL Int** prepared by diluting 1 mL of the **MS-SIMSL** to a final volume of 10 mL with a concentration of 2 µg/mL is used to prepare levels 1 through 3:

Vol Stock (µL)	Methylene Chloride (µL)	Internal Standard (µL)	Final Volume (µL)	Conc PAH (µg/mL)
5	495	50	500	0.02
25	475	50	500	0.1
75	425	50	500	0.3
15	485	50	500	0.6
30	470	50	500	1.2
62.5	437.5	50	500	2.5
125	375	50	500	5.0

LVI Method: Prepared using a PAH SIM stock standard with a concentration of 20 µg/mL for levels 6 and 7. A secondary PAH SIM stock standard with a concentration of 2 µg/mL is used to prepare levels 1 through 5:

Vol Stock (µL)	Methylene Chloride (µL)	Internal Standard (µL)	Final Volume (µL)	Conc PAH (µg/mL)
1	499	50	500	0.004
5	495	50	500	0.02
15	485	50	500	0.06
30	470	50	500	0.12
60	440	50	500	0.24
12.5	487.5	50	500	0.5
25	475	50	500	1.0

7.2.3 Initial Calibration Verification (ICV) Standard (MS-SIM SSV)

A second source initial calibration verification (ICV) standard is prepared using a standard solution that is obtained from a source independent from the source that supplies the standard used for the initial calibration. It is prepared by diluting 30 µL of a substock that is at a concentration of 20 µg/mL to a final volume of 0.5 mL. The final PAH SIM concentration for this ICV standard is 1.2 µg/mL (0.24 µg/mL for LVI).

The substock for **MS-SIMSSV** (above) is prepared by diluting 1 mL of another substock, **MS-HSLB1_STK,** to 10 mL final volume.

MS_HSLB1_STK is prepared by diluting 2 mL of **MS-570666.SEC** (Restek cat. # 570666.sec 8270 List 1/Std#1 Mega Mix at 500, 1,000, 2,000 µg/mL) and 2 mL of **MS-569731SEC** (Restek cat. #769731.sec 8270 List 1/Std #10 at 2,000 µg/ml) to a final volume of 10 ml. The final concentration of this stock varies as either 200 µg/mL or 400 µg/mL depending upon the compound.

The final PAH SIM concentration for this ICV standard is 1.2 µg/mL (0.24 µg/mL for LVI).

7.2.4 Continuing Calibration Verification (CCV) Standard

A standard with the same analytes and concentrations as the 600 ng/mL (120 ng/mL for LVI) calibration standard. The standard may be from the same preparation as the initial calibration or prepared at a later date.

7.2.5 Surrogate Spiking Solutions (8270SIM Surr)

The surrogate spike solution contains neutral surrogates at concentrations of 500 ng/mL in methanol. It is prepared by diluting 0.1 mL of **8270SurrStkHL** (Restek cat. #567685 at 5,000 µg/mL) to a final

volume of 1,000 mL with acetone. Table II lists the surrogate compounds for the standard list of PAHs.

- 1.0-liter water extractions, add 1.0 mL of the surrogate spike solution
- 250-mL LVI water extractions, add 0.250 mL of the surrogate spike solution
- 30-gram soil sample extractions, add 1.0 mL of the surrogate spike solution

7.2.6 Internal Standard (IS) Solutions (MS-SIM IS)

A 6,000 ng/mL solution of the internal standards is prepared in methylene chloride from vendor stock **MS-57604** (Restek cat. #567684 8270 Internal Standard at 2,000 µg/mL) by diluting 60 mL of this stock to 300 mL final volume. Then 1.5 mL of this stock, **MS-IS**, is diluted to 100 mL to yield the **MS-SIMIS** spiking solution. Table III lists the IS compounds.

To each sample extract, 20 µL of the respective IS solution is added to a 200 µL aliquot of the sample extract for both standard (1 L sample) and LVI extracts.

7.2.7 LCS, MS, and MSD Spike Solution (8270BO-SIMLCS)

A methanol solution containing the requested spike compounds at a concentration of 900 ng/mL each is prepared from the vendor stock solution by diluting 0.225 mL of **MS-570666** (Restek cat. #570666 HSL Mega Mix at 1,000 µg/mL) to a final volume of 250 mL with P&T methanol. Following are the final sample concentrations of the spiked compounds for the water and solid extractions:

- 1.0-liter water extractions, add 1.0 mL of the spike solution, [PAH] = 900 ng/L
- 250-mL LVI water extractions, add 0.250 mL of the spike solution, [PAH] = 900 ng/L
- 30-gram soil sample extractions, add 1.0 mL of the spike solution, [PAH] = 30 µg/kg

7.3 All stock and working standards are stored according to the manufacturer's instructions. Dilutions from stocks may not be assigned expiration dates that exceed the stock standard expiration date set by the manufacturer.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample Amounts

8.1.1 Water samples are collected in pre-cleaned amber glass bottles fitted with a Teflon-lined cap. To guarantee the ability to meet routine reporting limits, two full bottles of sample should be provided. Additional bottles are

needed to satisfy the requirements for matrix spikes and duplicate matrix spikes. For the standard method, each bottle should be 1.0 L; for the LVI method, each bottle should be 250 mL.

8.1.2 Soil samples are collected in an 8-ounce, pre-cleaned, wide-mouth jar with a Teflon-lined lid.

8.2 Samples are chilled to a temperature between 0 and 6 °C immediately after collection and shipped via overnight carrier to the laboratory.

8.3 Samples and excess sample volume must be stored refrigerated at ≤ 6 °C from when the log-in process is completed (see SOP DV-QA-0003) to storage after analysis.

8.4 Water samples must be extracted within 7 days of the time of sample collection, while solid samples must be extracted within 14 days of sampling. Extracts must be analyzed within 40 days from the start of the sample extraction.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), and Department of Energy (DOE), are described in TestAmerica Denver Policy SOP DV-QA-024P, *QA/QC Requirements for Federal Programs*. Table 8 details the components of the DoD QSM 5.0 and DoE QSAS 3.0 that are different from TestAmerica Denver's standard procedures, for further details see SOP DV-QA-024P. Also listed are the variances that TestAmerica is requesting for this analysis; these alternate criteria are only used with project-specific approval.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

A method blank is processed and analyzed with each analytical batch, not to exceed 20 samples. For aqueous samples, the method blank consists of reagent water spiked with surrogates. For soil samples, the method blank is Ottawa sand spiked with surrogates. This sand is mixed with sodium sulfate for extraction by ultrasonication. Method blanks are used to assess whether the laboratory has contributed contamination to the sample analysis process that adversely affects the accuracy of the determination of target analytes. The goal is to have no detectable contaminants in the method blank. However, due to the sensitivity of this analysis, it is not uncommon to detect target analytes at levels above the method detection limit (MDL).

Acceptance Criteria: MB results must be less than ½ the reporting limit.

Corrective Action: If the MB exceeds ½ the RL for any target analyte, then one of the following must apply for acceptance of the batch:

The blank contamination is less than $^{1}/_{10}$ of the measured concentration of any sample in the associated preparation batch, or

The blank contamination is less than the concentration present in the samples and is less than $^{1}/_{10}$ of the regulatory limit, or

The same contaminants are <u>not</u> found in the associated samples.

NOTE: Positive method blank results below the reporting limit should be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

9.3 Laboratory Control Samples (LCS)

A Laboratory Control Sample (LCS) is processed and analyzed with each analytical batch not to exceed 20 samples. For aqueous samples, the LCS consists of reagent water spiked with the analytes of interest and surrogates. For soil samples, the LCS is Ottawa sand spiked with analytes of interest and surrogates. For ultrasonic extraction, sodium sulfate is added to the reagent sand. The LCS spiking solution is described in Section 7.2.7. LCS results are used to determine whether the analytical system is in control. Depending on project requirements, a duplicate LCS (LCSD) may be required to assess the precision of the analytical system.

Acceptance Criteria: The percent recovery for each requested target analyte in the LCS must fall within the established control limits (found in TALS).

Corrective Action: If the percent recovery for any requested analyte in the LCS exceeds the upper control limit and the analyte is not detected in any of the associated samples, then no further action is required, and data are reported with an NCM.

If the percent recovery for any analyte in the LCS exceeds the upper control limit and the analyte is detected in any of the associated samples, then reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the preparation batch.

If the percent recovery for any analyte in the LCS is below the lower control limit, reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the preparation batch.

If re-extraction of samples is not possible or the client requests the samples not be re-extracted, qualify data and explain in a NCM.

9.4 Matrix Spike and Spike Duplicate (MS/MSD)

One matrix spike (MS) sample and one matrix spike duplicate (MSD) sample are prepared and analyzed for each preparation batch. An MS sample is a field sample to which known amounts of the target analytes, as well as the surrogates, have been added. An MSD is a second aliquot of the same sample that is spiked the same as the MS. The MS/MSD spiking solution is described in Section 7.2.7. MS results are used to assess the effects of the sample matrix on the accuracy of the analytical system. The MSD results are used to assess the effects of the sample matrix on the precision of the analytical system. Given the expected variability in sample matrix, the MS/MSD results are applicable to only the sample used to prepare the MS and MSD. MS/MSD results should not be extrapolated to other samples without extensive investigation and characterization to demonstrate similarity between samples. The DoD QSM 5 requires that the MS/MSD be prepared from samples from the same site.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM 5 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind

that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.5 Internal Standards

The internal standards listed in Table III and described in Section 7.2.6 are spiked at the same level in all field sample extracts, QC sample extracts, instrument blanks, and calibration standards.

Acceptance Criteria: The peak area for each internal standard in each field sample and QC sample extract should be between 50% and 200% of the peak area for the same internal standard in the midlevel standard of the initial calibration.

Corrective Action: If the internal standard fails acceptance criteria, then perform the following corrective actions:

- Inspect system for malfunction and correct as needed.
- Reanalyze the affected samples.
- If the interference cannot be corrected for field samples, the earlier analysis is reported with discussion in an NCM.
- If QC samples have internal standard failures that are confirmed by re-analysis, the cause of the failures must be investigated.

9.6 Surrogate Compound Analysis

Surrogate compounds listed in Table II and described in Section 7.2.4 are added to all field and QC samples prior to extraction. Surrogate recoveries are used to assess individual sample matrix effects on sample preparation and analysis.

Acceptance Criteria: Surrogate recoveries must fall within established control limits. QC sample results are not acceptable unless the surrogate recoveries for those samples are in control.

Corrective Action: Corrective action must be considered for any surrogate failure and may depend on project-specific instructions. Lacking instructions to the contrary the following actions shall be taken:

- Evaluate sample chromatogram and other QC.
- If the surrogate(s) fail in the LCS and/or method blank, then re-prepare and reanalyze all associated samples. Samples may be excepted where the surrogate recovers high in the MB and the MB does not have detection above ½ of the RL. Likewise, if the surrogate is out of control in the LCS but the LCS compounds recover in control then the samples may be reportable but the program requirements must be checked to see if this is acceptable. In any case an NCM must be written to describe the situation.
- For surrogate failures in field samples, re-prepare and reanalyze the samples, unless matrix interference is evident from earlier analysis or from chromatograms in which case the samples are reported with an NCM.

9.7 Instrument QC

9.7.1 Instrument Optimization

9.7.1.1 The GC/MS system must be tuned to meet manufacturer's specifications, using a suitable calibration such as perfluorotri-n-butylamine (FC-43). This is performed through the auto-tune feature in the software. The mass calibration and resolution of the GCMS system is then verified by the analysis of DFTPP prior to the analysis of any standards or samples. In some instances the laboratory will opt to omit the DFTPP. The DFTPP tune check is less useful for SIM analysis than it is for full scan analysis because the DFTPP analysis must necessarily be done in full scan mode. When this check is omitted, the FC-43 check will be performed daily.

9.7.1.2 The instrument is tuned for DFTPP (decafluorotriphenylphosphine), calibrated initially with a seven-point calibration curve, and verified each 12-hour shift that samples are to be run with one or more continuing calibration verification (CCV) standard(s).

9.7.2 Instrument Tuning

At the beginning of every 12-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table VI) are achieved for DFTPP.

9.7.2.1 Inject 1 µL of the 50 µg/mL GC/MS tuning standard (see Section 7.2.1) into the GC/MS system.

9.7.2.2 The mass spectrum of the DFTPP must be obtained in the following manner: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is also required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of the DFTPP. Do not subtract part of the DFTPP peak. A procedure compliant with these requirements is programmed into a Macro used to evaluate the DFTPP spectrum. Confirm that all the key m/z criteria in Table VI are achieved.

9.7.2.3 If all the criteria are not achieved, the analyst must adjust or retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

9.7.3 Initial Calibration (ICAL)

9.7.3.1 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns and source maintenance.

9.7.3.2 A minimum five-point initial calibration curve must be established for linear fit calibrations (weighted or unweighted). Six points or more are required for second order curve fits. See Section 9.7.4 for Calibration Acceptance Criteria.

- The concentrations of standards commonly used to construct the PAH calibration curve are 20, 100, 300, 600 (often analyzed before the rest of the standards and called the ICIS), 1,200, 2,500, and 5,000 ng/mL.

- For the LVI method, the concentrations of standards commonly used to construct the PAH calibration curve are 4, 20, 60, 120 (often analyzed before the rest of the standards and called the ICIS), 240, 500, and 1,000 ng/mL.

9.7.3.3 If the concentration of any target compound in a sample exceeds the calibration range, the extract must be diluted with methylene chloride so that the concentrations of all target compounds fall within the range of the calibration curve, and be reanalyzed. Any samples analyzed immediately following the sample that exceeded the linear range may require reanalysis due to possible carryover from the high-level sample.

9.7.3.4 Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is supported or adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 12 hours.

9.7.3.5 Calculate the response factor (RF) for each analyte for each calibration standard level as described in Section 11.3. Calculate the mean RF and relative standard deviation (RSD) for each analyte.

9.7.4 Calibration Acceptance Criteria and Corrective Action:

Acceptance Criteria 8270C:

The RSD of the initial calibration for each analyte of interest must be ≤ 35%.

Acceptance Criteria 8270D:

Refer to Table VII for the acceptance criteria for minimum response factor and RSD. Two target compounds and surrogates may fail to meet the minimum RRF criteria listed in Table VII but must still meet the minimum RRF criteria of 0.010 (excluding compounds with a minimum RRF requirement of 0.010). In addition, two target compounds and surrogates may fail to meet the RSD criteria listed in Table VII but must still meet the maximum RSD requirement of 40% (excluding compounds with a maximum RSD requirement of 40%). Refer to SOP DV-QA-024P for requirements for federal programs.

Acceptance Criteria for DoD5:

The RSD of the initial calibration for each analyte of interest must be <

15%. See SOP DV-QA-024P for further details for QSM 4.2 requirements.

Corrective Actions:

If these criteria cannot be met, least-squares weighted or unweighted linear regression may be used to establish a calibration function as described in Section 11.4. In this case, the correlation coefficient (r) must be greater than 0.995 (equivalent to $r^2 \geq 0.99$) or a second-order regression fit with coefficient of determination (COD, r^2) greater than 0.99 may be used. If these linearity criteria are not achieved, verify the standard preparation and instrument conditions, and then recalibrate the instrument. If technical acceptance criteria are not met, it may be necessary to clean the ion source, perform injector maintenance, change the column, or take other corrective actions.

9.7.5 In the event that a least-squares regression is used, the analyst should evaluate the bias at the lower portion of the curve. This can be accomplished by re-fitting the low point standard back into the curve. The recalculated concentration should be within ± 50% of the standard's true concentration. If these criteria are not met, the analyst may have to evaluate the concentration range of the standards, or the lower limit of quantitation.

9.8 Initial Calibration Verification (ICV)

The Initial Calibration Verification (ICV) is a second-source, mid-level standard that is analyzed immediately following the initial calibration standards.

Acceptance Criteria: The absolute value of the difference between the measured PAH analyte concentration and the true value must be ≤ 30 % or be ≤ 20 % for DoD QSM 4.2 or 5.0.

Corrective Action: If the ICV recovery fails, then take the following actions:

- Verify standard preparation, and if incorrect, re-prepare the ICV standard solution.
- If preparation of the ICV standard was correct, then re-prepare the initial calibration standards and recalibrate.

9.9 Continuing Calibration Verification (CCV)

Every 12 hours, the mass spectrometer response for each PAH relative to the internal standard is determined by analyzing a 600 ng/mL calibration standard (120 ng/mL for the LVI method). The RF for each compound in the continuing calibration verification (CCV) analysis is compared to the RF for that compound in the ICAL.

9.9.1 Acceptance Criteria 8270C

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must be ≤ 35 %.

9.9.2 Acceptance Criteria 8270D

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must meet the criteria in Table VII. The compounds must also meet the minimum response factor criteria listed in Table VII. Two target compounds and surrogates may fail to meet the minimum RRF criteria in Table VII (excluding compounds with a minimum RRF requirement of 0.010) but must still meet the minimum RRF criteria of 0.010. In addition, two target compounds and surrogates may fail to meet the difference criteria in Table VII (excluding compounds with a maximum percent difference requirement of ± 40%) but must still meet the maximum difference requirement of ± 40%. Refer to SOP DV-QA-024P for requirements for federal programs.

9.9.3 Acceptance Criteria for DoD QSM 4.2 or 5.0

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must be ≤ 20 % for DoD QSM 4.2 or 5.0.

9.9.4 Acceptance Criteria 8270C & 8270D

9.9.4.1 The internal standard response of the CCV must be within 50 - 200% of the internal standard response in the mid-level (ICIS) of the most recent ICAL sequence.

9.9.4.2 The internal standard retention time must be within ± 30 seconds of the internal standard retention time in the corresponding level of the most recent ICAL sequence.

9.9.5 Corrective Action:

9.9.5.1 If, for any analyte, the CCV RF does not meet the stipulated acceptance criteria, a five-point calibration curve must be repeated for that analyte prior to the analysis of samples.

9.9.5.2 If any internal standard retention time in the CCV changes by more than 30 seconds from that of the corresponding level of the most recent ICAL sequence, the chromatographic system must be inspected for malfunctions and corrections made, as required.

9.10 Closing CCV (DoD QSM 5.0 only)

DoD QSM 5.0 requires a closing CCV, injected within 12 hours of the DFTPP injection.

9.10.1 Acceptance Criteria

All reported analytes and surrogates must be within ± 50%.

9.10.2 Corrective Action

Recalibrate and reanalyze all affected samples since the last acceptable CCV

Or

Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails perform column maintenance and recalibrate; then reanalyze all affected samples sine the last acceptable CCV.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 Aqueous Sample Extraction and Concentration

10.3.1.1 Instructions for the extraction of aqueous samples may be found in SOP DV-OP-0006.

10.3.1.2 Instructions for the concentration of extracts may be found in SOP DV-OP-0007.

10.3.2 Soil Sample Extraction and Concentration

10.3.2.1 Instructions for the ultrasonic extraction of soil samples may be found in SOP DV-OP-0016.

10.3.2.2 Instructions for the microwave extraction of soil samples may be found in SOP DV-OP-0015.

10.3.2.3 Instructions for the concentration of extracts may be found in SOP DV-OP-0007.

10.4 Sample Analysis

10.4.1 All aliquotting, extract dilutions, and spike additions must be performed in the trace fume hood using equipment dedicated to PAH-SIM analysis. An aliquot of 200 µL of each sample extract is placed into a two-milliliter GC/MS autosampler vial. Sufficient volume of extract remains should reanalysis be necessary.

10.4.2 Prior to analysis, 20 µL of internal standard is added to the sample vial giving a final internal standard concentration of 600 ng/mL (150 ng/mL for LVI) in the extract.

10.4.3 Representative aliquots are injected into the gas chromatograph/mass spectrometer using similar conditions to those summarized in Table I. The injection volume is 1 µL (5 µL for LVI).

10.4.4 Whenever an unusually concentrated sample is encountered, it may be necessary to reanalyze the subsequent sample extracts after analyzing an instrument blank to demonstrate that there is no cross contamination.

10.4.5 The following is a typical analytical sequence:

- Solvent rinses, as needed
- MS tune
- ICAL plus ICV or CCV
- Instrument blank
- MB, LCS
- LCSD (if requested by client)
- Sample extracts
- MS and MSD are interspersed with sample extracts, and usually run after the sample from which they are produced.
- The last sample extract must be injected within 12 hours of the tune.

10.4.6 The sequence may be altered to accommodate reanalysis or additional instrument blank and calibration evaluations. At a minimum, an instrument blank or a method blank shall be included in the sequence. Refer to Policy DV-QA-003P for additional details.

10.4.7 The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each analyte as shown in Table IV.

10.4.8 All compounds detected at concentrations above the method MDL are checked to ensure that the confirmation ion is present at the appropriate ratio.

10.4.9 All compounds detected at concentrations above the highest calibration standard require dilution and reanalysis. In addition, any samples that

were analyzed immediately following a high-level sample should be reanalyzed to rule out carryover from the high-level sample, unless they are preceded by an acceptable instrument blank or the high compound(s) were not detected in the subsequent samples.

10.4.10 Manual Integrations

10.4.10.1 Upon completion of the analytical sequence, transfer the raw instrument data to Chrom for further processing. Review the chromatograms to ensure correct assigning of peaks and correct integration of each peak.

10.4.10.2 Note that certain compounds (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) may require frequent manual integrations. Special attention must be exercised by the analyst and secondary reviewer for compounds that are commonly mis-integrated in automated software or are manually integrated. If manual data manipulations are necessary, they must be justified and documented. See DV-QA-011P, *Acceptable Manual Integration Practices*.

10.5 Troubleshooting and Maintenance

10.5.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix B, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.5.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the “topboard” or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

Obtain electronic ion current profiles (EICP) for the primary mass ion and the confirmatory ion for detected compounds. The following criteria must be met to make a qualitative identification:

11.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

11.1.2 The retention time (RT) of unknown peaks must fall within ± 0.2 minutes of the RT for the compound in the daily calibration standard (mid-point ICAL or daily CCV).

11.1.3 The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. A compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive information includes correct relative retention time (RRT) and the presence of the secondary ion, but the ratio falls outside of ± 20% of the primary ion, which may be caused by an interference of the secondary ion.

11.1.4 Structural isomers that have very similar mass spectra and less than a 30-second difference in retention time can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if there is a definitive inflection between the two peaks, according to the analyst's judgment. Otherwise, structural isomers are identified as isomeric pairs.

11.2 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and the public folder, *Arizona Calibration Training*.

11.3 Average Response Factor Calibration

The following formula is used to calculate the response factor for each analyte of interest relative to the applicable internal standard for each of the calibration standards:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s	=	Area of the characteristic ion for the target analyte in the calibration standard
A_{is}	=	Area of the characteristic ion for the internal standard
C_{is}	=	Concentration of the internal standard, (ng/mL)
C_s	=	Concentration of the target analyte in the calibration standard (ng/mL)

The calibration uses the average response factor for each target analyte, which is calculated as follows:

$$\text{average(mean) RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where:

RF_i	=	Response factor for the i^{th} calibration level
n	=	Number of calibration levels

The standard deviation for the mean RF for each target analyte is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

The relative standard deviation (RSD) for the average response factor for each target analyte is calculated as follows:

$$RSD = \frac{SD}{\overline{RF}} \times 100\%$$

The concentration of each target analyte in the sample extract is calculated using the average response factor that was calculated in the equation above as follows:

$$C_e = \frac{A_e \times C_{is}}{A_{is} \times \overline{RF}}$$

Where:

C_e	=	Concentration of target analyte in the sample extract, ng/mL
A_e	=	Area of the characteristic ion for the target analyte in the sample extract.
A_{is}	=	Area of the characteristic ion for the internal standard
C_{is}	=	Concentration of the internal standard, (ng/mL)
$\overline{RF}$	=	Average response factor for the target analyte as determined by calibration

11.4 Linear Least-Squares Regression Calibration (Unweighted)

A linear least-squares regression is performed using the concentration of the target analyte in the calibration standard as the independent variable (x) and the instrument response as the dependent variable (y). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where:

y	=	instrument response (e.g., peak area)
x	=	concentration of target analyte in calibration standard
m	=	slope of the line

b = intercept of the line

For the internal standard calibration, the regression equation is rewritten as follows:

$$\frac{A_s C_{is}}{A_{is}} = mC_s + b$$

Where:

A_s = Area of the characteristic ion for the target analyte in the calibration standard
A_{is} = Area of the characteristic ion for the internal standard
C_s = Concentration of the target analyte in the calibration standard, (ng/mL)
C_{is} = Concentration of the internal standard, (ng/mL)
m = slope of the line
b = intercept of the line

The concentration in an unknown extract is then calculated by rearranging the calibration equation as follows:

$$C_e = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{m}$$

Where C_e is the concentration of the target analyte in the sample extract, and A_e is the area of the characteristic ion for the target analyte in the sample extract.

The actual sample concentration (C) for each compound is calculated as follows:

$$C = C_e \times \left(\frac{V_e}{V_o}\right) \times DF$$

Where:

C = Concentration of the target analyte in the original sample, ng/L (water sample) or ng/kg (solid sample)
C_e = Concentration of the target analyte in the sample extract, ng/mL
V_e = Final extract volume, mL.
V_o = The original volume or weight of the sample that was extracted, in L (aqueous sample) or kg (solid sample).
DF = Dilution factor, if appropriate.

11.5 Additional Regression Calibration Models

As needed, weighted linear least-squares or second order regressions may be utilized for this analysis. See Corporate SOP CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and the public folder, *Arizona Calibration Training*, for calculations and further explanations.

11.6 A second-level technical review of the organic data is performed prior to data reporting. This review is performed by a peer or supervisor using the guidelines and checklists detailed in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until

that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.4 Retention Time Study

12.4.1 Expected absolute retention times (RTs) are initially determined by analyzing all target analytes in the open-scan mode. Example RTs are listed in Table V.

12.4.2 Relative retention times (RRTs) are then calculated for samples in each analytical run based on the RTs found in the continuing calibration verification standard (CCV).

12.4.3 RTs are re-established after any significant instrument maintenance, including source cleaning and changing columns, or whenever compounds are not adequately detected in CCVs or LCSs.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Methylene chloride solvent rinse waste – Waste Stream B

14.2.3 Expired extract vial waste – Waste Stream A

14.2.4 Radioactive and potentially radioactive waste must be segregated from non-radioactive and mixed waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Soil Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final update IIB, January 1995; Final Update III, December 1996, Final Update IV January 2008.

15.1.1 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.2 Method 8000C, Determinative Chromatographic Separations, Revision 2, February 2007.

15.1.3 Method 8000D, Determinative Chromatographic Separations, Revision 5, March 2018.

15.1.4 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, December 1996.

15.1.5 Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007.

15.1.6 Method 3510C, Separatory funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.7 Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.8 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

15.1.9 Method 3546, Microwave Extraction, Revision 0, February 2006.

15.2 CLP Statement of work for Multi-Media, Multi-Concentration Organics Analysis, SOM01.2. June 2007.

16.0 Method Modifications

16.1 The CLP SOW referenced in 8270D does not require the analysis of DFTPP prior to the analysis of samples. The method relies on the successful analysis of calibration standards to verify acceptable function of the mass spectrometer. TestAmerica Denver utilizes the DFTPP check to identify any operational issues with the mass spectrometer prior to the analysis of the calibration standards. This allows the analyst to identify possible problems independent of the GC. As a result, the laboratory will start the 12 hour clock with the injection of the DFTPP, not the calibration standard as required in the method.

16.2 Method 8270C serves as the basis for this SOP, but the method has been modified extensively for low-level analysis using selected ion monitoring (SIM) and optimizing instrument conditions for the low-level analysis. Consequently the sensitivity of the method has been enhanced and it is not uncommon to detect low-level contamination in the method blank at levels well below the limits of detection for the less sensitive GC/MS method. For example, Method 8270C states that the RSD of the initial and continuing calibration must be less than or equal to 15% and 20% respectively. Due to the low-level nature of the analysis, this SIM procedure allows both of these criteria to be less than or equal to 35%.

16.3 Method 8270C stipulates qualitative identification based on relative retention time (RRT), which is calculated by dividing the retention time (RT) of the target analyte by the RT of the internal standard. The RRT of the suspected target analyte in the sample extract must be within ± 0.06 RRT units of the RRT for that analyte in the calibration standard. This SOP stipulates qualitative identification based on an absolute RT. Namely the RT of the suspected target analyte in the sample extract must be within ± 0.2 minute of the RT for that analyte in the calibration standard. Additionally, the RT for the internal standard in the sample extract must also be within ± 0.2 minute of the RT for the internal standard in the calibration standard. The criteria used in this SOP are more restrictive than those imposed by the referenced method. For the earliest eluting compounds, the RT for the internal standard is typically 8 minutes. The earliest eluting target analyte must be at a RRT of at least 0.8, which translates to a RT of 6.4 minutes. Assuming a worst-case scenario where the RT of the internal standard is 0.2 minute higher (i.e., 8.2 minutes) and the RT of the target analyte is 0.2 minute lower (i.e., 6.2 minutes), the calculated RRT is 0.76. The total deviation from the expected RRT is 0.04 RRT units, which is smaller than what is allowed by Method 8270C.

17.0 Attachments

Table I:	Routine Instrument Operating Conditions
Table II:	Surrogates for Standard List Analysis
Table III:	Internal Standards for Standard List Analysis
Table IV:	PAH Compounds and Ions Used for Analysis
Table V:	Example Retention Times, IS and Surrogate Associations
Table VI:	DFTPP Key Ions and Ion Abundance Criteria for 8270C and 8270D
Table VII:	8270D Relative Response Factor Criteria for Initial and Continuing Calibration
Table VIII:	Specific DoD QSM 5.0 and DoE QSAS 3.0 Requirements for 8270D
Appendix I:	Extended List PAHs
Appendix II:	Suggested Instrument Maintenance Schedule – Mass Spectrometer & Gas Chromatograph
Appendix III:	Mass Spectrometer Settings for Single Ion Monitoring

18.0 Revision History

- Revision 13: 4 February 2019
  - Minor formatting and language changes throughout
  - Updated Section 2.2.2 to add language concerning Characteristic Ions
  - Added additional Characteristic Ions to Table IV and Appendix III
- Revision 12: 2 May 2018
  - Annual Review
- Revision 11: 28 February 2017
  - Added Section 4.4 to address contamination by carryover.
  - Added details on instruments currently used in Section 6.1.1.
  - Added supplies as Sections 6.2.8-6.2.10.
  - Revised standards information in Section 7.0 to reflect current TALS names in the reagent module and to reflect current practice regarding makeup of solutions.

- Added clarification for when NCM is written in lieu of reprep/reanalysis for failed LCS.
- Revised Section 9.4 on MS/MSD to reflect current policy.
- Added detail to surrogate corrective actions in Section 9.6.
- Revised volume of IS added in Section 10.4.2 to reflect current practice.
- Revised Section 12 to reflect current practice.
- Added list of injection volumes by method chain in Table I.
- Editorial and formatting changes throughout.

- Revision 10: 2 September 2015
  - Formatting and editorial changes throughout
  - Updated Section 9.4 on corrective action for MS/MSD to reflect current practice
  - Added requirement for Initial Calibration to be %RSD = $\pm$15%, or Linear regression or 2nd order to be $r^2 \geq 0.99$; CCV and ICV to be $\pm$ 20% to meet DOD 5 criteria in appropriate sections per DOD requirement that these must be explicitly stated in the SOP.
  - Added new Section 9.10 to address closing CCV required by DoD QSM 5.0.
  - Updated references to corporate SOP on Calibration Curves in Section 11.2 and 11.5.
  - Updated Sections 12.1-12.3 to reflect current practice.
  - Updated Table IV analytes and ions used for analysis
  - Added Appendix III to identify MS Settings for SIM for each compound
- Revision 9: 31 August 2014
  - Added Table 8, Specific DoD QSM 5.0 and DoE QSAS 3.0 Requirements for 8270 C or D
  - Added reference to DoD QSM 5.0
  - Modified the large volume injection (LVI) internal standard concentration to 600ng/mL
  - Added Appendix II, Suggested Instrument Maintenance Schedules
- Revision 8: 31 August 2013
  - Annual Technical Review
  - Added references to analysis by LVI
  - Updated Appendix I to reflect current practice
- Revision 7: 31 July 2012
  - Annual Technical Review
  - Grammatical and formatting changes throughout
  - Updated the quant ion for surrogate terphenyl-d14 to IS#2 in Table V
  - Updated Table 1 to match current GC conditions

Earlier revision histories have been archived and are available upon request.

Table I: Routine Instrument Operating Conditions

GC Conditions[1]	
Inlet	Split or Pulsed Split at 275 °C Split ratio - 3.1 : 1 Split Flow – 10.4 mL / min
Capillary Column	Varian Vf-5MS, 30 m length, 0.25 mm diam ID, 0.5 μm thickness
Column Mode	Constant flow, 3.4 mL/min
Temperature Program	Initial temp = 55 °C 30 °C/min ramp to 256 °C 4 °C/min ramp to 296 °C 30 °C/min ramp to 340 °C and hold for at least 1 minute past the elution time of the last compound.
Run Time	About 20 minutes with a new column.
Carrier Gas	Helium Purge flow = 25.0 mL/min, 3.00 min Total flow ≈ 31 mL/min
Injection Volume	Injection volume will be 1.0 µL or 5.0 µL depending on the logged method chain. 1. 8270C_SIM/3510C = 1.0 µL 2. 8270C_SIM/3510C_LVI = 5.0 µL 3. 8270D_SIM/3510C = 1.0 µL 4. 8270D_SIM/3510C_LVI = 1.0 µL 5. 8270D_SIM_DOD5/3510C = 1.0 µL 6. 8270D_SIM_DOD5/3510C_LVI = 1.0 µL 1.0 µL injection uses Standard Method Calibration standards (Section 7.2.2) 5.0 µL injections uses LVI Method Calibration standards (Section 7.2.2)
Transfer Line	290 °C or 300 °C
Mass Spectrometer Conditions[1,2]	
MS Source	230 °C or 240 °C
MS Quadrupole	200 °C
Dwell Time per Ion	Ranges from 30 to 100 milliseconds
Ions	See following tables

1 The conditions listed above are subject to final fine adjustments to maximize instrument sensitivity. Changes to the above conditions are acceptable as long as method criteria are met.

2 Details on the mass assignments in each window along with start and dwell times are given in Appendix III.

Table II: Surrogates for Standard List Analysis

PAH Surrogates	Mass Ion	Confirmation Ion
Nitrobenzene-d_5	82	128
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122

Table III: Internal Standards for Standard List Analysis

Compound	Mass Ion	Confirmation Ion
Acenaphthene-d_{10}	164	162
Phenanthrene-d_{10}	188	94
Chrysene-d_{12}	240	120

Table IV: PAH Compounds and Ions Used for Analysis

Compound	Mass Ion	Confirmation Ions
Acenaphthene	153	152, 154
Acenaphthylene	152	151, 153
Anthracene	178	179,176
Benzo(a)anthracene	228	226, 229
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139, 84
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Indeno(1,2,3,cd)pyrene	276	138, 277
1-Methylnaphthalene	142	141, 115
2-Methylnaphthalene	142	141, 115
Naphthalene	128	129, 127
Phenanthrene	178	179, 176
Pyrene	202	101, 200
Morpholine	57	87

Table V: Example Retention Times, IS and Surrogate Associations

Compound	RT[1] (min.)	IS #	Surrogate #
Morpholine	4.001	1	1
Naphthalene	5.921	1	1
2-Methylnaphthalene	6.595	1	1
1-Methylnaphthalene	6.700	1	1
Acenaphthylene	7.512	1	2
Acenaphthene	7.686	1	2
Dibenzofuran	7.861	1	2
Fluorene	8.210	1	2
Phenanthrene	9.194	2	2
Anthracene	9.255	2	2
Fluoranthene	10.768	2	2
Pyrene	11.166	2	2
Benzo(a)anthracene	13.827	3	3
Chrysene	13.924	3	3
Benzo(b)fluoranthene	17.004	3	3
Benzo(k)fluoranthene	17.089	3	3
Benzo(a)pyrene	18.034	3	3
Indeno(1,2,3,cd)pyrene	21.509	3	3
Dibenz(a,h)anthracene	21.583	3	3
Benzo(g,h,i)perylene	22.306	3	3
Acenaphthene-d_{10} (IS)	7.657	1	-
Phenanthrene-d_{10} (IS)	9.177	2	-
Chrysene-d_{12} (IS)	13.856	3	-
Nitrobenzene-d_5 (Surr)	5.201	1	1
2-Fluorobiphenyl (Surr)	6.945	1	2
Terphenyl-d_{14} (Surr)	11.38	2	3

[1]Retention times may vary depending upon chromatographic conditions.

Table VI: DFTPP Key Ions and Ion Abundance Criteria
8270C

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	< 2% of mass 69
69	Mass 69 relative abundance
70	< 2% of mass 69
127	40 - 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass ion 198
275	10 - 30% of mass 198
365	> 1% of mass 198
441	Present, but less than mass 443
442	40 - 100% of mass 198
443	17 - 23% of mass 442

With the exception of mass 442, the tune criteria for SW846 method 8270D are less stringent for the criteria required in SW846 method 8270C. For 8270D, the 442 mass must be greater than 50% of mass 198 to meet the tune criteria. By using the 8270C criteria, the rest of the data will be within the 8270D criteria.

Table VII: 8270D Relative Response Factor Criteria for Initial and Continuing Calibration

Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Acenaphthene	0.900	20	25
Acenaphthylene	0.900	20	25
Anthracene	0.700	20	25
Benzo(a)anthracene	0.800	20	25
Benzo(a)pyrene	0.700	20	25
Benzo(b)fluoranthene	0.700	20	25
Benzo(g,h,i)perylene	0.500	20	25
Benzo(k)fluoranthene	0.700	20	25
Chrysene	0.700	20	25
Dibenzo(a,h)anthracene	0.400	20	25
Dibenzofuran	0.800	20	25
Fluoranthene	0.600	20	25
Fluorene	0.900	20	25
Indeno(1,2,3,cd)pyrene	0.500	20	25
1-Methylnaphthalene	0.400	20	25
2-Methylnaphthalene	0.400	20	25
Naphthalene	0.700	20	25
Phenanthrene	0.700	20	25
Pyrene	0.600	20	25

Table VIII
Specific DoD QSM 5.0 and DOE QSAS 3.0 Requirements for 8270D

This table includes components of the DoD QSM 5.0 and DoE QSAS 3.0 that are different from TestAmerica's standard procedures. For a complete description of the requirements, see DV-QA-024P. Also listed are the variances that TestAmerica is requesting for this analysis; these alternate criteria are only used with project-specific approval

Requirement	Variance (if allowed)	DoD QSM 5.0 and DoE QSAS 3.0
Initial Calibration Verification (ICV)	--	All analytes must be within ± 20% of the true value.
	4PP	Allow ± 30% of true value for known poor performers only if these compounds are not identified as critical compounds of concern by the client for the project under consideration.
Continuing calibration Verification (CCV)	--	Run before sample and at the end of the analytical batch (end of 12 hours). Acceptance limits for all analytes is ± 20% of true value for CCV at start of 12 hours.
	4PP	Allow ± 30% of true value for known poor performers only if these compounds are not identified as critical compounds of concern by the client
	3HR	If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project
	7MS	Allow ± 50% for end of analytical batch excluding poor performing compounds. Reanalysis performed due to failed closing CCV only for the analytes identified by the client as critical compounds of concern for the project, and to report qualified results for other analytes.
		If any analytes fail in a CCV, recalibrate and re-analyze all affected samples or immediately (within one hour) analyze two consecutive CCVs and if both pass for the analytes that failed, the CCV is acceptable.
Internal Standards (IS)	--	RT must be ± 10 seconds from RT of the midpoint standard in the ICAL
	8ISRT	RT must be ± 30 seconds from RT of the midpoint standard in the ICAL. Daily routine column maintenance often results in larger RT changes than 10 sec. within a short time.
LCS	--	Include all analyte(s) in LCS that are required to be reported, including surrogates, except those compounds listed as "Additional Analytes" by TestAmerica. These compounds are rarely requested and historical limits may not accurately reflect current performance.
	4PP	If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project
	3HR	Otherwise, correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch. Flagging is only appropriate when samples cannot be reanalyzed unless 3HR is accepted by the client.
	1SME	Marginal exceedances are not allowed for critical chemicals of concern (risk drivers). Client must notify TestAmerica of these targets or if marginal exceedances will not be allowed.
Surrogates	--	For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.
	4PP	If surrogate recoveries are above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project.

Appendix I: Extended List PAH Analysis by GC/MS

Summary of Method

This is the extended list for the SIM analysis that some clients require. All of the compounds listed in this appendix are analyzed for in addition to the standard compounds discussed throughout this SOP.

Modifications from the SIM analysis are as follows:

- The DFTPP tune has tailing factors that are calculated for Pentachlorophenol and Benzidine and a DDT breakdown check is performed.
- The instrument is calibrated at eight concentration levels. The calibration levels are made by diluting two stock standards with concentrations of 20 µg/mL [PAHXSIM stock (#1)] and 2 µg/mL [PAHXSIM 2º stock (#2)] down to the concentrations listed below, in methylene chloride. All phthalate compounds and 2-methylnaphthalene are at a ratio of 2:1 in the stock standards. Therefore, if the concentration is 0.02 µg/mL for the target analytes, the phthalates are at 0.04 µg/mL.

Level (µg/mL)	Stock ID	Stock Amt (µL)	Solvent amount (µL)	IS amount (µL)	Final Volume (µL)
0.02 µg/mL	#2	5	495	50	500
0.1 µg/mL	#2	25	475	50	500
0.3 µg/mL	#2	75	425	50	500
0.6 µg/mL	#1	15	485	50	500
1.2 µg/mL	#1	30	470	50	500
2.5 µg/mL	#1	62.5	437.5	50	500
5.0 µg/mL	#1	125	375	50	500
10.0 µg/mL	#1	250	250	50	500

Response factors for each compound must be ≤ 20% RSD. If any compound is > 20% RSD, must use the best curve fit.

Initial Calibration Verification

- The second source calibration stock is also at 20 µg/mL (PAHSIM SSV stock).
- The second source verification (SSV or ICV) is analyzed at 1.2 µg/mL.
- The acceptance criterion for the ICV is ± 25%D.

Continuing Calibration Verification

- The CCV is run at 0.6 µg/mL
- The criterion: The Average %D for all compounds must be < 20 %D, with no single compound exceeding 30 %D.

Sample extraction: See DV-OP-0008 (aqueous) and DV-OP-0009 (soil).

Sample concentration: See DV-OP-0007.

Sample analysis:

- Internal Standard final concentration is 6 µg/mL in standards and extracts. The stock is at 400 µg/mL
- For the MS/MSD, the recovery for the spike pair must be within the control limits stored in TALS. The MS/MSD pair is generally aliquotted and run two times on the instrument, to confirm the results. If the results to be reported are from the first analysis, it is not required that the second analysis be within the 12 hour tune clock.

Instrument Configuration:

The GCMS instrumentation is configured the same as in the SIM analysis.

Extended List Compounds, Reporting Limits and Ions Used for Analysis:

Compound	Water Reporting Limit (ng/L)	Soil Reporting Limit (µg/kg)	Mass Ion	Confirmation Ion
1,4-Dioxane	NA	20	88	58
N-Nitrosodiphenylamine	1,000	20	169	168
N-Nitrosodimethylamine	400	18	74	42
N-Nitrosodiethylamine (LVI)	100	--	102	44
N-Nitrosodi-n-propylamine (LVI)	100	--	70	42
Butyl Benzyl Phthalate	1,000	20	149	91
Dimethyl Phthalate	1,000	20	163	164
Diethyl Phthalate	1,000	20	149	177
Bis(2-Ethylhexyl) Phthalate	1,000	20	149	167
Di-n-octyl Phthalate	1,000	20	149	150
Di-n-butyl Phthalate	1,000	20	149	150

Extended List Compounds Example Retention Times, IS and Surrogate Associations:

Compound	RT[1] (min.)	IS #	Surrogate #
1,4-Dioxane	1.60	1	2
N-Nitrosodiphenylamine	6.75	2	2
N-Nitrosodimethylamine	2.16	1	2
N-Nitrosodiethylamine (LVI)	2.72	1	1
N-Nitrosodi-n-propylamine (LVI)	3.69	1	1
Butyl Benzyl Phthalate	10.33	2	2
Dimethyl Phthalate	5.92	1	2
Diethyl Phthalate	6.51	1	2
Bis(2-Ethylhexyl) Phthalate	11.67	2	2
Di-n-octyl Phthalate	13.69	3	2
Di-n-butyl Phthalate	7.95	2	2
Acenaphthene-d_{10} (IS)	7.657	1	-
Phenanthrene-d_{10} (IS)	9.177	2	-
Chrysene-d_{12} (IS)	13.856	3	-
Nitrobenzene-d_5 (Surr)	5.201	1	1
2-Fluorobiphenyl (Surr)	6.945	1	2
Terphenyl-d_{14} (Surr)	11.38	2	3

[1]Retention times may vary depending upon chromatographic conditions.

APPENDIX II

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	**Weekly**	**As Needed**	**Quarterly**	**Annually**
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43).	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		Change the oil in the mechanical rough pump.
Check inlets, septa.		Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

APPENDIX II (continued)

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	***As Needed***
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

APPENDIX III
Mass Spectrometer Settings for Single Ion Monitoring

Group ID	Group Start Time[1] (min)	Analyte	Masses	Dwell Times
1	1.45	N-Nitrosodimethylamine	74, 42	50, 50
		1,4-Dioxane	88, 58	50, 50
		Morpholine[2]	57, 87	50, 50
		N-Nitrosodiethylamine (LVI)[3]	102, 44	50, 50
2	2.60	Nitrobenzene-d_5	82, 128	50, 50
		Naphthalene	128, 129, 127	50, 50
		N-Nitrosodiethylamine (LVI)[3]	102, 44	50, 50
		N-Nitrosodi-n-propylamine (LVI)	70, 42	50, 50
3	4.79	2-Fluorobiphenyl	172, 171	50, 50
		2-Methylnaphthalene	142, 141, 115	50, 50
		1-Methylnaphthalene	142, 141, 115	50, 50
4	5.46	Dimethyl Phthalate	163, 164	50, 50
		Acenaphthene- d_5	164, 162	50, 50
		Acenaphthene	153, 152, 154	50, 50
		Acenaphthylene	152, 151, 153	50, 50
		Dibenzofuran[4]	168, 139, 84	50, 50
5	6.06	Diethyl Phthalate	149, 177	50, 50
		N-Nitrosodiphenylamine	169, 168	50, 50
		Fluorene	166, 165, 167	50, 50
		Dibenzofuran[4]	168, 139, 84	50, 50
6	6.78	Phenanthrene-d_{10}	188, 94	50, 50
		Phenanthrene	178, 179, 176	50, 50
		Di-n-butyl Phthalate	149, 150	50, 50
		Anthracene	178, 179, 176	50, 50
7	8.05	Butyl Benzyl Phthalate	149, 91	50, 50
		Terphenyl-d_{14}	244, 122	50, 50
		Fluoranthene	202, 101, 203	50, 50
		Pyrene	202, 101, 200	50, 50
8	10.48	Chrysene- d_{12}	240, 120	50, 100
		Bis(2-Ethylhexyl) Phthalate	149, 167	100, 100
		Chrysene	228, 226, 229	50, 50
		Benzo(a)anthracene	228, 226, 229	50, 50
9	12.33	Di-n-octyl Phthalate	149, 150	50, 50
		Benzo(a)pyrene	252, 253, 125	50, 50
		Benzo(b)fluoranthene	252, 253, 125	50, 50
		Benzo(k)fluoranthene	252, 253, 125	50, 50
10	16.48	Dibenzo(a,h)anthracene	278, 139, 279	50, 50
		Indeno(1,2,3-cd)pyrene	276, 138, 277	50, 50
		Benzo(g,h,i)perylene	276, 138, 277	50, 50

[1]Group start times may vary due to chromatographic conditions.
[2]Morpholine method detection limit verifications not kept current. Laboratory does not stock standards.
[3]N-Nitrosodiethylamine (LVI) elutes between windows 1 and 2 and was therefore included in both.
[4]Dibenzofuran elutes between windows 4 and 5 and was therefore included in both.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Determination of Volatile Organics by GC/MS [8260B/C/D and 624/624.1]

Approvals (Signature/Date):

Jonathan Waldorf 2/14/19 Technical Manager Date	Doug Gomer 2/14/19 Health & Safety Manager / Coordinator Date
Roxanne Sullivan 2/14/18 Quality Assurance Manager Date	Richard Clinkscales 2/14/19 Laboratory Director Date

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Table 1. Additional analytes that can be determined by this SOP are listed in Tables 2, 3 and 4.

1.2 This SOP is applicable to Method 8260B/C/D, which is appropriate for compliance testing under RCRA regulations and Method 624/624.1 (CWA compliance testing). It is important that the procedural differences described in this document for these methods are carefully observed.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 60 µg/L for 8260B/C/D waters, 1.0 to 200 µg/kg for low-level soils, and 200 to 30,000 µg/kg for medium-level soils. The working range for Method 624/624.1 (5 mL purge) is 1.0-200 µg/L.

1.5 Reporting limits for Method 8260B/C/D are listed in Tables 1, 2, and 3. Reporting limits for Method 624/624.1 are given in Table A1. Reporting limits for soil samples prepared by the AK methanol technique are listed in Table Bp-1.

1.6 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

2.0 Summary of Method

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved in water (with or without sodium bisulfate) and purged directly.

2.3 In the purge-and-trap process, an inert gas is bubbled through the solution at ambient temperature or at 40 °C (40 °C is required for low-level soils), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each

identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

2.5 Unless otherwise noted all requirements are for both 8260B, 8260C, 8260D and 624/624.1.1

3.0 Definitions

3.1 Contaminated glassware: Glassware that may have come into contact with samples and/or chemical standard and that have not been cleaned using this procedure.

3.2 Uncontaminated glassware: Glassware that has not come into contact with samples, chemical standards, or is otherwise believed to be uncontaminated.

NOTE: Newly purchased glassware is not necessarily clean. The manufacturing process can contaminate glassware with high molecular weight organic constituents that can be extracted by organic solvents. New glassware should be thoroughly Methanol rinsed prior to use for analytical applications.

3.3 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and percent drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.4 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.5 Initial Calibration Verification (ICV)

The ICV is a second-source calibration verification standard. In this SOP, the LCS and the MS/MSD spikes are second-source standards.

3.6 Continuing Calibration Verification (CCV)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.7 Lower Limit of Quantitation (LLOQ)

The lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be $\geq$ the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit. The required LLOQ verification is performed at a concentration of 1-2 times the LLOQ (or RL).

3.8 Other Quality Terms

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and in SOP DV-QA-003P, Quality Assurance Program.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2 It is imperative that all glassware used in the laboratory be free of contaminants and potential interferences before starting and analysis or extraction process. Many problems encountered in the laboratory can be traced to improperly cleaned glassware and can be avoided by following the proper cleaning procedures.

4.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.4 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.5 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.6 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample. (See Section 10.7.4.12.)

4.7 Interferences are observed with the surrogate Toluene-d_8 when the samples appear to be treated with potassium permanganate.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3.4 Inspect all glassware before use and remove from service any glassware that is chipped or broken.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.1.2 Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.1.3 Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax / Silica gel / Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.

6.1.4 Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.

6.1.5 Sample Heater: A heater capable of maintaining the purge device at 40 °C is necessary for low level soil analysis.

6.1.6 Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial, Varian Archon, or equivalent.

6.1.7 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.1.8 Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.1.8.1 Column 1: 60 m X 0.25 ID DB-624/624.1 with 1.4 µm film thickness.

6.1.8.2 Column 2: 75 m X 0.53 ID DB-624/624.1 wide bore with 3 µm film thickness.

6.1.9 Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.1.10 GC/MS interface: In general, glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.1.11 Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion

abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Microsyringes: 10 μL and larger, 0.006-inch ID needle.

6.3.2 Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

6.3.3 Balance: Analytical balance capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.3.4 Vials: 2 mL, 20 mL, and 40 mL with screw caps and Teflon liners

6.3.5 Disposable magnetic stirrers for low-level soil analyses

6.3.6 Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.3.7 Spatula: Disposable wooden tongue depressors.

6.3.8 Disposable pipettes: Pasteur.

6.3.9 pH paper: Wide range.

6.3.10 Gases:

6.3.10.1 Helium: Ultra high purity, grade 5, 99.999% (carrier gas).

6.3.10.2 Compressed nitrogen: Used for instrument pneumatics and sample purge.

7.0 Reagents and Standards

7.1 Methanol: Purge and Trap Grade, High Purity

7.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.4.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.3 Sand: Reagent grade Ottawa sand or equivalent.

7.4 Antifoam B, Silicon Emulsion, J. T. Baker, 100% purity.

7.5 Sodium bisulfate ($NaHSO_4$), reagent grade

7.6 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers' expiration date.

7.7 Calibration Stock Standard Solutions

Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10 to -20 °C. Stock standards and aliquots for gases must be replaced at least every week. The Gas Standards Tracking Log is used to verify and track open dates to assist in weekly replacement of the gas standards. See Attachment 1. Other stock standards must be replaced at least every 6 months.

7.8 Calibration Working standards

A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.9 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.10 Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Tables 8 and 8A for internal standard components.

7.11 Surrogate Standards: Refer to Tables 9 and 9A for surrogate standard components and spiking levels.

7.12 Laboratory Control Sample Spiking Solutions: A second source set of standards is used and contains the Main_B, Gas/Ket_B, and SS-2-Cleve. Compounds not included in these standards can be spiked with lab pre-approval. See TALS Reagent Module for these standards for complete list of target analytes.

7.13 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS.

7.14 Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of BFB in methanol is prepared from stock standards as described in Sections 7.7 and 7.8.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples

8.1.1 Water samples are collected in triplicate in 40 mL glass VOA vials with PTFE-lined septum caps with minimal headspace. There should be no bubbles present in the container larger than ~6 mm.

8.1.1.1 Analysts will select the VOA vial without a red sticker for analysis if possible. If all vials contain red stickers then the VOA vials with the smallest headspace/bubbles will be chosen for analysis. Any VOA vials analyzed with a red sticker (headspace >6mm) will be documented in an NCM.

8.1.2 Preservation depends upon the target analytes and the sampling location. At a minimum, aqueous samples are stored refrigerated at ≤ 6 °C and not frozen. Specific preservation requirements are given in the following table. If multiple analytes are requested, it may be necessary to provide aliquots with different preservations. For each preservation technique, the samples should be collected in triplicate.

8.1.3 SW-846 states that if carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. The holding time for these unpreserved samples is 7 days. SW-846 does not otherwise provide guidance for processing unpreserved samples. EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

Preservation and Holding Time for Volatiles in Water

Analyte(s)	Reference	Preservation	Holding time	Dechlorination Required[1]
Routine target analytes[2]	SW-846, Ch. 4	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	SW-846, Ch. 4	Cool, ≤6ºC	7 days	Y
	624/624.1	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	624/624.1	Cool, ≤6ºC	7 days	Y
Acrolein[3]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	40 CFR Part 136 (624/624.1)	Cool, ≤6ºC (no HCl)	3 days	Y
	40 CFR Part 136 (624/624.1)	Cool, ≤6ºC, pH 4-5	14 days	Y
Acrylonitrile[3]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	40 CFR Part 136 (624/624.1)	Cool, ≤6ºC (no HCl)	14 days	Y
	40 CFR Part 136 (624/624.1)	Cool, ≤6ºC, pH 4-5	14 days	Y
2-Chloroethylvinyl ether (2-CLEVE)[4]	SW-846, Ch. 4	Cool, ≤6ºC (no HCl)	7 days	Y
	624/624.1	Cool, ≤6ºC (no HCl)	14 days	Y

[1] If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added. *Never add acid preservative directly to a dechlorinating agent prior to sample collection.*

[2] Separate aliquots must be collected and preserved as indicated if acrolein, acrylonitrile, 2-CLEVE (by Methods 8260B/C/D or 624/624.1), vinyl chloride (by Method 8260B/C/D) or styrene (by Method 8260B/C/D) are also to be analyzed. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary.

[3] SW-846 only recommends a 7 day holding time for acrolein and acrylonitrile preserved to pH 4-5. As this preservation is difficult to achieve with HCl, the laboratory recommends unpreserved aliquots with a three day holding time.

According to 40 CFR Part 136, the preferred method for acrolein and acrylonitrile is Method 603. In the Method Update Rule published in the Federal Register on May 18, 2012 (40 CFR Parts 136) EPA approved Method 624/624.1 for the determination of acrolein and acrylonitrile in wastewater. The current sample preservation and holding time requirements for acrolein and acrylonitrile apply to these compounds when analyzed by Method 624/624.1. Implementation of this rule is subject to individual state program decisions and timetables.

[4] SW-846 includes vinyl chloride and styrene in the list of compounds which require unpreserved sample for analysis. Method 624/624.1 does not include these two analytes on the standard analyte list. The laboratory recommends that if these analytes are requested by Method 624/624.1 that unpreserved aliquots be submitted.

8.2 Soil Samples

8.2.1 Sampling Techniques and Containers

8.2.1.1 Soil samples can be taken using the EnCore™ sampler. Typically three Encores are collected per sampling location. At specific client request, unpreserved soil samples may be accepted for preservation at the lab.

8.2.1.2 The more common way to collect soils is with Terra Core kits. Typically three aliquots are collected. Terra Core kits consist of the Terra Core sampling device and three 40 mL tared VOA vials. Samples are extruded into empty tared vials and preserved at the laboratory or are extruded into pre-preserved, tared vials in the field and transported to the laboratory for analysis.

8.2.2 An additional bottle of unpreserved soil for each sampling location must be shipped for percent moisture determination.

8.2.3 A second bottle of unpreserved soil is sent for screening.

8.2.4 Unpreserved Soils

8.2.4.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and sub sampled in the laboratory. This is the old procedure based on Method 5030A. It is no longer included in subsequent revisions of Method 5030 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.2.4.2 The maximum holding time is 14 days from sampling until the sample is analyzed. Unpreserved samples should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative.

8.3 Trip blanks, consisting of laboratory prepared water samples with acid preservative, are also provided when bottles are supplied by the laboratory to the field. Trip blanks are used for both water and soil samples to monitor potential contamination from volatile compounds in transit and in the field.

8.4 A holding blank is stored in each refrigerator with the samples. This is analyzed every 14 days (see SOP DV-QA-0013).

8.5 Preservation and holding times for volatiles in soils are summarized in the following table, based on SW-846 Method 5035A.

Preservation and Holding Time for Volatiles in Soil
Method 5035A

Sample Handling in Field	Preservation[1,2,3]	Holding Time
Sample Extruded into Empty Vial in Field, sealed and shipped to Lab	Cool to ≤6°C or freeze on site to -7°C in field (do not freeze below -20°C), maintained cold in transit Upon receipt the laboratory must: a) Maintain the sample frozen or b) Add 5 mL water and freeze (-20°C to -7°C), or c) Add 5 mL water and 1 g $NaHSO_4$, store at ≤6°C or d) Add methanol, store at ≤6°C	48 hours from sampling to other preservation 14 days from sampling
Encore sampler used for transport	Cool to ≤6°C or freeze on site to -7°C in field (do not freeze below -20°C), maintained cold in transit Upon receipt at laboratory sample is extruded to a sealed vial and either frozen to <-7°C or chemically preserved (see above).	48 hours from sampling to other preservation 14 days from sampling
Field Preserved	Sample is extruded into a vial containing reagent water and frozen on –site to <-7°C	14 days
Field Preserved	Sample is extruded into a vial containing reagent water and cooled on –site to ≤6°C for 48 hours or less then frozen to <-7°C upon receipt at laboratory	14 days if samples frozen within 48 hours
Field Preserved	Sample is extruded into a vial containing reagent water and 1 g NaHSO4 and cooled to ≤6°C. Maintained ≤6°C upon receipt at laboratory.	14 days
Field Preserved	Sample is extruded into a vial containing methanol and cooled to ≤6°C. Maintained ≤6°C upon receipt at laboratory.	14 days

[1] If multiple aliquots of sample are provided a combination of preservation techniques may be used

[2] Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.

[3] For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Proper cleaning of laboratory glassware is an essential element of contamination control and therefore an important element of the laboratory's quality control program. Proper cleaning of glassware is reflected in acceptable method blanks results.

9.2.1 Gastight syringes must be triple rinsed with Methanol and air dried.

9.2.2 Class A volumetric flasks must be triples rinsed before and after each use and air dried. **Note:** Do not bake or use wire brushes to clean Class A glassware.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples (method blank, lab control sample, and matrix spike/matrix spike duplicate), processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. A method blank must be run on each instrument. See Policy DV-QA-003P for further details.

9.4 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is Ottawa sand preserved with DI water or Sodium Bisulfate depending on how the samples are preserved. For medium-level volatiles, the method blank consists of Ottawa sand to which surrogates are added and extracted with methanol. The method blank is carried through the entire preparation and analytical procedure.

NOTE: If both DI and sodium bisulfate preserved samples are analyzed the QC samples should be prepared with sodium bisulfate.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

For DOD QSM 4.2 or 5.0, common lab contaminants must not exceed the reporting limit.

The method blank must have acceptable surrogate recoveries. (See Section 9.5)

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Corrective Actions: If the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone, and carbon disulfide), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than one-half the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all

associated samples are flagged with a "B", and appropriate comments may be made in the narrative to provide further documentation.

9.5 Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 8A.

For DoD QSM 4.2 or 5.0, special method codes in TALS include program specific QC limits for surrogates. If limits are not available in the appropriate QSM, in-house historical limits are to be used.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean. Surrogate recovery limits are updated annually and stored in the LIMS.

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.6 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS spiking solution is prepared from a different source than are the calibration standards. See Section 7.12 for description of spiking solution. For low-level volatiles in soil, the LCS medium is Ottawa sand preserved with DI water or

sodium bisulfate depending on how the samples are preserved. For medium-level volatiles, the LCS consists of Ottawa sand to which surrogate and LCS spiking solutions are added and extracted with methanol. The LCS is carried through the entire preparation and analytical procedure. If antifoam agent is added to samples it must also be added to the LCS.

NOTE: If both DI and sodium bisulfate preserved samples are analyzed the QC samples should be prepared with sodium bisulfate.

NOTE: DoD and South Carolina require that all target compounds are spiked and reported. Specific reporting requirements for spiked compounds must be in the method comments for the job. Each project must be set up to include all spiked compounds to be reported. (NELAP requires that all targeted compounds be spiked across a two year period in the MS/MSD.)

Acceptance Criteria: The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Recovery limits are updated annually and stored in the LIMS. For DoD QSM 4.2 or 5.0, special method codes in TALS include program specific QC limits. If limits are not available in the appropriate QSM, in-house historical limits are to be used.

If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAP. DoD QSM 5.0 allows marginal exceedances but excludes them from being applied to specified critical compounds of concern. Marginal exceedances must not be applied to an analyte specified by the project team as a compound of concern.

The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Additional criteria are stated in the North Carolina QAS.

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QAS's in the public folders for the current requirements.

Note: DoD allows marginal exceedances except for critical compounds of concern. These must be specified in the communication of project requirements to the lab.

Corrective Actions: If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If samples are ND and LCS recovery for an analyte is out high, the sample may be reported with a qualifier, if acceptable to the client.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.7 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. See Section 7.13 for description of spiking solution. The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

NOTE: DoD and South Carolina require that all target compounds are spiked and reported. Specific reporting requirements for spiked compounds must be in the method comments for the job. Each project must be set up to include all spiked

compounds to be reported. (NELAP requires that all targeted compounds be spiked across a two year period in the MS/MSD.)

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated annually and stored in the LIMS.

DoD QSM 5.0 requires the RPD limit to be ≤ 20%. DoD MS/MSD recovery limits are the same as LCS limits. See Section 9.6.

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- When the parent sample and MS/MSD concentration are above the linear range; the MS/MSD and parent sample **MUST** be re-analyzed at a dilution.
- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits and note it on the final report.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this request.

NOTE: A sample duplicate is not performed as precision is obtained from either the MS/MSD or LCS/LCSD pair. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Any samples that have target analytes at such low levels do not provide useful precision data.

9.8 Acid Preservation or pH adjustment

The stability of 2-chloroethylvinyl ether, acrolein, and according to the regulations, acrylonitrile is reduced when subjected to low pH. It is therefore not recommended that these compounds be analyzed routinely from preserved VOA vials and since there is no reasonable way to achieve pH between 4 and 5, it is recommended that unpreserved vials be used for the analysis of these compounds.

Acceptance Criteria: To ensure detection of these compounds, samples must be processed correctly. Where Method 624/624.1 is being used for compliance purposes, the regulatory hold times take precedence.

Corrective Actions: If 624/624.1 data are not being generated for compliance purposes, the technical stability of the compounds may be considered. Where method 8260 is the base method, it is allowable to qualify the results as estimated. To deviate from the regulatory hold times, the following documentation must be maintained:

- A NCM must be generated by the lab that the samples are for non-compliance.
- A NCM must be generated that results are not method compliant.
- A NCM must be generated if the samples are not analyzed from an upreserved container.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preservation using EnCore™ Samplers.

10.3.1 Preservation in Methanol (Medium-Level Analysis)

10.3.1.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the

vial and record it in the prep batch. Select the correct cell in prep batch worksheet and once the balance is stable hit the print key. The weight will automatically be recorded in the batch record. Quickly add 5 mL of methanol and cap the vial.

10.3.1.2 If sufficient samplers are provided (or for the sample(s) designated by the client), prepare MS and MSD samples as above.

10.3.1.3 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.3.1.4 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.2 Preservation in Water (Low-Level Analysis)

10.3.2.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it in the prep batch. Select the correct cell in prep batch worksheet and once the balance is stable hit the print key. The weight will automatically be recorded in the batch record. Quickly add 5 mL of water and a magnetic stirrer. Cap the vial. Repeat for the remaining aliquot.

10.3.2.2 If requested by the client, 1 g of sodium bisulfate is added to the second sample preserved with water.

10.3.2.3 If sufficient samplers are provided for a sample in the batch, or for any samples identified by the client, prepare MS and MSD samples as above.

10.3.2.4 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of water to the MB and LCS. Add a magnetic stirrer. Cap tightly. Store with the samples.

10.3.2.5 After all of the samples in the prep batch have been weighed and preserved in water the status of the samples and QC in the prep batch will remain at “batched” status for backlog tracking purposes.

10.3.2.6 When the samples are loaded on the instrument for analyses then the analyst can change the status of the samples and QC to “2nd level” so that the prep method’s status will change from “Ready” to “2nd level” therefore changing the status of the analytical method from “wait” to “Ready”

10.3.2.7 Store the samples and QC samples in the freezer until screening is performed.

10.3.3 Screen the samples. (See Section 10.5.) If the screen indicates any samples will be analyzed as medium level, go to Section 10.5.1. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.4 Sample Storage for Field Preserved Samples

10.4.1 Obtain the weight of the soil added to each vial and record it in TALS. The samples will arrive with a tare weight (weight of vial, preservative and stir bar) and no other labels attached to it. The unlabeled vial is weighed and the difference between this weight and the tare weight is the weight of the sample used for analyses.

NOTE: Do not affix label to unprepared/unweighed sample vial.

10.4.1.1 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into tared VOA vials for each analysis method. The low level preparation is done in a 40mL VOA vial and the medium level preparation is done in a 20mL VOA vial.

10.4.1.1.1 For the medium level method, in a 20mL VOA vial add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.4.1.1.2 For the low level method in a 40ml VOA vial add water instead of methanol using the same volumes as in Section 10.4.1.1.1.

10.4.1.2 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.4.2 Screen the samples. (See Section 10.5) If the screen indicates any samples will be analyzed as medium level, go to Section 10.7.5. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.5 Sample Screening

10.5.1 Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. See SOP DV-MS-0009. Alternatively, an appropriate aliquot can be determined from sample histories.

10.6 Sample Preparation for Medium-Level Analysis – Field or Lab Preserved

10.6.1 For each of the samples that are determined to be Medium-Level samples by the screening procedure, add the correct amount of surrogate spiking mixture for a

final concentration of 2 μg/mL. Example: 4 μL of 2500 μg/mL for a nominal 5 g sample or 20 μg/mL for a nominal 25 g sample. Cap the sample vial. Surrogates are added to all QC samples as well as field samples.

10.6.2 Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples for a final concentration of 2 μg/mL.

10.6.3 Add the correct amount of matrix spiking solution to the LCS sample for a final concentration of 2 μg/mL. If 25 g samples are being used, adjust the proportions for the LCS accordingly.

10.6.4 Shake the samples for two minutes to distribute the methanol throughout the soil.

10.6.5 Centrifuge the samples to clarify the extract.

10.6.6 Remove a portion of methanol and store in a clean Teflon-capped vial with no headspace refrigerated at $\leq$ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

10.7 Sample Analysis Procedure

10.7.1 All analysis conditions for samples must be the same as for the initial and continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

10.7.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a method blank, an LCS, and a MS/MSD.

10.7.2.1 If there is insufficient time in the 12-hour 624/624.1 period to analyze 20 samples, the batch may be continued into the next tune period. The 12-hour tuning requirements in Section 10.7.12.3 and 12-hour continuing calibration requirements in 10.7.14 must still be met. However, if any re-tuning or recalibration of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new QC batch must be started. For high-level soils the batch is defined at the sample preparation stage. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

10.7.2.2 Any reruns must be part of a valid analytical batch. If dilutions of sample are analyzed in the same 12-hour tune they do not count towards the maximum batch count. (See DV-QA-003P.)

10.7.3 Water Samples

10.7.3.1 Purge-and-trap units that sample from a VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.3.2 All samples and standard solutions must be at ambient temperature before analysis.

NOTE: Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

10.7.3.3 To transfer a sample from its original container, fill a gas-tight syringe with the sample and adjust the sample volume based on the requested method. Place the measured sample into a clean VOA vial.

10.7.3.3.1 For Method 8260B, 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.) Sample aliquots are measured in 25 mL gas tight syringes. Separate syringes are used for each sample.

10.7.3.3.2 For Method 8260C/D, 5mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4) Sample aliquots are measured in 10 mL gas tight syringes. Separate syringes are used for each sample.

10.7.3.3.3 For Method 624/624.1, 5 or 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.). Sample aliquots are measured in 5 or 25 mL gas-tight syringes. Separate syringes are used for each sample.

10.7.4 Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe.

10.7.4.1 For dilutions of aqueous samples which require less than 1 mL of sample the sample volume is added to 20 mL of reagent water in a VOA vial or in a gas-tight syringe.

10.7.4.2 For dilutions of aqueous samples which require more than 1 mL of sample, the volume of reagent water is adjusted so that the total volume of sample and reagent water is 20 mL. The dilution is made in the VOA vial by adding the appropriate amount of reagent water to the vial. The sample aliquot is then added to the closed vial by injecting below the surface of the water.

10.7.4.3 If the dilution required would use less than 5 µL of sample, then serial dilutions must be made in volumetric flasks.

10.7.4.4 Check the pH of the sample by dipping the pH paper into the sample that is remaining in the VOA vial after the aliquot for analysis has been taken (the remaining sample is not used in analysis.) Document the pH value on the run log. If the pH is not as expected, based on the sample type and preservation, document in an NCM in the LIMS.

10.7.4.5 Sample remaining in the vial after sampling is no longer valid for further

analysis. A fresh VOA vial must be used for further sample analysis.

10.7.4.6 For TCLP samples, use 2.0 mL of TCLP leachate and spike it with 2.5 µL of the 40 µg/mL TCLP spiking solution. Bring to a volume of 20 mL with reagent water.

10.7.4.7 Surrogates and internal standards are added to each sample at the instrument at the time of purging.

10.7.4.8 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe needle through the septum into the water. Surrogates and internal standards are added to these samples by the instrument.

10.7.4.9 Purge the sample for eleven minutes (the trap should be below 50 ºC).

10.7.4.10 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 2-5 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

10.7.4.11 Desorb time, bake time, and temperature are optimized for the type of trap in use.

NOTE: The same conditions must be used for samples and standards.

10.7.4.12 If foaming of the sample occurs, reanalyze the sample with the addition of 1 µL of an antifoaming agent such as Antifoam B (J. T. Baker). A method blank spiked with 1 µL of the Antifoam B must also be analyzed with the sample. Make notation of “AF” on runlog for each sample to which antifoam is added.

10.7.5 Methanol Extracts of Soils

10.7.5.1 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations (typically 20 mL).

10.7.5.2 Add no more than 200 µL of methanolic extract (from Section 10.3.1 or 10.5.1) to the syringe for each sample and QC sample.

10.7.5.3 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe through the septum of the vial.

10.7.5.4 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe.

10.7.5.5 Only internal standards are added at the instrument for methanol extracts.

10.7.5.6 Load the sample onto the purge and trap device and analyze as for aqueous samples. (See Section 10.7.3.)

10.7.6 Liquid Wastes that are Soluble in Methanol and Insoluble in Water

10.7.6.1 Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

10.7.6.2 Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly.

10.7.6.3 For an MS/MSD pair, add 6 mL of methanol to 2 mL of the sample in a tared vial. Add 1 mL of surrogate solution and 1 mL of matrix spike solution.

10.7.6.4 Prepare an LCS by adding 1 mL of surrogate solution and 1 mL of matrix spike solution to 8 mL of methanol.

10.7.6.5 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations.

10.7.6.6 Add no more than 200 µL of methanolic extract (Section 10.7.6.2) to the syringe. Add internal standard (if used).

10.7.6.7 Load the sample onto the purge and trap device and analyze as for aqueous samples using 5 mL reagent water.

10.7.6.8 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe. (See Section 10.7.4.)

10.7.7 Low-Level Soil Sample Analysis following SW846 Method 5035A

10.7.7.1 This technique is to be used when samples are collected utilizing SW-846 Method 5035A. Pre-weighed vials are used to collect approximately a 5 gram aliquot of soil (see section 8.2).

10.7.7.2 The sample vial should not have any labels applied to it until after the sample weight has been taken. Any labels applied prior to weighing will affect the initial weight calculation of the sample, because it was not accounted for in the tare weight.

10.7.7.3 Purge-and-trap units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.7.4 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial.

10.7.7.5 The autosampler will heat and stir each sample as it is purged.

10.7.7.6 If any target analytes exceed the calibration range, analysis of the methanol preserved sample must be performed.

10.7.8 Low-Level Solids Analysis When Field Samples are Provided in a Jar

NOTE: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

10.7.8.1 This method is based on purging a heated sediment/soil sample mixed with water and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40 ºC.

10.7.8.2 Do not discard any supernatant liquids. Mix the contents of the container with a disposable tongue depressor as spatula.

10.7.8.3 Weigh out 5 g (or other appropriate aliquot) of sample into a clean VOA vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium-level method described in Section 10.7.5 with preparation described in Section 10.5.1.

10.7.8.4 Rinse a 5 mL gas-tight syringe with organic-free water, and fill. Compress to 5 mL. Inject the spiked water into the VOA vial that contains the soil sample and add a stirring bar.

10.7.8.5 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

10.7.8.6 Prepare a Method Blank and LCS using 5 g of Ottawa sand and 5 mL of water. Add a stirring bar to each. Prepare the MS/MSD (based on the sample requested by the client. The LCS spiking solution is added via a syringe inserted through the septum of the vial to the LCS and MS/MSD samples.

10.7.8.7 Low level soil samples may be analyzed with a 1 g aliquot in place of the 5 g aliquot, mixed with water. If higher dilutions are required, the methanol extract (medium level) will be analyzed.

10.7.8.8 Surrogate and internal standards are added automatically to all samples at the instrument.

10.7.8.9 The autosampler will heat and stir each sample as it is purged.

10.7.8.10 Soil samples that have low internal standard recovery when analyzed (< 50%) should be reanalyzed once to confirm matrix effect.

10.7.9 Initial Review and Corrective Actions

10.7.9.1 If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minute from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required. DoD QSM 5.0 requires that the retention time for any internal standard be within 10 sec of the midpoint of the ICAL. If accepted by the client, this window can be expanded to 30 sec of the midpoint of the ICAL.

10.7.9.2 If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

Sample internal standard areas are compared to the mid-point of the supplemental initial calibration internal standard areas. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

10.7.9.3 The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

10.7.10 Dilutions

10.7.10.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample or extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.7.10.2 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample

should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

10.7.10.3 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.7.11 Instrument Set-up

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.7.12 Recommended Instrument Conditions

10.7.12.1 General

Parameter	Setting
Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 2 second/scan
Injector Temperature:	200 – 250 ˚C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 ˚C
Purge Flow:	40 mL/minute
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

10.7.12.2 Gas Chromatograph Suggested Temperature Program

The following temperature programs vary with the column type used.

BFB Analysis

Initial Temperature:	150 ˚C
Initial Hold Time:	0.00 minutes
1st Temperature Program:	50.00 ˚C/minute

Final Temperature:	220 ˚C
Final Time:	4.00 minutes
2nd Temperature Program:	OFF
Post Temperature:	0 ˚C
Post Time:	0.00 minutes
Run Time:	5.40 minutes

Sample Analysis

Initial Temperature:	40 ˚C
Initial Hold Time:	4 minutes
1st Temperature Program:	8 ˚C/minute
Final Temperature:	184 ˚C
2nd Temperature Program:	40 ˚C/minute
Final Temperature:	240 ˚C
Final Hold Time:	2.6 minutes

10.7.12.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.7.13 Initial Calibration

10.7.13.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and in the public folder *Arizona Calibration Training.*

10.7.13.2 A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Certain analytes are prepared at higher concentrations due to poor purge performance. The following calibration curves are maintained. Calibration levels for each analyte are given in the stated tables. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements.

Initial Calibration by Matrix and Method

Method	Matrix	Purge Volume	Calibration Levels
624/624.1/8260C/D	Water	5 mL	Table 5A
8260B	Water	20 mL	Tables 5 and 5A
8260B/C/D	Soil (low level)	5 mL	Tables 4 and 4A

Method	Matrix	Purge Volume	Calibration Levels
8260B/C/D	Soil (Methanol Extract)	20 mL reagent water + 100 µL Methanol	Tables 6 and 6A
Alaska	Soil	See Appendix B	See Appendix B

10.7.13.3 Calibration levels below the reporting limit may be removed provided that there is a minimum of five calibration points for linear regression and six calibration points for second order calibration. The lowest standard used in the calibration must be at or below the TestAmerica reporting limit.

10.7.13.4 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.7.13.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.7.13.6 Internal standard calibration is used. The internal standards are listed in Tables 8 and 8A. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Equation 1, Section 11.5.1.1, for calculation of response factor.

10.7.13.7 Evaluation of retention times

The retention time of each target analyte in each calibration standard should agree within 0.5 min.

10.7.13.8 The % RSD of each of the calibration check compounds (CCC) must be less than or equal to 30%. Refer to Table 12. See Table A-2 for Method 624/624.1 criteria.

10.7.13.9 The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.7.13.10 If the software in use is capable of routinely reporting curve coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better

accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. The correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

NOTE: Additional criteria are stated in the North Carolina QAS. South Carolina does not allow the use of second order curves.

10.7.13.11 If the software in use is capable of routinely reporting curve coefficients for data, and if the average of all the %RSDs in the calibration is > 15%, then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be ≥ 0.990. DoD QSM 5.0 requires that the correlation coefficient for linear fits be ≥ 0.995 (i.e., $r^2 \geq 0.99$).

NOTE: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

10.7.13.12 Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. All compounds in the ICV must be <35% drift when compared to the initial calibration, except poor performers (see Table 15) which must be <55% drift. The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against spike recovery data and method performance data, where available, to validate each compound as a "poor performer." The ICV is generally run at the same concentration as the level 5 standard. See Table A-2 for method 624/624.1 criteria.

DoD QSM 5.0 requires the ICV analytes must be within ± 20% of the true value. Poor performers listed in the TestAmerica Technical Specifications may be ± 30% if approved by the client.

South Carolina requires that all analytes of interest in the ICV must recover at ≤ 30% (≤ 40%) for poor purgers prior to sample analysis.

10.7.13.13 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 10.7.14.

10.7.13.14 A separate five point calibration must be prepared for analysis of low-level soils. Low-level soils analysis requires the use of a closed vial autosampler. Each standard is prepared by spiking the methanol standard solution through the septum of a VOA vial containing 5 mL of water. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Methanol soil extracts should be analyzed using the methanol

calibration curve.

10.7.13.15 Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. The primary ion for the single standard must generate a peak clearly visible over background noise (greater than three standard deviations at a minimum) and be free of spectral interferences. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary. A footnote or narrative comment should describe the basis of the reported result.

10.7.14 Continuing Calibration

10.7.14.1 The initial calibration must be verified every twelve hours.

10.7.14.2 Continuing calibration begins with analysis of BFB as described in Section 10.7.12.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 5 calibration standard is used as the continuing calibration standard. See Table A-2 for method 624/624.1 criteria.

10.7.14.3 The RF data from the standards are compared with the initial five-point calibration to determine the percent drift of the CCC compounds.

10.7.14.4 The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift for most non-CCC analytes must be ≤ 35 %, and for poor performers ≤ 50 % (See Table 15), with allowance for up to six target analytes to have a % drift greater than the applicable limit. For agencies that require specific control limits for non-CCC compounds (i.e., State of Arizona) see Table 14. See Table A-2 for method 624/624.1 criteria.

For South Carolina, the percent drift for non-CCC analytes must be ≤ 30 % (≤ 40%) for poor purgers.,

Note: Additional criteria are stated in the North Carolina QAS.

10.7.14.4.1 If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 12) must be agreed to with the client.

10.7.14.4.2 Cyclohexanone is unstable in the calibration solution forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists.

10.7.14.5 The retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds when compared to the most recent five-point calibration. The internal standard areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent five-point calibration.

10.7.14.6 If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.7.14.3 and 10.7.14.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration.

10.7.14.7 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

10.7.14.8 Sodium Bisulfate must be added to the CCV when analyzing samples preserved with it.

10.7.14.9 Example analytical sequence for GC/MS volatile analysis:

1. BFB
2. CCV Main
3. CCV Supp
4. LCS
5. LCSD (if required)
6. MB

20 client samples

The above example sequence is the only way that client samples can be analyzed. If any part of lines 1 through 3 does not meet method criteria for running client samples it must be re-analyzed before continuing on to the next step. For example, the CCV(s) can not be run without first passing the BFB tune and LCS/LCSD/MB must follow passing CCV(s).

DoD QSM 5.0 requires a closing CCV be run. The injection time must be within the 12-hour tune time. Acceptance criteria are $\pm$ 50%. See Section 10.10 for alternate acceptance criteria if approved for the project.

10.8 Maintenance

See section 20 of the Denver QAM

10.9 Troubleshooting

10.9.8 For RT shifts check purge flow for possible leaks or blockage depending on which way the RT is shifted.

10.9.9 If no peaks appear in chromatogram

10.9.9.1 Check water and standard reservoirs and helium switch is to on position.

10.9.9.2 Check that purge needle is not broken/leaking.

10.9.9.3 Check tune to make sure filaments are still functional.

10.9.9.4 Check GC column for breaks

10.9.9.5 If maintenance was recently performed, re-check that area to make sure all connections are tight.

10.9.10 Samples with high concentrations of hydrocarbons may cause internal standards to fail high in samples analyzed afterwards. Depending on severity either running Methanol blanks or replacing contaminated parts may be needed.

10.9.11 Samples with a basic pH (>10) will cause the surrogate Dibromofluoromethane to fail low.

10.9.12 Samples that contain organic matter may cause internal standard failures.

10.9.13 Temperature variations in the laboratory will cause shifts in the recoveries of water soluble compounds (i.e., Ketones and Alcohols).

10.9.14 If instrument has difficulty passing BFB tune, front end maintenance can solve the problem.

10.9.15 Samples with extreme foaminess can cause issues ranging from failing QC to contamination of instrumentation which may require replacement of contaminated parts.

10.10 Method specific requirements for QSM 5.0

Method Comment Codes and definitions for possible method variances. Method Comment indicates that client has accepted the variance. Requirement if variance NOT accepted is provided in the table below. DoD QSM 5.0 requirements are listed throughout this SOP in the appropriate sections.

Code	If Variance accepted	If Variance not accepted
1SME	Laboratory must be informed of critical compounds of concern to ensure ME not applied to these compounds.	SME is allowed (see table in section 9.5 for number of analytes) except for critical compounds of concern.
2CLC	Common lab contaminants allowed up to RL – Variance identifies those compounds identified as common lab	Common lab contaminants allowed up to RL. See section 9.4

Code	If Variance accepted	If Variance not accepted
	contaminants	
3HR	Allowed to report biased high failures in CCV/LCS/surrogate when samples ND. Report with NCM and flags.	All QC failures not matrix related must be re-analyzed
4PP	30%D CCV/ICV criteria allowed for poor performers as long as not listed as constituent of concern (COC) by client	Does not recognize poor performers. All targets must pass 20% criteria.
6CCVOrg	If closing CCV fails due to the matrix of samples run prior to it and failure is confirmed once, consult client before reporting	All samples must be bracketed by passing CCV
7MS	Poor performers excluded from 50%D in closing CCV. Re-analyze closing CCV for non-COC's and report others with NCM and flags.	Closing CCV must pass 50%D criteria for all target compounds.
8ISRT	Internal standard window +/- 30 seconds from ICIS	Internal standard window +/- 10 seconds from ICIS

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-P-003 "Calibration Curves and Selection of Calibration Points" and in the public folder, *Arizona Calibration Training*.

11.2 Qualitative Identification

11.2.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

11.2.1.1 The sample component retention time must compare to within ± 0.5 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

11.2.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

11.2.1.3 The relative intensities of ions should agree to within ±30% between the

standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

11.2.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

11.2.3 All data are subject to two levels of technical review, as described in SOP DV-QA-0020.

11.3 Tentatively Identified Compounds (TICs)

11.3.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. The following guidelines apply:

11.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.3.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

11.3.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.3.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.3.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

11.3.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst *assign a tentative identification.*

11.3.2 Chrom setup

Set up for identification of TICs in Chrom is done by using the following settings in the Chrom Method Editor.

Item	Setting
Enable TIC quantitation	Checkmark on

Quant from	Nearest ISTD
Min % for ID	10%
Start and end time	Set both to 0.000
Match Threshold	85%
Disable calibrated TICs	Checkmark on
Enable TIC search	Checkmark on
Path to Library	Must be set to correct path
Subtraction method	Generally best set to no subtraction
Chrom match RT window	Set to 0.1 min

11.4 Data Review

Common Oversights and Possible Fixes

Finding	Fix
Level 4 QC summary report error	8260B/C/D: Set 1,4-Dioxane-d_8 set to "not needed" status in samples and ICAL 624/624.1: Set 1,4-Dioxane-d_8 and TBA-d_9 to "not needed" status in samples and ICAL
Missing MS/MSD % recoveries and/or RPD	MS/MSD linked to incorrect parent (all three must be run at same concentration); relink. Missing reagent value – verify and correct on reagent tab. Parent and MS/MSD not processed with "all compounds" list; verify worklist is correct; re-upload if error found in worklist.
Dilution factor/initial volume incorrect	Double check that what is being reported reflects what was performed; correct as needed
Proper sample preservative	8260B/C/D: Create NCM when 2–CLEVE is a target and sample has pH<2. 624/624.1: Create NCM when 2-CLEVE and/or Acrolein is a target and sample pH<2. All: Create NCM when pH does not match preservative type.
QC not in deliverables	All associated QC must be at 2nd level review status to appear in deliverables. This includes any ICIS, ICV, CCVC, LCS/LCSD, MB, LB, and MS/MSD. Use Job Data Review or QC checker to verify and correct as needed.
Incorrect flagging	Re-calculate sample, do not manually remove flag without permission.
False hit for 1,2,3-Trichloropropane	When anti-foam is used take extra care in evaluating any hit for 1,2,3-Trichloropropane. Ensure correct identification.

Finding	Fix
TIC with large ion 44 early in run	This is usually Carbon Dioxide labeled as an unknown. Verify identification. If reporting TICs it should be marked as undetected in order to not report.

11.4.4 Linear Fit

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The weighting used is the reciprocal of the concentration or the reciprocal of the square of the concentration. The regression produces the slope and intercept terms for a linear equation in the following:

$$R_x = m_1(C_s) + b \qquad \textit{Equation 6}$$

Where:

C_s	=	Analyte concentration in calibration standard, µg/L
R_x	=	Response for analyte
b	=	y - Intercept
m_1	=	Slope

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equations, where C_{ex} is the concentration of the target analyte in the unknown sample:

$$C_{ex} = \frac{[R_x - b]}{m_1} \qquad \textit{Equation 7}$$

Where:

C_{ex}	=	Extract analyte concentration, µg/L
R_x	=	Response for analyte
b	=	y - Intercept
m_1	=	Slope

11.4.5 Evaluation of the Linear Least-Squares Regression Calibration Function:

11.4.5.1 With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations.

11.4.5.2 Note that the August 7, 1998 EPA memorandum “Clarification Regarding Use of SW-846 Methods”, Attachment 2, Page 9, includes

the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria:

11.4.5.3 To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit (RL), and preferably less than the MDL.

11.4.5.4 Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

11.4.5.5 The linear regression must have a correlation coefficient (r) ≥ 0.99 (r^2 ≥ 0.98). Some programs (e.g., DoD) require a correlation coefficient (r) ≥ 0.995 (r^2 ≥ 0.99).

11.4.6 Quadratic Fit

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$R_x = m_2(C_s)^2 + m_1(C_s) + b \qquad \textit{Equation 8}$$

Where:

C_s = Analyte concentration in calibration standard, µg/L
R_x = Response for analyte
m_2 = Curvature
m_1 = Slope
b = y - Intercept

To calculate the concentration in an unknown sample extract, the roots of the quadratic equation are solved for:

$$C_{ex} = \frac{-m_1 \pm \sqrt{(m_1)^2 - 4(m_2)(b - R_x)}}{2m_2} \qquad \textit{Equation 9}$$

Where:

C_{ex} = Extract analyte concentration, µg/L
R_x = Response for analyte
m_2 = Curvature
m_1 = Slope
b = y - Intercept

11.4.7 Evaluation of Second-Order Regression Calibration:

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria:

11.4.7.1 Second-order regressions should be the last option, and note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

11.4.7.2 The coefficient of determination (COD, r^2) must be ≥ 0.99.

11.4.7.3 The absolute value of the intercept is not large relative to the lowest concentrations being reported.

11.4.7.4 The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).

11.4.7.5 The distribution of concentrations is adequate to characterize the curvature.

11.5 Calculations.

11.5.1 Linear Calibration Using Average Response Factors

11.5.1.1 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x} \qquad \textit{Equation 1}$$

Where:

A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

11.5.1.2 For each target analyte, calculate the average response factor as follows:

$$\text{AverageResponseFactor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad \textit{Equation 2}$$

Where: n = Number of calibration levels
RF_i = Response factor for the i^{th} level

11.5.1.3 The relative standard deviation (RSD) is calculated as follows:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \textit{Equation 3}$$

Where:

$\overline{RF}$ = Mean of RFs from the initial calibration for a compound
SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{N}\left(RF_i - \overline{RF}\right)^2}{N-1}}$$ *Equation 4*

RF_i = RF for each of the calibration levels
n = Number of RF values

11.5.1.4 Average Calibration Fit Evaluation

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average response factor.

Acceptance Criteria: The RSD of the average response factor must be ≤15%. Also examine the residuals, especially for the high points versus the fitted function. If the residual values are large, a linear regression should be considered.

Corrective Action: If the RSD is > 15%, average response factor cannot be used and least-squares linear regression should be attempted.

11.5.1.5 Calculating the Concentration in the Extract

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$ *Equation 5*

Where:

C_{ex} = Concentration in the extract, µg/mL
R_x = Response for the analyte
R_{is} = Response for the internal standard
C_{is} = Concentration of the internal standard
$\overline{RF}$ = Average response factor

11.6 Acceptance Criteria Independent of Calibration Model – Method 8000D

11.6.1 Method 8000D in SW-846 recommends the use of either the Percent Error or Relative Standard Error to evaluate the calibration model as described below.

NOTE: For South Carolina work, one of these methods of evaluation must be used.

11.6.2 Percent Error

11.6.2.1 The percent error assesses the refitting of the calibration data back to the model. The percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards except for the lowest data point which should be ≤ 50%.

11.6.2.2 Calculation of the Percent Error

$$\%Error = \frac{x_i - x_i'}{x_i} \times 100$$ *Equation 10*

Where:

x'_I = Measured amount of analyte at calibration level *i*, in mass or concentration units

x'_I = True amount of analyte at calibration level i, in mass or concentration units

11.6.3 Relative Standard Error (RSE)

11.6.3.1 Relative standard error provides a measure of the fit of the calibration that is independent of the calibration model. RSE is calculated using the following equation:

$$\text{RSE} = 100 \times \sqrt{\frac{\sum_{i=1}^{N}\left(\frac{x'_i - x_i}{x_i}\right)^2}{(n-p)}}$$ *Equation 11*

Where:

x'_I = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_I = True amount of analyte at calibration level *i*, in mass or concentration units

p = Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)

n = Number of calibration terms

11.6.3.2 The RSE acceptance limit criterion is the same as the RSD limit.

11.7 Calculations for reporting original samples

11.7.1 Calculating concentration in aqueous samples:

$$\text{Concentration, } \mu\text{g/L} = C_{ex} D$$ *Equation 12*

Where:

C_{ex} = Analyte concentration, µg/L
D = Dilution factor

11.7.2 Calculating concentration in low level soil samples:

$$\text{Concentration, } \mu\text{g/Kg} = C_{ex} \times \frac{Vp}{Ws} \times \frac{1}{(100 - M)} \times D$$ *Equation 13*

Where:

C_{ex}	=	Analyte concentration, µg/L
Vp	=	Purge volume (5 mL)
Ws	=	sample weight (in grams)
M	=	Percent Moisture
D	=	Dilution factor

Note: Units conversions for purge volume and sample weight yield conversion factor of 1 and are not shown in the equation for simplicity.

11.7.3 Calculating concentration in medium level soil samples

$$Concentration, \mu g/Kg = C_{ex} \times \frac{V_p}{V_a} \times \frac{V_e}{W_s} \times \frac{1}{(100 - M)} \times DF$$ *Equation 14*

C_{ex}	=	Uncorrected concentration of sample from instrument quantitation report (µg/L)
V_p	=	Final purge volume (mL)
V_a	=	Volume of methanol extract added to the purge volume (mL)
V_e	=	Volume of methanol used for extraction (mL)
W_s	=	Weight of sample extracted (g)
DF	=	Dilution Factor

Note: Units conversions for purge volume and sample weight yield conversion factor of 1 and are not shown in the equation for simplicity.

11.7.4 LCS and CCV Percent Recovery

$$\text{ControlSpike Recovery} = \frac{S_{SR}}{S_A} \times 100\%$$ *Equation 15*

Where (in µg/L):

S_{SR}	=	Calculated analyte concentration of spiked sample
S_A	=	Concentration of standard added

11.7.5 MS/MSD Recovery

$$\%\,\text{Recovery} = \frac{SSR - SR}{SA} \times 100\%$$ *Equation 16*

Where:

SSR	=	*Spike sample result.*
SR	=	*Sample result.*
SA	=	*Spike added.*

11.7.6 RPD calculation for the MS/MSD:

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100\%$$ *Equation 17*

Where:

RPD = *Relative percent difference.*
MSR = *Matrix spike result.*
MSDR = *Matrix spike duplicate result.*

11.7.7 See SOP CA-Q-S-005 for more detailed calibration equations. All data are subject to two levels of technical review, documented on a checklist, as described in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements (e.g., DoD) indicate a greater frequency.

12.1.2 MDLV and LOQV

12.1.2.1 MDLVs and LOQVs must be performed on blank matrix as defined for the method blank (Section 9.3), spiked with the appropriate analytes at the programmatically required spike amounts.

12.1.2.2 MDLVs are analyzed for compounds on the DOD certificate and those required for Texas TRPP on each instrument each quarter. For DOD the spike requirement is 2-4X the MDL and for Texas it is 1-4X the MDL. There are no recovery requirements for MDLV. The MDLs for all other compounds are verified at least annually

12.1.2.3 LOQVs (aka LLOQV) are analyzed for compounds on the DOD certificate on each instrument each quarter. The spiking requirement is 1-2X the RL and must pass percent recovery limits. For all compounds analyzed by Method 8260, LOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the LCS.

12.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.1.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.1.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methanol Waste - Vial Waste and Flammable – Waste Streams A and C

14.2.2 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.3 Acidified Water – Waste Stream W

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 8260B/C/D, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996.

15.1.2 Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December, 1996.

15.1.3 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December, 1996.

15.1.4 Method 5035A (R1-MIR), Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

15.1.5 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.6 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

15.1.7 Method 8000D, Determinative Chromatographic Separations, Revision 4, July 2014.

15.2 40 CFR Part 136, Appendix A (Method 624/624.1, Method 603) and update on August 28, 2018.

15.3 Method AK101 For the Determination of Gasoline Range Organics, Alaska DEC, Version 04/08/02.

16.0 Method Modifications:

Item	Method	Modification
1	SW-846 8260B/8260C	Ion 119 is used as the quantitation ion for chlorobenzene-d_5.
2	SW-846 8260B/8260C	The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
3	SW-846 8260B/8260C	This SOP has been written to allow for a 20 mL purge volume for waters. An additional 5 mL of DI water is added to all samples, QC and calibration standards. The final purge volume is 25 mL.
4	SW-846 8260B/8260C	Method 8260B recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.
5	EPA 624/624.1	Method 624/624.1 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.
6	EPA	The tune period for this method is defined as 12 hours, which is the maximum elapsed time before the tune check is performed. Calibration

Item	Method	Modification
	624/624.1	verifications are done at the same 12 hour frequency.
7	EPA 624/624.1	The initial calibration curve for this method requires at least five points, as shown in Table 5A.
8	EPA 624/624.1	Sample concentrations are calculated using the average RRF from the initial calibration curve.
9	EPA 624/624.1	Each target analyte is assigned to the closest eluting internal standard.
10	EPA 624/624.1	Initial demonstration of Proficiency • The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table A-2
11	EPA 624/624.1	Initial calibration curve requirements: • Target compounds must have RSD ≤ 35%. • If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. Then the r^2 was be greater than 0.920.
12	EPA 624/624.1	Continuing calibration verification requirements: • The continuing calibration standard is from a different source than the initial calibration standard. The daily CCAL concentration is 20 µg/L. The acceptance criteria are listed in Table A-2. Matrix Spike and LCS Requirements • The matrix spike and LCS are spiked at 20 µg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table A-2.
13	EPA 624/624.1	Consistent with the other volatile methods, corrections for recovery are not allowed.
14	EPA 624/624.1	Qualitative Identification – The source method states that the relative intensities of ions should agree to within ±20% between the standard and sample spectra. This SOP uses ±30%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)
15	EPA 624/624.1	Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the OI #10 trap which consists of Tenax/Silica Gel/ Carbon Molecular Sieve or the Supelco Vocarb 3000 which consists of Carbopack B, Carbonxen1000 and 1001.
16	EPA 624/624.1	Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
17	EPA	The source method provides a suggested list of compounds for internal

Item	Method	Modification
	624/624.1	and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including chlorobenzene-d_5 and 1,4-dichlorobenzene-d_4, which are not listed in Table 3 of the source method. Toluene-d_8 is used as a surrogate compound, which is also not listed in the source method.
18	EPA 624/624.1	The lab is preparing internal standards at 10 µg/L and applying the same criteria designed for 30 µg/L in the Method. The lower concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the method. It could only be more challenging for the lab to meet the acceptance criteria at 10 µg/L; it provides a higher level of data quality.
19	EPA 624/624.1	Method 624/624.1 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, the laboratory scans from 35 to 300 and includes all ions needed for analysis.
20	EPA 624/624.1	Method 624/624.1 describes dilutions "if response of any m/z" exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.10) is equivalent.
21	EPA 624/624.1	Method 624/624.1 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta- and para-xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. meta- and para-xylenes are reported as an isomeric pair. Any development work to add compounds would take this into account.
22	624/624.1	The source method recommends Method 603 as the preferred method for Acrolein and Acrylonitrile. Method 624/624.1 is recommended as a screening method (see section 1.2 of Methods 603 and 624/624.1). Calibration and quality control samples indicate that the conditions described in this SOP are suitable for the analysis of Acrolein and Acrylonitrile. EPA's Method Update Rule (MUR), May 18, 2012, allows the addition of acrolein and acrylonitrile to Method 624/624.1, using the preservation, holding time and QC acceptance criteria from Method 603. As states implement the MUR Method 624/624.1 becomes a determinative method for these two analytes. Until such time, Method 624/624.1 remains a screening method for regulatory compliance.
23	SW846 5035	The source method recommends adding approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~ 1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. TestAmerica Denver does not recommend the use of sodium bisulfate to preserve soil samples, but encourages clients to collect samples using other available methods. The use of this preservative has been shown to cause difficulties recovering more reactive analytes on the purge and trap system (e.g. 2-Chloroethyl vinyl ether, acrylamide).

17.0 Attachments

18.0 Changes from Previous Revision

- Revision 24, dated 15 February 2019
  - SOP references updated to included 8260D.
- Revision 23, dated 08 January 2019
  - Updated 624 to 624/624.1
- Revision 22, dated 05 November 2018
  - Updated section 8.1.1.1 concerning headspace/bubbles.
- Revision 21, dated 30 September 2018
  - Added section 8.1.1.1 regarding headspace/bubble check requirements by Sample Receiving and Analysts.

- Revision 20, dated 31 August 2018
  - Updated for 8260C.
- Revision 19, dated 31 March 2018
  - Updated for 624/624.1/624/624.1.1.1 MUR update.
- Revision 18, dated 15 August 2017
  - Changed concentration of Ethanol in calibration tables to reflect new standard concentration.
  - Fixed spelling of Tetrachloroethene in IS/Surr association table.
  - Added instructions on handling status change of soil prep batches.
- Revision 17, dated 20 October 2016
  - Added definition for LLOQ as new Section 3.7 per 8000D
  - Removed comment about Colorado allowing 14 day holding time for unpreserved samples in existing section 8.1.3
  - Revised Holding time table for water to reflect Update V and clarify HT for method 624/624.1 based on 40 CFR Part 136; revised footnotes to the table.
  - Revised section 10.3 regarding how soil sample weights are documented to reflect current practice
  - Added note in Section 10.4.1 to NOT affix labels on terracore vials before sample is analyzed or prepped.
  - Added information on Chrom setup for TICs in new section 11.3.2
  - Added new Section 11.5 for Accetpance Criteria Independent of Calibration Model for 8000D (Percent Error and Relative Standard Error)
  - Added equations for calculation of soil concentrations in new Sections 11.6.5 and 11.6.6
  - Added MDLV and LOQV information in Section 12.1.2
  - Added reference to Method 8000D in Section 15

- Revision 16, dated 21 April 2016
  - Added requirements for glassware cleaning in appropriate sections throughout.
  - Added requirements for LCS and MS spikes to Sections 7.12, 7.13, 9.6 and 9.7, clarifying requirements for DoD, South Carolina and NELAP (TNI).
  - Added requirement to Section 9.6 to add antifoam to LCS when added to samples.
  - Clarified Section 10.7.4.4.
  - Removed requirement to document use of antifoam in NCM in Section 10.7.4.12. Record made on run log.
  - Revised Section 10.7.13.12 to include South Carolina criteria for ICV.
  - Revised Section 10.7.14.4 to include South Carolina criteria for CCV (non-CCC compounds).
  - Added new section 11.4 to list common data review findings and appropriate fixes.
  - Revised Section 11.5.1.4 (old section 11.4.1.4) to correctly refer to average response factor rather than calibration factor as this is an internal standard calibration technique.
  - Removed Table 10 (LCS short list) and made reference to spikes actually used throughout. Renumbered remaining tables.
  - Removed references to AFCEE throughout; laboratory follows requirements of DoD QSM for all DoD work.
  - Corrected terminology for evaluation of calibration using %RSD from calibration factor (external standard) to response factor (internal standard)
  - Reviewed SOP to ensure that all DoD QSM 5.0 requirements were appropriately stated in addition to Section 10.10. Added specifications as needed throughout document for clarity.

- Deleted Item 5 from previous Method Modifications table. No longer applicable. Renumbered remaining items.

- Revision 15, dated 30 September 2015
  - Revised section 6.3.7 for use of disposable wooden spatulas
  - Revised Sections 9.3 and 9.5 to describe matrix used for QC samples

- Revision 14, dated 08 September 2015
  - Revised description of method blank with respect to blank matrix used for each type of analysis (Section 9.3)
  - Added description of LCS for medium level soil.
  - Revised corrective actions for matrix spike/matrix spike duplicate in Section 9.6.
  - Added notes to Section 10.7.2.1 and Appendix A requiring 12-hour tune for Method 624/624.1 for South Carolina samples.
  - Revised Section 10.10 to clarify QSM requirements and lab requested variances.
  - Updated SOP reference in Section 11.1 to current corporate SOP.
  - Added statement to Section 12.1 that MDLV be performed using matrix for method blank.
  - Added note to Method Modification #7 limiting Method 624/624.1 tune time to 12 hours for South Carolina samples.

- Revision 13, dated 30 April 2015
  - Revised discussion and preservation/HT table for soil samples in Section 8
  - Corrected retention time window in 11.2.1.1 for qualitative identification
  - Added clarification to ICAL tables in Attachments regarding use of calibration points below standard reporting limits
  - Corrected RL in water for 2-chloroethylvinyl ether
  - Corrected Table 13 entry for methyl-*tert*-butyl ether characteristic ions
  - Corrected footnotes on Table A-2, Method 624/624.1 QC Acceptance Criteria to reflect current practice

- Revision 12, dated 31 December 2014
  - Added Section 10.7.14.9 to describe run sequence.

- Revision 11, dated 31 January 2014
  - Changed calibration tables to reflect changes due to standard standards
  - Added internal standard reference table (Table 17)
  - Added TBA-d_9 and 1,4-Dioxane-d_8 as internal standards
  - Removed SIM (see SOP DV-MS-0016)
  - Removed section 9.8

- Revision 10, dated 19 August 2013
  - Formatting changes

- Revision 9, dated 04 January 2013
  - Added section 9.8 to address the 2012 MUR QC requirements

- Revision 8, dated 28 September 2012
  - Added to compounds to the reporting limit, characteristic ion and calibration tables to match TALS.

- Revision 7, dated 27 July 2012
  - Added sodium bisulfate to Section 7.
  - Revised Section 8 to include Terra Core samplers and moved instructions on sample preparation and handling in the lab to Section 10. Reorganized sampling and preservation information into tables. Updated information including footnote on Holding Time and preservation table for water regarding Method Update Rule that approves use of Method 624/624.1 for analysis of acrolein and acrylonitrile.
  - Removed flowcharts from Section 8.
  - Revised Section 9.1
  - Revised Section 10.
  - Updated reference section to include Method 603, Method 5035A, and Method 8000B and 8000C.
  - Revised Method Modifications #23
  - Updated tables to reflect current practice.
  - Added Appendix A for the analysis of soils using the AK methanol extraction procedure.
  - Formatting and editorial changes throughout

- Revision 6.4, dated 28 December 2011
  - Changed the column ID and film thickness in section 6.1.8.1

- Revision 6.3, dated 26 October 2011
  - Added Section 4.6 regarding interferences with toluene-d_8 surrogate when potassium permanganate may have been added to sample
  - Updated path to QAS folders in the public folders, section 9.7
  - Added J. T. Baker Antifoam B and reagent sand, sections 7.3, 7.4
  - Added description of procedure for use of antifoaming agent B, section 10.1.3.8
  - Formatting

- Revision 6.2, dated 25 August, 2011
  - Added requirements to section 9.4 for the use of Ottawa sand in soil LCS's.

- Revision 6.1, dated 31 January, 2011
  - Added Attachment 1, Gas Standards Tracking Log
  - Added section 11.1 referencing corporate SOP CA-Q-S-005 "Calibration Curves"

Earlier revision histories have been archived and are available upon request.

Table 1. TestAmerica Primary List Reporting Limits for 8260B/C/D

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
Dichlorodifluoromethane	75-71-8	2	10	500
Chloromethane	74-87-3	2	10	500
Bromomethane	74-83-9	2	10	500
Vinyl chloride	75-01-4	1	5	500
Chloroethane	75-00-3	2	10	500
Trichlorofluoromethane	75-69-4	2	10	500
Acrolein	107-02-8	20	50	5,000
Acetone	67-64-1	10	20	1,000
Trichlorotrifluoroethane	76-13-1	3	20	1,000
Ethanol	64-17-5	300	600	10,000
Iodomethane	74-88-4	1	5	250
Carbon disulfide	75-15-0	2	5	250
Methylene chloride	75-09-2	2	5	250
tert-Butyl alcohol	75-65-0	50	200	10,000
1,1-Dichloroethene	75-35-4	1	5	250
1,1-Dichloroethane	75-34-3	1	5	250
trans-1,2-Dichloroethene	156-60-5	1	2.5	125
Acrylonitrile	107-13-1	20	50	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	5	20	250
Hexane	110-54-3	2	5	250
cis-1,2-Dichloroethene	156-59-2	1	2.5	125
1,2-Dichloroethene (Total)	540-59-0	1	5	250
Tetrahydrofuran	109-99-9	7	20	1,000
Chloroform	67-66-3	1	10	250
1,2-Dichloroethane	107-06-2	1	5	250
Dibromomethane	74-95-3	1	5	250
2-Butanone	78-93-3	6	20	1,000
1,4-Dioxane	123-91-1	200	500	25,000
1,1,1-Trichloroethane	71-55-6	1	5	250
Carbon tetrachloride	56-23-5	1	5	250
Bromodichloromethane	75-27-4	1	5	250
1,2-Dichloropropane	78-87-5	1	5	250

Table 1. TestAmerica Primary List Reporting Limits for 8260B/C/D (continued)

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
cis-1,3-Dichloropropene	10061-01-5	1	5	250
Trichloroethene	79-01-6	1	5	250
Dibromochloromethane	124-48-1	1	5	250
1,2-Dibromoethane	106-93-4	1	5	250
1,2,3-Trichloropropane	96-18-4	2.5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	250
Benzene	71-43-2	1	5	250
Ethylmethacrylate	97-63-2	3	5	250
trans-1,3-Dichloropropene	10061-02-6	3	5	250
Bromoform	75-25-2	1	5	250
4-Methyl-2-pentanone	108-10-1	5	20	1,000
2-Hexanone	591-78-6	5	20	1,000
Tetrachloroethene	127-18-4	1	5	250
Toluene	108-88-3	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	250
2-Chloroethyl vinyl ether[2]	110-75-8	3[2]	50	2,500
Vinyl acetate	108-05-4	3	10	500
Chlorobenzene	108-90-7	1	5	250
Ethylbenzene	100-41-4	1	5	250
Styrene	100-42-5	1	5	250
trans-1,4-Dichloro-2-butene	110-57-6	3	5	250
m- and p-Xylenes	179601-23-1	2	3.5	250
o-xylene	95-47-6	1	2.5	125
Total xylenes	1330-20-7	2	10	250
1,3-Dichlorobenzene	541-73-1	1	5	250
1,4-Dichlorobenzene	106-46-7	1	5	250
1,2-Dichlorobenzene	95-50-1	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethylvinyl ether cannot be reliably recovered from acid preserved samples

Table 2. TestAmerica 8260 Secondary List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
2,2-Dichloropropane	590-20-7	1	5	250
Bromochloromethane	74-97-5	1	5	250
1,1-Dichloropropene	563-58-6	1	5	250
1,3-Dichloropropane	142-28-9	1	5	250
1-Chlorohexane	544-10-5	1	5	500
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
Isopropylbenzene	98-82-8	1	5	250
Bromobenzene	108-86-1	1	5	250
n-Propylbenzene	103-65-1	1	5	250
2-Chlorotoluene	95-49-8	1	5	250
4-Chlorotoluene	106-43-4	1	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	250
tert-Butylbenzene	98-06-6	1	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	250
sec-Butylbenzene	135-98-8	1	5	250
4-Isopropyltoluene	99-87-6	1	5	250
n-Butylbenzene	104-51-8	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	250
Naphthalene	91-20-3	1	5	500
Hexachlorobutadiene	87-68-3	1	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	250
2-Pentanone	107-87-9	5	10	500
cis-1,4-Dichloro-2-butene	1476-11-5	3	5	250
Ethylene oxide	75-21-8	600	3,000	150,000

Table 3. TestAmerica Appendix IX List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
Allyl Chloride	107-05-1	2	10	500
Acetonitrile	75-05-8	30	100	5,000
Dichlorofluoromethane	75-43-4	2	10	25,000
Isopropyl ether	108-20-3	10	50	2,500
Chloroprene	126-99-8	1	5	500
n-Butanol	71-36-3	60	200	10,000
Propionitrile	107-12-0	20	50	1,000
Methacrylonitrile	126-98-7	10	50	2,500
Isobutanol	78-83-1	110	200	10,000
Methyl methacrylate	80-62-6	4	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	10	500
Ethyl ether	60-29-7	2	10	500
Ethyl Acetate	141-78-6	5	10	500
2-Nitropropane	79-46-9	5	10	500
Cyclohexanone[2]	108-94-1	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4. TestAmerica Non-Standard List Reporting Limits for 8260B/C/D

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
1,1,1-Trifluoro-2,2-Dichloroethane	306.83-2	2.0	5.0	1000
1,2,3-Trimethylbenzene	526-73-8	2.0	5.0	250
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	2.0	5.0	250
1,2-Dichloro-1,1,2-Trifluoroethane	354-23-4	2.0	5.0	250
1,3,5-Trichlorobenzene	108-70-3	5.00	***	***
2,2,3-Trimethylbutane	464-06-2	5.00	***	***
2,2-Dimethylpentane	590-35-2	5.00	***	***
2,3-Dimethylpentane	565-59-3	5.00	***	***
2,4-Dimethylpentane	108-08-7	5.00	***	***
2-Chloro-1,1,1-Trifluoroetane	75-88-7	5.00	5.0	250
2-Methylhexane	591-76-4	5.00	***	***
3,3-Dimethylpentane	562-49-2	5.00	***	***
3-Ethylpentane	617-78-7	5.00	***	***
3-Methylhexane	589-34-4	5.00	***	***
Chlorotrifluoroethene	79-38-9	5.00	5.0	250
Cyclohexane	110-82-7	2.0	5.0	250
Dimethyl Disulfide	624/624.1-92-0	5.00	***	***
Isopropyl Alcohol	67-63-0	40	200	10,000
Methyl Acetate	79-20-9	5.0	10	1000
Methylcyclohexane	108-87-2	1.0	5.0	250
n-Heptane	142-82-5	5.00	***	***
n-Nonyl Aldehyde	124-19-6	10.00	***	***
Pentachloroethane[2]	76-01-7	0.5	***	***
Propene Oxide	75-56-9	50	3000	250
Sec-Butyl Alcohol	78-92-2	***	200	***
Tert-amyl methyl ether	994-05-8	5	5.0	1000
Tert-butyl ethyl ether	637-92-3	5	5.0	1000
Tetrahydrothiophene	110-01-0	2.0	5.0	***

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] Due to breakdown, QC and samples being analyzed for Pentachlroethane must be preserved with acid (pH<2).

Calibration levels:
Table 5. 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water and soil-5mL purge
(Standard Mixes: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,1-Trichloroethane	1	2	5	10	20	50	100	200
1,1,2,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,2-Trichloro-1,2,2-Trifluoroethane	1	2	5	10	20	50	100	200
1,1,2-Trichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethene	1	2	5	10	20	50	100	200
1,1-Dichloropropene	1	2	5	10	20	50	100	200
1,2,3-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,3-Trichloropropane	1	2	5	10	20	50	100	200
1,2,4-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,4-Trimethylbenzene	1	2	5	10	20	50	100	200
1,2-Dibromo-3-chloropropane	1	2	5	10	20	50	100	200
1,2-Dichlorobenzene	1	2	5	10	20	50	100	200
1,2-Dichloroethane	1	2	5	10	20	50	100	200
1,2-Dichloropropane	1	2	5	10	20	50	100	200
1,3,5-Trimethylbenzene	1	2	5	10	20	50	100	200
1,3-Dichlorobenzene	1	2	5	10	20	50	100	200
1,3-Dichloropropane	1	2	5	10	20	50	100	200
1,4-Dichlorobenzene	1	2	5	10	20	50	100	200
1,4-Dioxane	20	40	100	200	400	1,000	2,000	4,000
1-Chlorohexane	1	2	5	10	20	50	100	200
2-Chloroethyl vinyl ether	1	2	5	10	20	50	100	200
2,2-Dichloropropane	1	2	5	10	20	50	100	200
2-Butanone	4	8	20	40	80	200	400	800
2-Chlorotoluene	1	2	5	10	20	50	100	200
2-Hexanone	4	8	20	40	80	200	400	800
2-Pentanone	4	8	20	40	80	200	400	800
3-Chloro-1-propene (Allyl Chloride	1	2	5	10	20	50	100	200
4-Chlorotoluene	1	2	5	10	20	50	100	200
4-Isopropyltoluene	1	2	5	10	20	50	100	200

Calibration levels:
Table 5. 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water and soil-5mL purge
(Standard Mixes: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
4-Methyl-2-pentanone	4	8	20	40	80	200	400	800
Acetone	4	8	20	40	80	200	400	800
Acrolein	10	20	50	100	200	500	1,000	2,000
Acrylonitrile	10	20	50	100	200	500	1,000	2,000
Benzene	1	2	5	10	20	50	100	200
Bromobenzene	1	2	5	10	20	50	100	200
Bromoform	1	2	5	10	20	50	100	200
Bromomethane	1	2	5	10	20	50	100	200
Carbon Disulfide	1	2	5	10	20	50	100	200
Carbon tetrachloride	1	2	5	10	20	50	100	200
Chlorobenzene	1	2	5	10	20	50	100	200
Chlorobromomethane	1	2	5	10	20	50	100	200
Chloroethane	1	2	5	10	20	50	100	200
Chloroform	1	2	5	10	20	50	100	200
Chloromethane	1	2	5	10	20	50	100	200
cis-1,2-Dichloroethene	1	2	5	10	20	50	100	200
cis-1,3-Dichloropropene	1	2	5	10	20	50	100	200
Cyclohexane	1	2	5	10	20	50	100	200
Cyclohexanone	40	80	200	400	800	2,000	4,000	8,000
Chlorodibromomethane	1	2	5	10	20	50	100	200
Dibromomethane	1	2	5	10	20	50	100	200
Dichlorobromomethane	1	2	5	10	20	50	100	200
Dichlorofluoromethane	1	2	5	10	20	50	100	200
Dichlorodifluoromethane	1	2	5	10	20	50	100	200
Ethylbenzene	1	2	5	10	20	50	100	200
Ethyl Ether	1	2	5	10	20	50	100	200
Ethyl methacrylate	1	2	5	10	20	50	100	200
Ethylene dibromide	1	2	5	10	20	50	100	200
Hexachlorobutadiene	1	2	5	10	20	50	100	200
Hexane	1	2	5	10	20	50	100	200
Iodomethane	1	2	5	10	20	50	100	200
Isobutyl alcohol	25	50	125	250	500	1,250	2,500	5,000
Isopropylbenzene	1	2	5	10	20	50	100	200

Calibration levels:
Table 5. 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water and soil-5mL purge
(Standard Mixes: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
m- and p-Xylenes	1	2	5	10	20	50	100	200
Methyl Acetate	5	10	25	50	100	250	500	1,000
Methylcyclohexane	1	2	5	10	20	50	100	200
Methylene chloride	1	2	5	10	20	50	100	200
Methyl tert-butyl ether (MTBE)	1	2	5	10	20	50	100	200
Naphthalene	1	2	5	10	20	50	100	200
n-Butylbenzene	1	2	5	10	20	50	100	200
n-Propylbenzene	1	2	5	10	20	50	100	200
o-Xylene	1	2	5	10	20	50	100	200
sec-Butylbenzene	1	2	5	10	20	50	100	200
sec- Butyl Alcohol	30	60	150	300	600	1,500	3,000	6,000
Styrene	1	2	5	10	20	50	100	200
2-Methyl-2-propanol (tert-Butyl alcohol)	10	20	50	100	200	500	1,000	2,000
tert-Butylbenzene	1	2	5	10	20	50	100	200
Tetrachloroethene	1	2	5	10	20	50	100	200
Tetrahydrofuran	2	4	10	20	40	100	200	400
Toluene	1	2	5	10	20	50	100	200
trans-1,2-Dichloroethene	1	2	5	10	20	50	100	200
trans-1,3-Dichloropropene	1	2	5	10	20	50	100	200
trans-1,4-Dichloro-2-butene	1	2	5	10	20	50	100	200
Trichloroethene	1	2	5	10	20	50	100	200
Trichlorofluoroethane	1	2	5	10	20	50	100	200
Vinyl Acetate	2	4	10	20	40	100	200	400
Vinyl chloride	1	2	5	10	20	50	100	200

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Calibration levels:

Table 5A: 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water and soil-5mL purge

(Standards: MV-Supp A and Freon_A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloro-ethane	2	5	10	20	50	100	200
1,2,3-Trimethylbenzene	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2,2-tetrafluroethane	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2-trifluoroethane	2	5	10	20	50	100	200
2-Chloro-1,1,1-Trifluoroethane	2	5	10	20	50	100	200
2-Chloro-1,3-butadiene (Chloroprene)	2	5	10	20	50	100	200
2-Nitropropane	4	10	20	40	100	200	400
Acetonitrile	20	50	100	200	500	1,000	2,000
cis-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Chlorotrifluoroethene	2	5	10	20	50	100	200
Ethanol	120	300	600	1200	3000	6,000	12,000
Ethyl Acetate	4	10	20	40	100	200	400
Ethylene Oxide	400	1,000	2,000	4,000	10,000	20,000	40,000
Isopropyl alcohol	20	50	100	200	500	1,000	2,000
Isopropyl ether	2	5	10	20	50	100	200
Methylacrylonitrile	20	50	100	200	500	1,000	2,000
Methyl methacrylate	4	10	20	40	100	200	400
n-Butanol	50	125	250	500	1,250	2,500	5,000
Propene Oxide	100	250	500	1,000	2,500	5,000	10,000
Propionitrile	20	50	100	200	500	1,000	2,000
tert-Amyl methyl ether	2	5	10	20	50	100	200
tert-Butyl ethyl ether	2	5	10	20	50	100	200
Tetrahydrothiophene	2	5	10	20	50	100	200

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.3*	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.3*	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.3*	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.3*	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.3*	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.3*	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.3*	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.3*	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.3*	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.3*	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.3*	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.3*	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.3*	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.3*	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.3*	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,4-Dioxane	6*	20	40	100	200	600	1,200
1-Chlorohexane	0.3*	1.0	2.0	5.0	10	30	60
2-Chloroethyl vinyl ether	0.3*	1.0*	2.0	5.0	10	30	60
2,2-Dichloropropane	0.3*	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	1.2*	4.0	8.0	20	40	120	240
2-Chlorotoluene	0.3*	1.0	2.0	5.0	10	30	60
2-Hexanone	1.2*	4.0	8.0	20	40	120	240
2-Pentanone	1.2*	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl Chloride)	0.3*	1.0	2.0	5.0	10	30	60
2-Methyl-2-propanol (tert-Butyl alcohol)	3.0*	10	20	50	100	300	600
4-Chlorotoluene	0.3*	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.3*	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	1.2*	4.0	8.0	20	40	120	240
Acetone	1.2*	4.0*	8.0	20	40	120	240

Table 6: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Acrolein	3*	10	20	50	100	300	600
Acrylonitrile	3*	10	20	50	100	300	600
Benzene	0.3	1.0	2.0	5.0	10	30	60
Bromobenzene	0.3*	1.0	2.0	5.0	10	30	60
Bromoform	0.3*	1.0	2.0	5.0	10	30	60
Bromomethane	0.3*	1.0	2.0	5.0	10	30	60
Carbon Disulfide	0.3*	1.0*	2.0	5.0	10	30	60
Carbon tetrachloride	0.3	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.3	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.3*	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.3	1.0	2.0	5.0	10	30	60
Chloroethane	0.3*	1.0*	2.0	5.0	10	30	60
Chloroform	0.3	1.0	2.0	5.0	10	30	60
Chloromethane	0.3*	1.0*	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.3*	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Cyclohexane	0.3*	1.0	2.0	5.0	10	30	60
Cyclohexanone	12*	40	80	200	400	1,200	2,400
Dibromomethane	0.3*	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	0.3*	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.3*	1.0*	2.0	5.0	10	30	60
Ethylbenzene	0.3*	1.0	2.0	5.0	10	30	60
Ethyl Ether	0.3*	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	0.3*	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.3*	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.3	1.0	2.0	5.0	10	30	60
Hexane	0.3*	1.0	2.0	5.0	10	30	60
Iodomethane	0.3*	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	7.5*	25	50	125	250	750	1,500
Isopropylbenzene	0.3*	1.0	2.0	5.0	10	30	60
m and p Xylenes	0.3*	1.0	2.0	5.0	10	30	60
Methyl Acetate	1.5*	5.0	10	25	50	150	300
Methylcyclohexane	0.3*	1.0	2.0	5.0	10	30	60
Methylene chloride	0.3*	1.0	2.0	5.0	10	30	60

Table 6: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Methyl tert-butyl ether (MTBE)	0.3*	1.0	2.0	5.0	10	30	60
Naphthalene	0.3*	1.0	2.0	5.0	10	30	60
n-Butylbenzene	0.3*	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.3*	1.0	2.0	5.0	10	30	60
o-Xylene	0.3*	1.0	2.0	5.0	10	30	60
sec-Butylbenzene	0.3*	1.0	2.0	5.0	10	30	60
sec-Butyl Alcohol	9.0*	30	60	150	300	900	1,800
Styrene	0.3*	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.3*	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.3*	1.0	2.0	5.0	10	30	60
Tetrahydrofuran	0.6*	2.0	4.0	10	20	60	120
Toluene	0.3*	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.3*	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.3*	1.0	2.0	5.0	10	30	60
trans-1,4-Dichloro-2-butene	0.3*	1.0	2.0	5.0	10	30	60
Trichloroethene	0.3*	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.3*	1.0*	2.0	5.0	10	30	60
Vinyl Acetate	0.6*	2.0	4.0	10	20	60	120
Vinyl chloride	0.3	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
* These compounds are not routinely used at this concentration of the calibration.

Table 6A: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-Supp A and Freon_A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,1-Trifluoro-2,2-dichloroethane	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	1.0	2.0	5.0	10	30	60
2-Chloro-1,1,1-Trifluoroethane	1.0	2.0	5.0	10	30	60
2-Chloro-1,3-butadiene (Chloroprene)	1.0	2.0	5.0	10	30	60
2-Nitropropane	1.0	2.0	5.0	10	30	60
Acetonitrile	10	20	50	100	300	600
cis-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Chlorotrifluoroethene	1.0	2.0	5.0	10	30	60
Ethanol	60	120	300	600	1,800	3,600
Ethyl acetate	2.0	4.0	10	20	60	120
Ethylene oxide	200	400	1,000	2,000	6,000	12,000
Isopropyl Alcohol	10	20	50	100	300	600
Isopropyl Ether	1.0	2.0	5.0	10	30	60
Methylacrylonitrile	10	20	50	100	300	600
Methyl methacrylate	2.0	4.0	8.0	20	60	120
n-Butanol	25	50	125	250	750	1,000
Propene oxide	50	100	250	500	1,500	3,000
Propionitrile	10	20	50	100	300	600
tert-Amyl methyl ether	1.0	2.0	5.0	10	30	60
tert-Butyl ethyl ether	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane-d4 (surr)	1.0*	2.0	5.0	10	30	60
Toluene-d8 (surr)	1.0*	2.0	5.0	10	30	60
4-Bromofluorobenzene (surr)	1.0*	2.0	5.0	10	30	60
Dibromofluoromethane (surr)	1.0*	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
* These compounds are not routinely used at this concentration of the calibration.

Table 7: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main A, MV-GasKet A, and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,4-Dioxane	10	20	40	100	200	600	1,200
1-Chlorohexane	0.5	1.0	2.0	5.0	10	30	60
2-Chloroethyl vinyl ether	0.5	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	2.0	4.0	8.0	20	40	120	240
2-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
2-Hexanone	2.0	4.0	8.0	20	40	120	240
2-Pentanone	2.0	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl Chloride)	0.5	1.0	2.0	5.0	10	30	60
2-Methyl-2-propanol (tert-Butyl alcohol)	10	20	40	100	200	600	1,200
4-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.5	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	2.0	4.0	8.0	20	40	120	240
Acetone	2.0	4.0	8.0	20	40	120	240
Acrolein	5	10	20	50	100	300	600

Table 7: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main A, MV-GasKet A, and MV-2 Cleve) (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Acrylonitrile	5	10	20	50	100	300	600
Benzene	0.5	1.0	2.0	5.0	10	30	60
Bromobenzene	0.5	1.0	2.0	5.0	10	30	60
Bromoform	0.5	1.0	2.0	5.0	10	30	60
Bromomethane	0.5	1.0	2.0	5.0	10	30	60
Carbon Disulfide	0.5	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.5	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.5	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.5	1.0	2.0	5.0	10	30	60
Chloroethane	0.5	1.0	2.0	5.0	10	30	60
Chloroform	0.5	1.0	2.0	5.0	10	30	60
Chloromethane	0.5	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexane	0.5	1.0	2.0	5.0	10	30	60
Cyclohexanone	20	40	80	200	400	1,200	2,400
Dibromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethylbenzene	0.5	1.0	2.0	5.0	10	30	60
Ethyl Ether	0.5	1.0	2.0	5.0	10	30	60
Ethyl Methacrylate	0.5	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.5	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.5	1.0	2.0	5.0	10	30	60
Hexane	0.5	1.0	2.0	5.0	10	30	60
Iodomethane	0.5	1.0	2.0	5.0	10	30	60
Isobutyl Alcohol	12.5	25	50	125	250	750	1,500
Isopropylbenzene	0.5	1.0	2.0	5.0	10	30	60
m and p Xylenes	0.5	1.0	2.0	5.0	10	30	60
Methyl Acetate	2.5	5.0	10	25	50	150	300
Methylcyclohexane	0.5	1.0	2.0	5.0	10	30	60
Methylene chloride	0.5	1.0	2.0	5.0	10	30	60
Methy tert-butyl ether	0.5	1.0	2.0	5.0	10	30	60

Table 7: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main A, MV-GasKet A, and MV-2 Cleve) (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Naphthalene	0.5	1.0	2.0	5.0	10	30	60
n-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.5	1.0	2.0	5.0	10	30	60
o-Xylene	0.5	1.0	2.0	5.0	10	30	60
sec-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
sec-Butyl Alcohol	15	30	60	150	300	900	1,800
Styrene	0.5	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.5	1.0	2.0	5.0	10	30	60
Tetrahydrofuran	1.0	2.0	4.0	10	20	60	120
Toluene	0.5	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
trans-1,4-Dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Trichloroethene	0.5	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Vinyl Acetate	1.0	2.0	4.0	10	20	60	120
Vinyl chloride	0.5	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 7A: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp A and Freon_A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
2-Chloro-1,1,1-Trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
2-Chloro-1,3-butadiene (Chloroprene)	0.5	1.0	2.0	5.0	10	30	60
2-Nitropropane	0.5	1.0	2.0	5.0	10	30	60
Acetonitrile	5.0	10	20	50	100	300	600
cis-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Chlorotrifluoroethene	0.5	1.0	2.0	5.0	10	30	60
Ethanol	30	60	120	300	600	1,800	3,600
Ethyl acetate	1.0	2.0	4.0	10	20	60	120
Ethylene oxide	100	200	400	1,000	2,000	6,000	12,000
Isopropyl Alcohol	5	10	20	50	100	300	600
Isopropyl Ether	0.5	1.0	2.0	5.0	10	30	60
Methylacrylonitrile	5	10	20	50	100	300	600
Methyl methacrylate	1.0	2.0	4.0	8.0	20	60	120
n-Butanol	12.5	25	50	125	250	750	1,000
Propene oxide	25	50	100	250	500	1,500	3,000
Propionitrile	5	10	20	50	100	300	600
tert-Amyl methyl ether	0.5	1.0	2.0	5.0	10	30	60
tert-Butyl ethyl ether	0.5	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.5	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 8. Manually Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	20	96
Chlorobenzene-d_5	20	119
1,4-Dichlorobenzene-d_4	20	152
TBA-d9	400	65
1,4-Dioxane-d8*	400	96

NOTES:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

Table 8A. Automatically Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	250	96
Chlorobenzene-d_5	250	119
1,4-Dichlorobenzene-d_4	250	152
TBA-d9	5000	65
1,4-Dioxane-d8*	5000	96

NOTES:

1) 1 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the internal standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added internal standard concentrations.

* 1,4-Dioxane-d8 is in the Internal Standard spiked into every sample but is not associated to any analytes due to poor stability. 1,4-Dioxane must be set to “rejected” or “not needed” status in TALs.

Table 9. Manually Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	20
Dibromofluoromethane	20
Toluene-d_8	20
4-Bromofluorobenzene	20

NOTES:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9A. Automatically Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	250
Dibromofluoromethane	250
Toluene-d_8	250
4-Bromofluorobenzene	250

NOTES:

1) 1 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the surrogate standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added surrogate standard concentrations.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10. BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	Greater than 50 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

Table 11. SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	> 0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12. CCC Compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤ 30.0	≤ 20.0
1,1-Dichloroethene	≤ 30.0	≤ 20.0
Chloroform	≤ 30.0	≤ 20.0
1,2-Dichloropropane	≤ 30.0	≤ 20.0
Toluene	≤ 30.0	≤ 20.0
Ethylbenzene	≤ 30.0	≤ 20.0

Table 13. Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	--
Dichlorodifluoromethane	85	87	50, 101,103
Dibromofluoromethane	111	113	--
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	--
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	--
Acetone	43	58	--
Methylene chloride	84	49	51, 86
Tert-Butyl alcohol	59	74	--
Trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl-*tert*-butyl ether	73	41	57
Hexane	57	43	--
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	--
Tetrahydrofuran	42	71	--
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	--
Vinyl acetate	43	86	--
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121

Table 13. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Benzene	78	52	77
Trichloroethene	95	130***	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	--
Xylenes	106	91	--
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
Trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	41	76	78
Acetonitrile	41	40	--
Dichlorofluoromethane	67	69	--
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74

Table 13. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	--
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	--
2,2-Dichloropropane	77	97	--
Bromochloromethane	128	49	130
1,1-Dichloropropene	75	39	110
1,3-Dichloropropane	76	41	78
1-Chlorohexane	91	55	41
1,1,1,2-Tetrachloroethane	131	133	--
Bromobenzene	156	158	77
n-Propylbenzene	120	91	65
2-Chlorotoluene	126	91	65
1,3,5-Trimethylbenzene	105	120	77
4-Chlorobenzene	126	91	89
t-Butylbenzene	119	134	91
sec-Butylbenzene	134	105	--
4-Isopropyltoluene	119	134	91
n-Butylbenzene	91	92	134
1,2,4-Trichlorobenzene	180	182	--
Hexachlorobutadiene	225	227	223
Naphthalene	128	127	--
1,2,3-Trichlorobenzene	180	182	--
1,1,1-Trifluoro-2,2-Dichloroethane	83	133	--
1,2,3-Trimethylbenzene	105	120	91
1,2,4-Trimethylbenzene	105	120	119
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	85	87	--
1,2-Dichloro-1,1,2-Trifluoroethane	117	67	85
1,3,5-Trichlorobenzene	180	182	184
2,2,3-Trimethylbutane	57	43	85
2,2-Dimethylpentane	57	43	85
2,3-Dimethylpentane	56	71	73

Table 13. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
2,4-Dimethylpentane	43	57	85
2-Chloro-1,1,1-Trifluoroetane	118	83	69
2-Methylhexane	43	85	57
3,3-Dimethylpentane	43	71	--
3-Ethylpentane	43	70	71
3-Methylhexane	43	57	71
4-Chlorotoluene	126	91	89
2-Pentanone	43	86	--
Chlorotrifluoroethene	116	66	97
Cis-1,4-Dichloro-2-butene	53	75	89
Cyclohexane	56	84	55
Dimethyl Disulfide	94	79	45
Ethylene Dibromide	107	109	--
Ethylene Oxide	43	44	--
Isopropyl Alcohol	45	43	--
Methyl Acetate	43	74	59
Methylcyclohexane	55	83	41
m-Xylene & p-Xylene	91	106	77
n-Heptane	43	100	71
n-Nonyl Aldehyde	46	44	207
O-Xylene	106	91	--
Propene Oxide	58	43	57
Sec-Butyl Alcohol	45	59	--
Tert-amyl methyl ether	73	55	87
Tert-butyl ethyl ether	59	87	57
Tetrahydrothiophene	60	88	45

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-butanone, but m/z 72 must be present for positive identification.

*** Used as quantitation ion for method 624/624.1.

Table 14. State of Arizona ICV/CCV Quality Control Limits

QC Limits not specified in method	Default QC (method specified or laboratory historical if not specified)
CCV Non-CCC compounds	CCC limits (≤30%)
ICV	Same as CCV (≤30%)
Reporting Limit	Must be supported by low level initial calibration standard
LCS/LCSD	Lab historical
MS/MSD	Lab historical

NOTES:

1) Based on ADHS Rule A.A.C.R9-14-615.C.8. Director approved on June 29, 2005 for the labs to use default limits as an alternative to developing statistically derived limits.

Table 15. List 1 Poorly Performing Compounds

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer."

Acetone	1,2-Dichloro-1,1,2,2-tetrafluoroethane
Acetonitrile	Ethanol
Acrolein	Ethyl acetate
Acrylonitrile	Ethylene oxide
n-Butanol	2-Hexanone
2-Butanone (MEK)	Isobutyl alcohol
tert-Butyl alcohol	Isopropanol
Carbon disulfide	Methacrylonitrile
2-Chloroethyl vinyl ether	Methyl acetate
2-Chloro-1,1,1-trifluoroethane	4-Methyl-2-pentanone
Chlorotrifluoroethene	2-Nitropropane
cis-1,4-Dichloro-2-butene	2-Pentanone
trans-1,4-Dichloro-2-butene	2-Propanol
Dichlorodifluoromethane	Propionitrile
Dichlorofluoromethane	Tetrahydrofuran
1,2-Dibromo-3-chloropropane (DBCP)	Tetrahydrothiophene
1,2-Dichlorotetrafluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane
1,2-Dichloro-1,1,2-trifluoroethane (Freon 123a)	Trichlorofluoromethane
2,2-Dichloro-1,1,1-trifluoroethane	Vinyl acetate
1,4-Dioxane	

Table A-1. Method 624/624.1 Analytes and Reporting Limits, 5-mL Purge

Analytes	µg/L
Acrolein[1]	100
Acrylonitrile[1]	100
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	10
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	10
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	10
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	15
Vinyl chloride	10

[1] Acrolein and Acrylonitrile have been added to the 624/624.1 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624/624.1 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis.

Table A-2. Method 624/624.1 QC Acceptance Criteria

Table 7 – LCS (Q), DOC (s and x̄), and MS/MSD (P and RPD) Acceptance Criteria [1]					
Analyte	**Range for Q (%)**	**Limit for s (%)**	**Range for x̄(%)**	**Range for P_1, P_2**	**Limit for RPD**
Acrolein	60-140	30	50-150	40-160	60
Acrylonitrile	60-140	30	50-150	40-160	60
Benzene	65-135	33	75-125	37-151	61
Bromodichloromethane	65-135	34	50-140	35-155	56
Bromoform	70-130	25	57-156	45-169	42
Bromomethane	15-185	90	D-206	D-242	61
Carbon tetrachloride	70-130	26	65-125	70-140	41
Chlorobenzene	65-135	29	82-137	37-160	53
Chloroethane	40-160	47	42-202	14-230	78
2-Chloroethylvinyl ether	D-225	130	D-252	D-305	71
Chloroform	70-135	32	68-121	51-138	54
Chloromethane	D-205	472	D-230	D-273	60
Dibromochloromethane	70-135	30	69-133	53-149	50
1,2-Dichlorobenzene	65-135	31	59-174	18-190	57
1,3-Dichlorobenzene	70-130	24	75-144	59-156	43
1,4-Dichlorobenzene	65-135	31	59-174	18-190	57
1,1-Dichloroethane	70-130	24	71-143	59-155	40
1,2-Dichloroethane	70-130	29	72-137	49-155	49
1,1-Dichloroethene	50-150	40	19-212	D-234	32
trans-1,2-Dichloroethene	70-130	27	68-143	54-156	45
1,2-Dichloropropane	35-165	69	19-181	D-210	55
cis-1,3-Dichloropropene	25-175	79	5-195	D-227	58
trans-1,3-Dichloropropene	50-150	52	38-162	17-183	86
Ethyl benzene	60-140	34	75-134	37-162	63
Methylene chloride	60-140	192	D-205	D-221	28
1,1,2,2-Tetrachloroethane	60-140	36	68-136	46-157	61
Tetrachloroethene	70-130	23	65-133	64-148	39
Toluene	70-130	22	75-134	47-150	41
1,1,1-Trichloroethane	70-130	21	69-151	52-162	36
1,1,2-Trichloroethane	70-130	27	75-136	52-150	45
Trichloroethene	65-135	29	75-138	70-157	48
Trichlorofluoromethane	50-150	50	45-158	17-181	84
Vinyl chloride	5-195	100	D-218	D-251	66

[1] Criteria were calculated using an LCS concentration of 20 µg/L

Q = Percent recovery in calibration verification/LCS (Section 8.4)
s = Standard deviation of percent recovery for four recovery measurements (Section 8.2.4)
x̄ = Average percent recovery for four recovery measurements (Section 8.2.4)
P = Percent recovery for the MS or MSD (Section 8.3.3)
D = Detected; result must be greater than zero

Table 17
Associated Surrogates and Internal Standards for 8260B/C/D

Analyte	Associated IS	Associated Surrogate
1,1,1,2-Tetrachloroethane	2	2
1,1,1-TCA	1	1
1,1,2,2-Tetrachloroethane	3	3
1,1,2-TCA	1	4
1,1-DCA	1	1
1,1-DCE	1	1
1,1-Dichloropropene	1	4
1,2,3-Trichlorobenzene	3	3
1,2,3-Trichloropropane	3	3
1,2,4-Trichlorobenzene	3	3
1,2,4-Trimethylbenzene	3	3
1,2-DCA	1	4
1,2-DCB	3	3
1,2-Dibromo-3-chloropropane	3	3
1,2-Dichloropropane	1	4
1,2-EDB	2	2
1,3,5-Trimethylbenzene	3	3
1,3-DCB	3	3
1,3-Dichloropropane	2	2
1,4-DCB	3	3
1-Chlorohexane	2	2
2,2-Dichloropropane	1	1
2-Chlorotoluene	3	3
4-Chlorotoluene	3	3
Acetone	1	1
Benzene	1	4
Bromobenzene	3	3
Bromochloromethane	1	1
Bromodichloromethane	1	4
Bromoform	2	2
Bromomethane	1	1
Carbon Tetrachloride	1	4
Chlorobenzene	2	2
Chloroethane	1	1
Chloroform	1	1
Chloromethane	1	1
Cis-1,2-DCE	1	1
Cis-1,3-Dichloropropene	1	4
Dibromochloromethane	2	2
Dibromomethane	1	4
Dichlorodifluoromethane	1	1
Ethylbenzene	2	2
Hexachlorobutadiene	3	3
Isopropylbenzene	3	3
M,P-Xylene	2	2
Methylene Chloride	1	1
Methyl tert-butyl ether (MTBE)	1	1

Table 17
Associated Surrogates and Internal Standards for 8260B/C/D (cont.)

Analyte	Associated IS	Associated Surrogate
MEK (2-Butanone)	1	1
MIBK (4-Methyl-2-Pentanone)	1	4
n-Butylbenzene	3	3
n-Propylbenzene	3	3
Napthalene	3	3
o-Xylene	2	2
p-Isopropyltoluene	3	3
sec-Butylbenzene	3	3
Styrene	2	2
TCE	1	4
tert-butylbenzene	3	3
Tetrachloroethene	2	2
Toluene	1	4
trans-1,2-DCE	1	1
trans-1,3-Dichloropropene	1	4
Trichlorofluoromethane	1	1
Vinyl Chloride	1	1
Chlorotrifluoroethene	1	1
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	1	1
2-Chloro-1,1,1-Trifluoroethane	1	1
Ethylene Oxide	1	1
Ethanol	4	1
Ethyl Ether	1	1
1,2-Dichloro-1,1,2-Trifluoroethane	1	1
Propene Oxide	1	1
1,1,1-Trifluoro-2,2-dichloroethane	1	1
Acrolein	1	1
Iodomethane	1	1
Carbon Disulfide	1	1
3-Chloro-1-propene	1	1
Acetonitrile	1	1
Methyl Acetate	1	1
2-Methyl-2-propanol (TBA)	4	1
Acrylonitrile	1	1
Hexane	1	1
Vinyl Acetate	1	1
Isopropyl Alcohol	4	1
Isopropyl Ether	1	1
2-Chloro-1,3-butadiene	1	1
Tert-butyl ethyl ether (ETBE)	1	1
Propionitrile	1	1
Ethyl Acetate	1	1
sec-Butyl Alcohol	4	1
Methacrylonitrile	1	1
Tetrahydrofuran	1	1
Cyclohexane	1	1
Tert-amyl methyl ether (TAME)	1	1
Isobutyl Alcohol	4	1
n-Butanol	4	1

Table 17
Associated Surrogates and Internal Standards for 8260B/C/D (cont.)

2-Pentanone	1	4
Methycyclohexane	1	4
Methy Methacrylate	1	4
1,4-Dioxane	1	4
2-Nitropropane	1	4
2-Chloroethyl vinyl ether (2-Cleve)	1	4
Ethyl Methacrylate	1	4
2-Hexanone	2	2
Tetrahydrothiophene	2	2
1-Chlorohexane	2	2
cis-1,4-Dichloro-2-butene	3	3
Cyclohexanone	2	2
Trans-1,4-Dichloro-2-butene	3	3
Surrogates	Associated IS	Associated Surrogate
Dibromofluoromethane	1	1
Toluene-d8	2	2
4-Bromofluorobenzene	3	3
1,2-DCA-d4	1	4
Internal Standards	Associated IS	Associated Surrogate
Fluorobenzene	1	-
Chlorobenzene-d_5	2	-
1,4-Dichlorobenzene	3	-
TBA-d_9	4	-
1,4-Dioxane-d_8*	5	-

* 1,4-Dioxane-d_8 is in the Internal Standard spiked into every sample but is not associated to any analytes due to poor stability. 1,4-Dioxane-d_8 must be set to “rejected” or “not needed” status in TALS.

In the event that a compound is not covered by this table is requested the associated internal standard/surrogate will be used as required by the method.

Table 18: 8260C (D) minimum response factors

Compound	Minimum response factor
1,1,1,2-Tetrachloroethane	0.010
1,1,1-Trichloroethane	0.100 (0.050)
1,1,2,2-Tetrachloroethane	0.300 (0.200)
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.100 (0.050)
1,1,2-Trichloroethane	0.100 (0.200)
1,1-Dichloroethane	0.200 (0.300)
1,1-Dichloroethene	0.100 (0.060)
1,1-Dichloropropene	0.010
1,2,3-Trichlorobenzene	0.010 (0.400)
1,2,3-Trichloropropane	0.010
1,2,4-Trichlorobenzene	0.200
1,2,4-Trimethylbenzene	0.010 (0.400)
1,2-Dibromo-3-chloropropane	0.050 (0.010)
1,2-Dichlorobenzene	0.400 (0.600)
1,2-Dichloroethane	0.100 (0.070)
1,2-Dichloropropane	0.100 (0.200)
1,3,5-Trimethylbenzene	0.010
1,3-Dichlorobenzene	0.600 (0.500)
1,3-Dichloropropane	0.010
1,4-Dichlorobenzene	0.500 (0.600)
1,4-Dioxane	0.001
1-Chlorohexane	0.010
2-Chloroethyl vinyl ether	0.010
2,2-Dichloropropane	0.010
2-Butanone (MEK)	0.100 (0.010)
2-Chlorotoluene	0.010
2-Hexanone	0.100 (0.010)
2-Pentanone	0.001
3-Chloro-1-propene (Allyl Chloride)	0.010
2-Methyl-2-propanol (tert-Butyl alcohol)	0.001
4-Chlorotoluene	0.010
4-Isopropyltoluene	0.010
4-Methyl-2-pentanone	0.100 (0.030)
Acetone	0.050 (0.010)
Acrolein	0.001
Acrylonitrile	0.010

Compound	Minimum response factor
Benzene	0.500 (0.200)
Bromobenzene	0.010
Bromoform	0.100 (0.100)
Bromomethane	0.100 (0.010)
Carbon Disulfide	0.100 (0.100)
Carbon tetrachloride	0.100 (0.100)
Chlorobenzene	0.500 (0.400)
Chlorobromomethane	0.010 (0.100)
Chlorodibromomethane	0.100 (0.300)
Chloroethane	0.100 (0.010)
Chloroform	0.200 (0.300)
Chloromethane	0.100 (0.010)
cis-1,2-Dichloroethene	0.100 (0.200)
cis-1,3-Dichloropropene	0.200 (0.300)
Cyclohexane	0.100 (0.010)
Cyclohexanone	0.001
Dibromomethane	0.010
Dichlorobromomethane	0.200 (0.200)
Dichlorofluoromethane	0.010 (0.010)
Dichlorodifluoromethane	0.100
Ethylbenzene	0.100 (0.400)
Ethyl Ether	0.010
Ethyl methacrylate	0.010
Ethylene dibromide (EDB)	0.100 (0.200)
Hexachlorobutadiene	0.010
Hexane	0.010
Iodomethane	0.010
Isobutyl alcohol	0.001
Isopropylbenzene	0.100 (0.400)
m and p Xylenes	0.100 (0.200)
Methyl Acetate	0.100 (0.010)
Methylcyclohexane	0.100 (0.050)
Methylene chloride	0.100 (0.010)
Methyl tert-butyl ether (MTBE)	0.100 (0.100)
Naphthalene	0.010
n-Butylbenzene	0.010
n-Propylbenzene	0.010
o-Xylene	0.300 (0.200)

Compound	Minimum response factor
sec-Butylbenzene	0.010
Styrene	0.300 (0.200)
tert-Butylbenzene	0.010
Tetrachloroethene	0.200 (0.100)
Tetrahydrofuran	0.001
Toluene	0.400 (0.300)
trans-1,2-Dichloroethene	0.100 (0.100)
trans-1,3-Dichloropropene	0.100 (0.300)
trans-1,4-Dichloro-2-butene	0.010
Trichloroethene	0.200 (0.200)
Trichlorofluoromethane	0.100 (0.010)
Vinyl Acetate	0.010
Vinyl chloride	0.100 (0.010)
1,2-Dichloroethane-d4 (surr)	0.010
Toluene-d8 (surr)	0.010
4-Bromofluorobenzene (surr)	0.010
Dibromofluoromethane (surr)	0.010

8260B versus 8260C/D

Method 8260C/D does not have CCC or SPCC. In the ICV and CCV all target compounds must meet the listed minimum response factor and 20% drift. In the initial calibration for 8260C/D compliant work each compound must meet 20% RSD to be a valid calibration for that compound. 8260B and 8260C/D have different control limits for spikes and surrogates. See TALs for limits.

APPENDIX A

Modifications for Analysis of Soils Collected for the State of Alaska

1. Collection and Preservation Requirements

Preservation and Holding Time for Volatiles in Soil
Method 5035A for Alaska

Container/Contents[1]	Preservation	Holding time	Analysis
Vial containing methanol and TFT surrogate	Sample is extruded into pre-tared 4 oz jar, containing 25 mL of methanol spiked with 2.5 ppm (ug/mL) of α, α, α-trifluorotoluene, cooled to ≤ 6°C and frozen upon receipt at laboratory.	14 days	Medium Level

Sample weights are calculated in the laboratory by adding the received weight of the sample into the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

2. Sample Preparation for Medium-Level Analysis – Field Preserved, AK method

a. Fill a 40 mL VOA vial with reagent water ~ 42 mL (no head space), and remove 1000 µL of water using a volumetric pipette or syringe.

b. Add 1050 µL of methanol extract to the vial and immediately cap. Invert the vial to ensure that there is no air bubble larger than 4 mm present. If a > 4 mm air bubble is present, re-prepare the sample.

c. Load the sample in the auto sampler and proceed to analyze against the methanol calibration curve.

d. As with water samples, surrogate and internal standard solutions are added by the autosampler (see Tables 7 and 7A in the main body of this SOP). The surrogate α, α, α-trifluorotoluene is added to the samples at the time of sampling. Recoveries for this surrogate will be reported in addition to recoveries for the surrogate compounds added at the time of analysis.

e. Prepare laboratory control samples by filling a 40 mL VOA vial with reagent water, and remove 1000 µL of water using a volumetric pipette or syringe. Add reagents as needed plus sufficient methanol for a total methanol volume of 1050 µL. The recommended concentration for the LCS is the same as the Level 5 of the initial calibration curve.

f. Remove a portion of the methanol extract for each sample and store in a clean Teflon-capped vial with no headspace at ≤ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

3. Percent Moisture Correction for Soils from the State of Alaska

A percent moisture correction is required for soil samples submitted from the state of AK to adjust the extraction final volume in order to allow for the miscible solvent effects. The following

formula is used to determine the corrected final volume. This calculation is performed in the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

$$V_t = [V_m + (M * W_s/100)]$$

Where:

V_t = final extract volume, corrected for moisture (mL)
V_m = volume methanol used for extraction (mL)
M = moisture content of the sample (%)
W_s = aliquot of sample extracted (g)

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Dichlorodifluoromethane	75-71-8	80
Chloromethane	74-87-3	40
Bromomethane	74-83-9	40
Vinyl chloride	75-01-4	40
Chloroethane	75-00-3	40
n-Butanol	71-36-3	800
Trichlorofluoromethane	75-69-4	40
Acrolein	107-02-8	200
Acetone	67-64-1	400
Trichlorotrifluoroethane	76-13-1	400
Iodomethane	74-88-4	500
Carbon disulfide	75-15-0	40
Methylene chloride	75-09-2	40
tert-Butyl alcohol	75-65-0	800
1,1-Dichloroethene	75-35-4	40
1,1-Dichloroethane	75-34-3	40
trans-1,2-Dichloroethene	156-60-5	40
Acrylonitrile	107-13-1	400
Methyl *tert*-butyl ether (MTBE)	1634-04-4	200
Hexane	110-54-3	400
cis-1,2-Dichloroethene	156-59-2	40
1,2-Dichloroethene (Total)	540-59-0	40
Tetrahydrofuran	109-99-9	80
Chloroform	67-66-3	40
1,2-Dichloroethane	107-06-2	40
Dibromomethane	74-95-3	40
2-Butanone	78-93-3	160
1,4-Dioxane	123-91-1	2,000
1,1,1-Trichloroethane	71-55-6	40
Carbon tetrachloride	56-23-5	40
Bromodichloromethane	75-27-4	40
1,2-Dichloropropane	78-87-5	40
Isopropyl Alcohol	67-63-0	1,000
Isopropyl ether	108-20-3	200

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
cis-1,3-Dichloropropene	10061-01-5	40
Trichloroethene	79-01-6	40
Dibromochloromethane	124-48-1	40
1,2-Dibromoethane	106-93-4	40
1,2,3-Trichloropropane	96-18-4	40
1,1,2-Trichloroethane	79-00-5	40
Benzene	71-43-2	16
Ethylmethacrylate	97-63-2	80
trans-1,3-Dichloropropene	10061-02-6	40
Bromoform	75-25-2	40
4-Methyl-2-pentanone	108-10-1	160
2-Hexanone	591-78-6	160
Tetrachloroethene	127-18-4	40
Toluene	108-88-3	40
1,1,2,2-Tetrachloroethane	79-34-5	40
2-Chloroethyl vinyl ether	110-75-8	80
Vinyl acetate	108-05-4	80
Chlorobenzene	108-90-7	40
Ethylbenzene	100-41-4	40
Styrene	100-42-5	40
trans-1,4-Dichloro-2-butene	110-57-6	400
m- and p-Xylenes	179601-23-1	80
o-Xylene	95-47-6	40
Total xylenes	1330-20-7	80
1,3-Dichlorobenzene	541-73-1	40
1,4-Dichlorobenzene	106-46-7	40
1,2-Dichlorobenzene	95-50-1	40
2,2-Dichloropropane	590-20-7	40
Bromochloromethane	74-97-5	40
1,1-Dichloropropene	563-58-6	40
1,3-Dichloropropane	142-28-9	40
1-Chlorohexane	544-10-5	80
1,1,1,2-Tetrachloroethane	630-20-6	40

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Isopropylbenzene	98-82-8	40
Bromobenzene	108-86-1	40
n-Propylbenzene	103-65-1	40
2-Chlorotoluene	95-49-8	40
4-Chlorotoluene	106-43-4	40
1,3,5-Trimethylbenzene	108-67-8	40
tert-Butylbenzene	98-06-6	40
1,2,4-Trimethylbenzene	95-63-6	40
sec-Butylbenzene	135-98-8	40
4-Isopropyltoluene	99-87-6	40
n-Butylbenzene	104-51-8	40
1,2-Dibromo-3-chloropropane	96-12-8	200
1,2,4-Trichlorobenzene	120-82-1	40
Naphthalene	91-20-3	40
Hexachlorobutadiene	87-68-3	40
1,2,3-Trichlorobenzene	87-61-6	40
Propionitrile	107-12-0	400
Cylcohexanone	108-94-1	1,600
Methyl methacrylate	80-62-6	80
Acetonitrile	75-05-8	400
Methacrylonitrile	126-98-7	400
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	160
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	160
2-Pentanone	107-87-9	600
cis-1,4-Dichloro-2-butene	1476-11-5	400
Cyclohexane	110-82-7	40
Methyl acetate	79-20-9	200
Methylcyclohexane	108-87-2	160
2-Chloro-1,3-butadiene	126-99-8	80
2-Methyl-2-propanol	75-65-0	800

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
tert-Butyl ethyl ether	637-92-3	80
1,2,3-Trimethylbenzene	526-73-8	40
Ethyl acetate	141-78-6	80
Ethyl ether	60-29-7	200
Isobutyl alcohol	78-83-1	800
Dichlorofluoromethane	75-43-4	120
Tetrahydrothiophene	110-01-0	40

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table Ap-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
1,1,1,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,1-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethene	20	40	80	200	600	2000	4000	8000
1,1-Dichloropropene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichloropropane	20	40	80	200	600	2000	4000	8000
1,2,4-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,4-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dibromo-3-chloropropane	20	40	80	200	600	2000	4000	8000
1,2-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,3,5-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,4-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,4-Dioxane	1000	2000	4000	10000	30000	100000	200000	400000
1-Chlorohexane	20	40	80	200	600	2000	4000	8000
2,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
2-Butanone (MEK)	80	160	320	800	2400	8000	16000	32000
2-Chloro-1,3-butadiene (chloroprene)	20	40	80	200	600	2000	4000	8000
2-Chlorotoluene	20	40	80	200	600	2000	4000	8000
2-Hexanone	80	160	320	800	2400	8000	16000	32000
2-Methyl-2-propanol (tert-Butyl alcohol)	400	800	1600	4000	12000	40000	80000	160000
4-Chlorotoluene	20	40	80	200	600	2000	4000	8000
4-Isopropyltoluene	20	40	80	200	600	2000	4000	8000
4-Methyl-2-pentanone	80	160	320	800	2400	8000	16000	32000
Acetone	80	160	320	800	2400	8000	16000	32000
Acetonitrile	200	400	800	2000	6000	20000	40000	80000
Acrolein	200	400	800	2000	6000	20000	40000	80000
Acrylonitrile	200	400	800	2000	6000	20000	40000	80000

Table Ap-2

Calibration Levels for 8260, 5035FM_AK (ug/Kg)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Benzene	20	40	80	200	600	2000	4000	8000
Bromobenzene	20	40	80	200	600	2000	4000	8000
Bromoform	20	40	80	200	600	2000	4000	8000
Bromomethane	20	40	80	200	600	2000	4000	8000
Carbon tetrachloride	20	40	80	200	600	2000	4000	8000
Chlorobenzene	20	40	80	200	600	2000	4000	8000
Chlorobromomethane	20	40	80	200	600	2000	4000	8000
Chlorodibromomethane	20	40	80	200	600	2000	4000	8000
Chloroethane	20	40	80	200	600	2000	4000	8000
Chloroform	20	40	80	200	600	2000	4000	8000
Chloromethane	20	40	80	200	600	2000	4000	8000
cis-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
cis-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Cyclohexanone	20	40	80	200	300	1000	2000	4000
Dibromomethane	20	40	80	200	600	2000	4000	8000
Dichlorobromomethane	20	40	80	200	600	2000	4000	8000
Dichlorodifluoromethane	20	40	80	200	600	2000	4000	8000
Ethylbenzene	20	40	80	200	600	2000	4000	8000
Ethylene dibromide (EDB)	20	40	80	200	600	2000	4000	8000
Hexachlorobutadiene	20	40	80	200	600	2000	4000	8000
Iodomethane	20	40	80	200	600	2000	4000	8000
Isopropyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Isopropyl ether	100	200	400	1000	3000	10000	20000	40000
Isopropylbenzene	20	40	80	200	600	2000	4000	8000
m- and p-Xylenes	40	80	160	400	1200	4000	8000	16000
Methacrylonitrile	200	400	800	2000	6000	20000	40000	80000
Methylene chloride	20	40	80	200	600	2000	4000	8000
Naphthalene	20	40	80	200	600	2000	4000	8000
n-Butanol	600	1200	2400	6000	18000	60000	120000	240000
n-Butylbenzene	20	40	80	200	600	2000	4000	8000
n-Propylbenzene	20	40	80	200	600	2000	4000	8000
o-Xylene	20	40	80	200	600	2000	4000	8000
Propionitrile	200	400	800	2000	6000	20000	40000	80000
sec-Butylbenzene	20	40	80	200	600	2000	4000	8000
Styrene	20	40	80	200	600	2000	4000	8000
tert-Butylbenzene	20	40	80	200	600	2000	4000	8000

Table Ap-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
Tetrachloroethene	20	40	80	200	600	2000	4000	8000
Tetrahydrothiophene	20	40	80	200	600	2000	4000	8000
Toluene	20	40	80	200	600	2000	4000	8000
trans-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
trans-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Trichloroethene	20	40	80	200	600	2000	4000	8000
Trichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Vinyl chloride	20	40	80	200	600	2000	4000	8000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table Ap-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1-Trifluoro-2,2-dichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloro-1,2,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
1,2,3-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2,2-tetrafluoroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
2-Chloroethy vinyl ether	20	40	80	200	600	2000	4000	8000
2-Nitropropane	20	40	80	200	600	2000	4000	8000
2-Pentanone	80	160	320	800	2400	8000	16000	32000
3-Chloro-1-propene (Allyl chloride)	20	40	80	200	600	2000	4000	8000
Carbon disulfide	20	40	80	200	600	2000	4000	8000
cis-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Cyclohexane	20	40	80	200	600	2000	4000	8000
Dichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Ethyl acetate	40	80	160	400	1200	4000	8000	16000
Ethyl ether	20	40	80	200	600	2000	4000	8000
Ethyl methacrylate	40	80	160	400	1200	4000	8000	16000
Ethylene oxide	2500	5000	10000	25000	75000	250000	500000	1000000

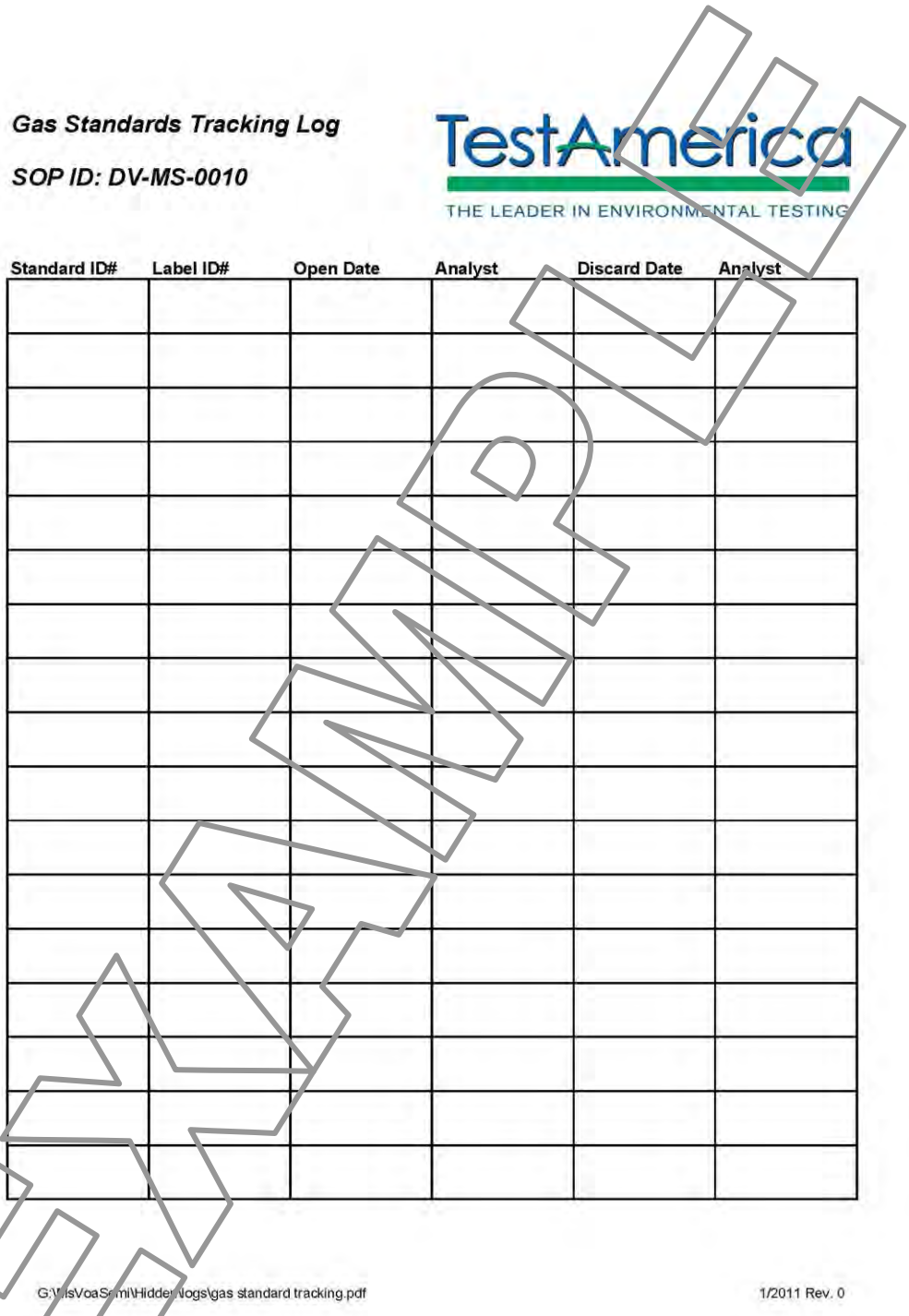
Table Ap-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Hexane	20	40	80	200	600	2000	4000	8000
Isobutyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Methyl acetate	100	200	400	1000	3000	10000	20000	40000
Methylcylcohexane	20	40	80	200	600	2000	4000	8000
Methyl methacrylate	40	80	160	400	1200	4000	8000	16000
Methyl *tert*-butyl ether (MTBE)	20	40	80	200	600	2000	4000	8000
Propene oxide	400	800	1600	4000	12000	40000	80000	160000
sec-Butyl alcohol	600	1200	2400	6000	18000	60000	120000	240000
tert-Amyl methyl ether	100	200	400	1000	3000	10000	20000	40000
tert-Butyl ethyl ether	100	200	400	1000	3000	10000	20000	40000
Tetrahydrofuran	40	80	160	400	1200	4000	8000	16000
trans-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Vinyl acetate	40	80	160	400	1200	4000	8000	16000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Attachment 1

Gas Standards Tracking Log



Gas Standards Tracking Log

SOP ID: DV-MS-0010

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Standard ID#	Label ID#	Open Date	Analyst	Discard Date	Analyst

G:\MsVoaSemi\Hidden\logs\gas standard tracking.pdf 1/2011 Rev. 0

eurofins | Environment Testing TestAmerica

Eurofins TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

Eurofins TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Mercury in Solids by Cold Vapor Atomic Absorption

Approvals (Signature/Date):

Doug Gomer — 9/18/19 — Date
Technical Manager

Reed Pottruff — 9/18/19 — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan — 9/18/19 — Date
Quality Assurance Manager

Scott Hall — 9/19/19 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS# 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A or 7471B.

1.2 Method 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, and sludge-type materials. All matrices require sample preparation prior to analysis. This is not an appropriate procedure for the digestion of tissues or other organic matrices, which require the use of EPA 245.6 instead.

1.3 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

1.4 The routine reporting limit for mercury in solid matrices is 17 µg/kg.

2.0 Summary of Method

A representative portion of the sample is digested in aqua regia in the first digestion cycle and potassium permanganate in the second cycle. Mercury is reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorption of light at 253.7 nm is calibrated as a function of mercury concentration.

3.0 Definitions

3.1 Total Mercury: Inorganic forms of mercury are effectively dissolved by the acids used in the digestion. The potassium permanganate reagent breaks down organo-mercury compounds to inorganic forms that are detected by this procedure.

3.2 Aqua Regia: A 3:1 mixture of hydrochloric and nitric acids. This mixture is effective at dissolving metals in the solid form.

3.3 General Analytical Terms: Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Potassium permanganate “suitable for mercury determination” is specified because of the potential for mercury contamination in the reagent. In addition, potassium permanganate crystals will absorb mercury vapors from the air. Reagent bottles must be kept tightly closed to avoid contamination.

4.2 Potassium permanganate, in addition to breaking down organic compounds, also

eliminates possible interferences from sulfide. Concentrations as high as 20 ppm of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 ppm had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

NOTE: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be lessened by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe,

nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. A disposable face shield should be used when making up aqua regia.

5.3.2 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury Nitrate Solutions	Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydroxylamine Hydrochloride	Corrosive Poison	No OSHA PEL listed for this compound	Direct contact with skin or eyes causes irritation. May cause skin sensitization, an allergic reaction. Inhalation or ingestion may cause methemoglobinemia and resulting cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood), and labored breathing.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90 – 95 °C.

6.1.2 Mercury Autoanalyzers:

CETAC Mercury Analyzer with Autosampler and Auto-Diluter

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Disposable digestion tubes with caps from Environmental Express, volume accuracy verified to ASTM Class A standards at all gradations.

6.3.2 Disposable glass autosampler tubes, 16 mm x 100 mm

6.3.3 Argon, 99.999% purity

6.3.4 Calibrated mechanical pipettes (see SOP DV-QA-0008 for details on calibrating mechanical pipettes).

6.3.5 Class A volumetric flasks.

6.3.6 Thermometer, non-mercury column, accurate to ± 1 °C at 95 °C (see SOP DV-QA-0001 for calibration details).

6.3.7 Glass beads, < 1 mm diameter, acid washed.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Primary and secondary standards used for data production are recorded in the Reagent Module of the TestAmerica LIMS (TALS).

7.1 Reagent water: Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 Nitric acid (HNO_3): Concentrated, trace metal grade or better.

7.3 Hydrochloric acid (HCl): Concentrated, trace metal grade or better.

7.4 Aqua Regia: Add 75 mL concentrated HCL and 25 mL concentrated HNO3 to a 100 mL container. Aqua Regia will be prepared immediately before use.

7.5 Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO_3:

7.5.1 Add 0.5 L of concentrated HNO_3 to a 50 L carboy partially filled with reagent water.

7.5.2 Dilute to 50 L with reagent water.

7.5.3 Record the acid lot number and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.6 Stannous chloride solution ($SnCl_2$), Hg grade, 10% (w/v) per manufacturer's instructions:

7.6.1 Place approximately 100 mL of reagent water into a 2 L volumetric flask.

7.6.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.3 Add 200 grams of $SnCl_2$ to the flask.

7.6.4 Mix the contents of the flask until the reagent is completely dissolved.

7.6.5 Bring solution to a final volume of 2 L with reagent water.

7.7 Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):

Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (Hg grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate, 5% solution (w/v):

Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.9 Purchased Mercury Stock Solutions

7.9.1 Second source initial calibration verification (ICV) stock solution 100 mg/L (Hg ICV Stock).

7.9.2 Primary Mercury Calibration Standard Solution, 1,000 mg/L (Hg Ultra Prim).

7.10 Monthly Calibration Working Standard Solution, 10 mg/L (Hg Mnth Spike)

7.10.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.10.2 Pipet 1.0 mL of the 1,000 mg/L primary mercury calibration standard solution (Section 7.9.2) into the flask.

7.10.3 Dilute to the mark on the flask with 1% HNO_3.

7.10.4 Stopper the flask and shake to mix.

7.10.5 Transfer the solution to a 125 mL Nalgene bottle.

7.10.6 Document the preparation of the solution in the Reagent Module in TALS.

7.10.7 Prepare this solution fresh monthly or more often if necessary.

7.11 Daily Calibration Working Spike, 100 μg/L (Hg Daily Spk)

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL volumetric flask.

7.11.2 Add 1.0 mL of the 10 mg/L Calibration Working Standard (see Section 7.10).

7.11.3 Bring the solution to a final volume of 100.0 mL.

7.11.4 Stopper and mix thoroughly.

7.11.5 Document the preparation in the Reagent Module in TALS.

7.11.6 Prepare this solution each day prior to calibration.

7.12 Initial Calibration (ICAL) Standards

The initial calibration standards are prepared directly in the digestion tubes as follow:

ICAL	Daily Calibration Working Spike (mL)	1% HNO_3 (mL)	Final Conc. (µg/L)
Blank	0.0	5.0	0.0
Std 1	0.10	4.9	0.20
Std 2	0.25	4.75	0.50
Std 3	0.50	4.5	1.0
Std 4	1.0	4.0	2.0
Std 5	2.5	2.5	5.0
Std 6	5.0	0.0	10.

7.13 Second-Source Initial Calibration Verification Intermediate Standard, 400 µg/L (Hg Biwk ICV)

Add 40 µL of the 1000 mg/L ICV Standard to a 100 mL volumetric flask partially filled with 1% HNO_3 and dilute to the mark. Record this information in the Reagent Module in TALS.

7.14 Second-Source Initial Calibration Verification (ICV) Daily Working Standard, 4.00 µg/L (Hg Soil ICV)

Add 0.5 mL of the 400 µg/L ICV Intermediate Standard (see Section 7.13) to a soil digestion tube and add 4.5 mL of 1% HNO_3. Record this information in the Reagent Module in TALS.

7.15 Continuing Calibration Verification (CCV) Standards, 5 µg/L (Hg Soil CCV)

7.15.1 The CCVs are prepared exactly as the 5.0 µg/L ICAL standard shown above (see Section 7.12).

7.15.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.16 Laboratory Control Sample (LCS), 333 µg/kg

7.16.1 The LCS is prepared in an empty digestion tube or 0.6 g of glass beads (< 1 mm) are used if required.

7.16.2 Add 2.0 mL of the 100 µg/L Daily Calibration Working Spike (see Section 7.11) to a digestion tube. See Section 9.4 for additional detail.

7.16.3 This is equivalent to 4.0 µg/L, which is the concentration that appears on the raw data printout from the instruments.

7.17 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 333 µg/kg

MS/MSD pairs are spiked in the same manner as the LCS (see section 7.16) and prepared in the same manner as the samples, using 0.6 g of sample.

7.18 Reporting Limit (CRA) Check Standard, 0.2 µg/L (Hg Soil RL)

7.18.1 Add 0.1 mL of 100 µg/L Daily Calibration Working Spike (see section 7.11) and 4.9 mL of reagent water to a digestion tube.

7.18.2 This is equivalent to a 0.2 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	10 grams	Cool, ≤ 6 °C	28 Days	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for the DoD/DOE QSM (5.0 – 5.3) unless otherwise stated. Any deviation or exceptions

from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate pair (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.3 Method Blank (MB)

The MB consists of an empty vessel (or with glass beads for DoD/DOE projects) containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank (MB) must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than ½ RL or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

NOTE: DOD QSM 5 does not allow this exception. Results may not be reported without a valid

method blank unless sample cannot be re-prepared or re-analyzed.

9.4 Laboratory Control Sample (LCS), 333 µg/kg

The preparation of the LCS is described in Section 7.16. At least one LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. DoD requires when there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Maximum control limits for LCS recoveries are 80 - 120%. In-house control limits based on three standard deviations of the mean of historical results are used as long as they are at least as tight as 80-120% (see Policy DV-QA-003P for further details on establishing control limits).

NOTE: DOD QSM 5 Solid matrix LCS Limits are 80 - 124%.

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above control limits and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

NOTE: Reporting sample results with failing LCS recoveries is not acceptable for DOD QSM 5 unless samples cannot be re-prepared or reanalyzed.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD), 333 µg/kg

One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported only for project-related samples. Samples identified as field blanks cannot be used for MS/MSD analysis. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The MS/MSD recovery must fall within 75-125%; the relative percent difference (RPD) between the MS and MSD cannot exceed 20%.

NOTE: DOD QSM 5 Solid matrix MS/MSD Limits are 80-124%.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits and note it on the final report.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the linear range and the MS/MSD results are above the linear range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within linear range and the MS/MSD results

are above the linear range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

  NOTE 1: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

  NOTE 2: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

9.6 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 2 for details).

NOTE: DOD QSM 5: Performed only when required by the project and the SD or PDS fails. Must be documented with an NCM and included in the case narrative.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure. Prepare digestion tubes containing volumes of standards required for each tube as listed in Section 7.

10.3.2 Weigh 0.5 – 0.6 g of each well-homogenized sample into a sample digestion tube. See SOP DV-QA-0023 for additional information on subsampling.

10.3.3 If preparing Incremental Samples a 3 g sample aliquot is required. This aliquot will be prepared by utilizing the procedure described in *Incremental Sampling Methodology for Soils and Sediments* (DV-OP-0013). Divide the 3 g aliquot into five 0.6 g samples. Digest the five individual aliquots and combine them back into one after adding the sodium chloride-hydroxylamine hydrochloride reagent. All batch QC samples must also be processed in this fashion.

10.3.4 Prepare a MB, LCS, MS, and MSD for each batch. The MB is either an empty digestion tube or is prepared by placing 0.6 g of glass beads in a digestion tube, depending on client requirements. The LCS is prepared by adding 2.0 mL of the 100 µg/L Daily Calibration Working Spike to a digestion tube. The MS is prepared by adding 2.0 mL of the 100 µg/L Daily Calibration Working Spike to a digestion tube containing a second aliquot of the chosen matrix sample. The MSD is prepared in the same manner as the MS using a third aliquot of the chosen sample.

NOTE: The spike must be added after the sample aliquot but before the addition of reagents.

10.3.5 Add 5.0 mL of reagent water to all un-spiked field samples and the method blanks. Add 3.0 mL of reagent water to the LCS, MS and MSD.

10.3.6 Add 5.0 mL of Aqua Regia to each tube.

10.3.7 Heat for 2 minutes at 95 ± 3 °C. Record the start and stop times and the temperature on the bench sheet in TALS.

10.3.8 Allow the samples and standards to cool at room temperature.

10.3.9 Add 19 mL of reagent water.

10.3.10 Add 15 mL of 5% potassium permanganate solution. A purple color must persist for at least 15 minutes. If the color does not persist, the sample must be re-prepared using a smaller sample aliquot.

NOTE: It is important that equal volumes of the potassium

permanganate solution are added to all solutions in the batch. Unequal volumes used with the automated method will result in dilution errors.

10.3.11 Cap the samples and standards and heat for 30 minutes at 95 ± 3 °C. Record the start and stop times and the temperature on the bench sheet in TALS. The analyst will verify that a purple color persists or a black precipitate is present after the thirty minutes of heating. If this is not true, the digestion must be repeated using a smaller sample aliquot.

10.3.12 Allow the samples and standards to cool at room temperature.

10.3.13 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate. Verify that the volume is at 50 mL.

10.3.14 For samples aliquoted using the Incremental Sampling Method combine the 5 individual sample cups for each sample and QC into one marked 250 mL container.

10.4 Calibration

10.4.1 All calibration standards are digested together with samples, as described in Section 10.3, prior to analysis. Preparation of calibration standards is described in Section 7.12.

10.4.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4.3 Detailed information regarding calibration models and calculations can be found in Corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

10.4.4 Initial Calibration (ICAL)

10.4.4.1 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4.4.2 Calibrate using six standards and a blank (see Section 7.12).

10.4.4.3 The calibration curve must have a correlation coefficient (r^2) ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. The calibration curve is processed with an unweighted linear regression.

10.4.4.4 Record the microAbsorbance (µAbs.) for the 10 ppb standard

in the instrument maintenance log.

NOTE: It is not acceptable to reject calibration points for this method.

10.4.5 Initial and Continuing Calibration Blanks

10.4.5.1 An initial calibration blank (ICB) is tested immediately after the daily ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Client specific requirements take precedence. For example, DOD QSM 5 requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.4.5.2 Continuing calibration blanks (CCBs) are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than ½ the reporting limit. As just noted, DOD QSM 5 requires that results for blanks must be less than the LOD (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.4.6 Initial Calibration Verification (ICV), 4.0 µg/L (Hg Soil ICV)

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV recovery must be within 90 - 110%.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

10.4.7 Reporting Limit Check Standard (CRA), 0.2 µg/L (Hg Soil RL)

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for QSM 5.1).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, the instrument recalibrated, and associated samples reanalyzed.

10.4.8 Continuing Calibration Verification (CCV), 5.0 µg/L (Hg Soil CCV)

Calibration accuracy is monitored during the analytical run through the analysis of a known standard after every 10 samples and at the end of the run.

Acceptance Criteria: The CCV recovery must be within 80 - 120% except for QSM 5.0 where the CCV recovery limits are 90 - 110%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be reanalyzed once without modification to the instrument‘s operating conditions. If the reanalyzed CCV is found to be in control, the CCV analysis must be repeated with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

10.4.9 CCV Acceptance Criteria for sample run under a DOD QSM 5.0 program

CCVs must have a percent recovery of 90 - 110%. If the CCV fails the following options are available: Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately (within an hour) analyzed two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate then reanalyze all affected samples since the last acceptable CCV.

10.5 Sample Analysis

10.5.1 Set up the instrument and autosampler according to the manufacturer’s

instructions.

10.5.2 Allow the samples to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.5.3 Pipet 10 mL of each sample and calibration standard into a disposable test tube for analysis

10.5.4 Analyze the standards and samples according to the manufacturer's instructions.

10.5.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples with mercury concentrations that exceed the highest calibration standard.

NOTE: The instrument auto-dilutes samples. Any samples that require greater than a 10x dilution MUST be diluted manually.

10.5.6 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be reanalyzed.

10.5.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB.

10.5.8 The analytical sequence listed below must be followed. Refer to *Quality Control* Section 9.0 for quality control limits.

Instrument Calibration
ICV
ICB
CRA
CCV
CCB
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.5.9 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.0 Calculations / Data Reduction

Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{spike concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spike concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration

$$\text{Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mL)

Note: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4 Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

11.4.1 Sample data entered into the preparation batch in TALS, which includes the batch number, list of samples, preparation analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes).

11.4.2 Raw data (direct instrument printout as a PDF) with the analyst name and all required calibration information.

11.5 Reporting

11.5.1 Standard units for reporting solid sample results are mg/kg.

11.5.2 If dilutions are required due to insufficient sample, interferences, or other problems, the reporting limit and MDL are multiplied by the dilution factor,

and the data may require flagging.

11.5.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.5.4 All associated data are entered or uploaded into TALS as required.

11.5.5 Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.5.6 The initial data review is performed by the analyst while the second-level data review is performed by the area supervisor or designee. Both reviews are documented in TALS. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. MDL verifications are performed quarterly.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require such verification. A blank matrix is spiked at 1 - 2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for

each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee) the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and Policy DV-HS-001P, *Waste Management Plan*.

14.1 The following waste streams are produced when this method is carried out:

14.1.1 Aqueous Acidic (Metals) - Corrosive - (J)

14.1.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update IV, February 2007, Method 7471B (Mercury).

15.3 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013.

15.4 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.2, 2018.

15.5 U.S.EPA Statement of Work for Inorganic Analysis, ILMO3.0

16.0 Method Modifications:

Item	Method	Modification
1	7471A	An additional QC analysis, RL verification, is added
2	7471A	Methods 7470A and 7471A state that working standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory had developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing (PT) results for samples that were prepared and analyzed within 24 hours, but on successive days • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
3	7471A	Chapter 1 of SW-846 specifies the use of reagent water with a purity equivalent of ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
4	7471A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of

Item	Method	Modification
		interest at or above ½ the reporting limit.
5	7471B	Method 7471B uses reagent water for the method blank. TestAmerica Denver is currently using glass beads when required.
6	7471B	Section 11.1 requires 50 mL of reagent water to be added to the sample with 15 mL of Potassium permanganate. TestAmerica Denver utilizes digestion tubes which do not allow for 50 mL of reagent water. 19 mL of reagent water is currently being added.
7	7471A	Section 7.1 specifies triplicate 0.2 g portions of sample for solids analysis. TestAmerica Denver instead utilizes a 0.5 - 0.6 g weight range in order to avoid targeting of specific weights and to comply with the requirements of the most recent method revision.

17.0 Attachments

Attachment 1: Summary of Quality Control Requirements
Attachment 2: MSA Guidance
Attachment 3: Instrument Maintenance
Attachment 4: Troubleshooting Guide

18.0 Revision History

- Revision 13 dated 19 September 2019
  - Updated standard concentration and spiking amount in Section 7.13.

- Revision 12 dated 31 May 2019
  - Annual technical review.
  - Minor formatting and language corrections throughout
  - Removed Section 3.1 due to redundancy
  - Updated amount of reagent water added in Section 10.3.5 to 3 mL.
  - Added QSM 5.1 CRA criteria to Section 10.4.7 and Attachment #1.
  - Removed Section 11.4.3 regarding data checklists
  - Removed reference to QSM 4.2 and added reference to QSM 5.2 in Section 15

- Revision 11 dated 31 May 2018
  - Annual Technical review.
  - Updated the spike amount used in Section 10.3.4 to 2ml.

- Revision 10 dated 31 May 2017
  - Updated the spike concentration for the LCS, MS and MSD in sections 7.16 and 7.17
  - Updated true value for LCS and MS/MSD in sections 9.4 and 9.5
  - Added DoD requirement of LCSD if no MS/MSD to sections 9.4 and 9.5
  - Added clarification to section 9.5 regarding parent and MS/MSD samples that are “over range”

- Revision 9 dated 01 Feb 2017

- Annual Technical Review
- Added a paragraph to Section 6.0 regarding the documentation of equipment IDs
- Updated Section 7.4 to make the aqua regia in 100 mL container
- Added Note 2 to Section 9.5 Referencing MS/MSD Policy Memoranda
- Added a paragraph to Section 10.3 regarding the documentation of equipment IDs
- Updated language in Section 12.1 regarding MDL studies
- Added current Section 12.2 defining LOQVs

- Revision 8 dated 28 Feb 2016
  - Annual Technical Review
  - Added current Section 3.1
  - Removed reference to AFCEE program in Section 9.3
  - Modified/Updated Section 9.5 – Corrective Actions for MS/MSD failures
  - Updated Sections 12.2 & 12.3 to be more consistent with other SOPs
  - Archived revision histories 2010 and earlier – available upon request

- Revision 7 dated 28 Feb 2015
  - Annual Technical Review
  - Removed reference to purging sample headspace from Section 4.4, outdated.
  - Section 4.6 – Removed reference to method 245.6
  - Changed MSDS to SDS
  - Removed references to “Cetac Only”
  - Added Section 7.5.3 regarding recording of data in TALS
  - Added TALS reagent IDs to standard names
  - Corrected concentrations of Cal Stds
  - Corrected Section references
  - Changed units of RL std to water units
  - Changed minimum sample volume to 10 g to accord with corporate policy
  - Added DoD V5 references
  - Changed sample aliquot to 0.5 – 0.6 g
  - Deleted references to adjust volume with 1% HNO3
  - Updated temperature of digestion to 95 ± 3 oC
  - Deleted Section 10.4.4.3 (Redundant)
  - Changed “counts” in Section 10.4.4.4 to microAbsorbance
  - Removed “Approximately” from ICV true values
  - Removed GC references from Section 10.4.9
  - Removed references to “resloping”
  - Added initial CCV/CCB pair to sequence

- Revision 6 dated 28 Feb 2014
  - Annual Technical Review
  - Removed references to Serial Dilutions and Post Digestion Spikes
  - Section 10.4 for incremental sampling was merged into Section 10.3
  - Updated Section 10.4.4.1 and 10.4.4.2 to note DOD QSM 5 criteria
  - Updated Section 10.4.7 to note DOD QSM 5 CCV criteria is 90-110.
  - Updated Attachment 2 for ICB,CCB and CCV criteria to DOD QSM 5
  - Added Attachment 5 for Troubleshooting

- Revision 5 dated 15 July 2013
  - Annual Technical Review

- Correction to formatting
- Changed reference to Standards Log to Reagent Module in the LIMS
- Added General Analytical Terms information to definition section
- Edited section 7.6, 10.4.8, 12.1 & 12.2 to reflect current practices.
- Changed RL reference in sections 7.18, 10.5.6, 10.6.8 and Attachment 2 to CRA
- Removed bullet point 5 under 11.5
- Removed Attachment 3 and renumbered the subsequent attachments
- Corrected references date for section 15.2
- Added Texas TRRP to section 12.1

- Revision 4 dated 30 September 2012
  - Clarified the language in Section 9.4 to be one LCS per batch.
  - Modified Section 7.16.2 to refer to Section 10.4.4 for additional detail.

- Revision 3.2 dated 13 July 2012
  - Updated Sections 7.6 and 7.7 to state Hg grade reagents are used
  - Updated Sections 10.4.11 to include a note about bringing samples to a final volume before the sample is mixed
  - Updated Section 10.5.4 and Attachment 2 to control calibrations blanks to ½ the RL.
  - Added Sections 10.5.3.5 to record the number of counts for the 10 ppb standard
  - Formatting and grammatical changes throughout

- Revision 3.1 dated 03 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling throughout document
  - Annual Technical Review
  - Section 1.3 Added Incremental Sampling Method statement to SOP
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated section 9.5 and Attachment 2 for method blank control criteria
  - Section 10.2 Added Incremental Sampling Method preparation amount
  - Section 10.2.12 Added Incremental Sampling Method combination procedure
  - Section 10.2.13 Added Incremental Sampling Method final volume
  - Added dilution note to Section 10.3.5
  - Updated section 12.0 to reflect current laboratory practices

Earlier revision histories have been archived and are available upon request.

Attachment 1

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Immediately following ICAL	Absolute value < ½ RL (< LOD for QSM 5.0)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICV	Following ICB	90 - 110% recovery	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
RL Check Standard (CRA)	Following the ICV	50 - 150% recovery 80 - 120% for QSM 5.1	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery. 90 - 110% for QSM 5.0	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
CCB	Immediately following each CCV	Absolute value < ½ RL (< LOD for QSM 5.0)	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ≤ ½ RL Sample results greater than 10% the blank concentration are acceptable.	Redigest and reanalyze samples. Note exceptions under criteria section.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 80 - 120%	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the failed LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 75 - 125%	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate	See Matrix Spike	Recovery within statistical control limits, not to exceed 75 - 125% recovery or in-house control limits; RPD ≤ 20%	See Corrective Action for Matrix Spike.

Attachment 2

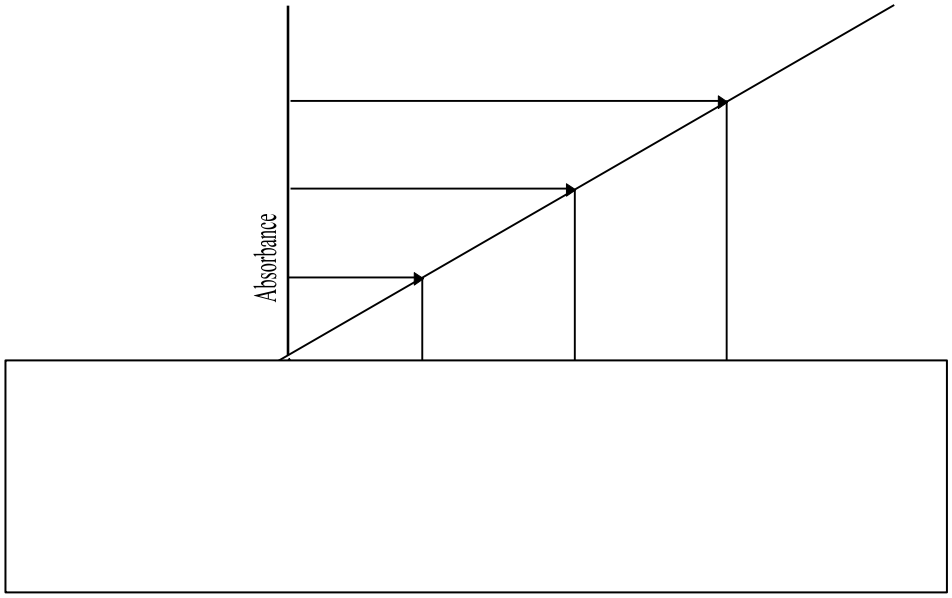
MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 3

Instrument Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time and instrument number, then identify the problem and corrective action in the maintenance log. When the instrument is returned to service, record the return to service, the date, and any tests performed to verify proper operation.

The following preventative maintenance procedures are <u>required</u> to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

Attachment 4

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell

eurofins | Environment Testing TestAmerica

Eurofins TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

Eurofins TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Mercury in Water by Cold Vapor Atomic Absorption (CVAA) [SW 7470A]

Approvals (Signature/Date):

Doug Gomer 9/18/19 Doug Gomer Date Technical Manager	Reed Pottruff 9/18/19 Reed Pottruff Date Health & Safety Manager / Coordinator
Roxanne Sullivan 9/18/19 Roxanne Sullivan Date Quality Assurance Manager	Scott Hall 9/19/19 Scott Hall Date Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A.

1.2 Method 7470 is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts.

1.3 All matrices require sample preparation prior to analysis.

1.4 The final reporting limit is 0.0002 mg/L (0.2 μg/L), except for TCLP leachates that have a 0.002 mg/L (2 μg/L) reporting limit.

2.0 Summary of Method

This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration). All sample data are uploaded to the TestAmerica LIMS (TALS).

3.0 Definitions

3.1 Dissolved Metals: Those elements that pass through a 0.45-μm membrane. (Sample is acidified after filtration).

3.2 Total Metals: The concentration determined on an unfiltered sample following digestion.

3.3 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Assurance Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Potassium permanganate, which is used to breakdown organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper also has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides will require dilution. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents,

and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.3 Potassium permanganate is a strong oxidizing agent. It is incompatible with and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.

Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90-95°C.

6.1.2 Mercury Auto-analyzers: The laboratory currently uses two CETAC QuickTrace™ Mercury Analyzer M-7500s with Autosamplers.

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Disposable 50 mL digestion tubes with caps. Accuracy at 30 mL verified to ±3% gravimetrically prior to use (by lot). See DV-QA-0008 for more information regarding volume verifications.

6.3.2 Disposable glass test tubes, 16 mm x 100 mm

6.3.3 Argon, 99.999% purity

6.3.4 Calibrated automatic pipettes or Class A glass volumetric pipettes (see SOP DV-QA-0008 for details on calibrating mechanical pipettes).

6.3.5 Class A volumetric flasks.

6.3.6 Thermometer, non-mercury column, accurate to ±1 °C at 95 °C (see SOP DV-QA-0001 for calibration details).

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the

determination. Suggested reagent and standard recipes are listed below. Alternate weights and volumes may be used as long as the final concentrations are maintained as listed and the details are recorded in the Reagent module in TALS. All standard concentrations listed below refer to the on-instrument concentration except where otherwise noted.

7.1 **Reagent water:** Must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 **Nitric acid (HNO_3):** concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** concentrated, trace metal grade or better.

7.4 **Sulfuric acid (H_2SO_4):** concentrated, trace metal grade or better.

7.5 **Reagent Blank:** This blank solution is used as the Calibration Blank (STD0), Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and as the starting solution for the Method Blanks (MBs). It is made as follows:

Add 0.5 L of concentrated HNO_3 to a 50-liter carboy partially filled with reagent water. Dilute to 50 L with reagent water. Mix carefully. Record the acid lot numbers and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.6 **Stannous Chloride Solution, Hg grade, 10% (w/v) per manufacturer's (CETAC) instructions**

7.6.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.6.2 Add 200 g of $SnCl_2$ to the flask.

7.6.3 Add deionized water until the total weight is 2000 g.

7.6.4 Place the jar in a fume hood and slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.5 Close the jar and agitate until the reagent is dissolved (prepare every 2 weeks).

7.7 **Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):**

7.7.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.7.2 Add 240 g of NaCl and 240 g of hydroxylamine hydrochloride (Hg grade) to the jar.

7.7.3 Add deionized water until the total weight is 2480 g.

7.7.4 Close the jar and agitate until the reagent is dissolved. (prepare

annually).

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate ($KMnO_4$), 5% solution (w/v):

7.8.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.8.2 Add 100 g of $KMnO_4$ (Hg grade) to the jar.

7.8.3 Add deionized water until the total weight is 2100 g.

7.8.4 Close the jar and agitate until the reagent is dissolved (prepare annually).

7.9 Potassium persulfate ($K_2S_2O_8$), 5% solution (w/v):

7.9.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.9.2 Add 100 g of $K_2S_2O_8$ (Hg grade) to the jar.

7.9.3 Add deionized water until the total weight is 2100 g.

7.9.4 Close the jar and agitate until the reagent is dissolved (prepare annually).

7.10 Purchased Mercury Stock Solutions

7.10.1 Primary Mercury Calibration Standard Solution, 1,000 mg/L

7.10.2 Second-source Mercury Standard (Hg ICV Stock), 1000 mg/L. This standard is obtained from a different vendor than the Primary Mercury Calibration Standard.

7.11 Calibration Working Standard Solution (Hg Month Spike), 10 mg/L.

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.11.2 Pipet 1.00 mL of the 1000 mg/L primary mercury calibration standard solution (see Section 7.10.1) into the flask.

7.11.3 Dilute to the mark on the flask with Reagent Blank.

7.11.4 Stopper the flask and shake to mix.

7.11.5 Transfer the solution to a 125 mL Nalgene bottle.

7.11.6 Document the preparation of the solution in the Reagent Module in TALS.

7.11.7 Prepare this solution fresh monthly or more often if necessary.

7.12 Daily Calibration Working Solution (Hg Daily Spk), 100 µg/L

7.12.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.12.2 Pipet 1.00 mL of the 10 mg/L Calibration Working Standard solution (see Section 7.11) into the flask.

7.12.3 Dilute to the mark on the flask with the Reagent Blank solution (final volume of 100.0 mL).

7.12.4 Stopper the flask and shake to mix.

7.12.5 Transfer the solution to a 125 mL Nalgene bottle.

7.12.6 Document the preparation of the solution in the Reagent Module in TALS.

7.13 Daily Initial Calibration (ICAL) Standards.

7.13.1 To each of seven 50 mL digestion tubes, add approximately 30 mL of the Reagent Blank solution.

7.13.2 For each calibration level, add the appropriate amount of Daily Calibration Working Solution to the tube as indicated in the following table. The final concentration for each calibration level is listed in the following table:

Daily ICAL Standards

Calibration Level	Volume of Daily Calibration Working Solution (100 µg/L) Added (mL)	Final Hg Concentration (µg/L)
1 (Hg STD1 0.1)	0.03	0.06
2 (Hg STD2 0.2)	0.06	0.12
3 (Hg STD3 0.5)	0.15	0.3
4 (Hg STD4 1.0)	0.3	0.6
5 (Hg STD5 2.0)	0.6	1.2
6 (Hg STD6 5.0)	1.5	3.0
7 (Hg STD7 10.0)	3.0	6.0

7.13.3 Close each tube and swirl to mix.

7.13.4 Prepare the calibration standards as samples.

7.13.5 Document the preparation of the solutions in the Reagent Module in TALS.

7.13.6 Prepare the calibration solutions each day prior to calibration.

7.13.7 The calibration blank is titled STD0 in TALS.

7.14 Continuing Calibration Verification Standard (Hg H20 CCV), 3.0 µg/L.

7.14.1 The CCV is prepared exactly as the 3.0 µg/L calibration standard, and from the same source. Refer to Section 7.13.

7.14.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.15 Second-Source Initial Calibration Verification Intermediate Standard (Hg Biwk ICV), 400 µg/L.

Add 0.04 mL of the 1000 mg/L ICV stock standard (see Section 7.10.2) to a 100 mL volumetric flask partially filled with the Reagent Blank solution and dilute to the mark. Record this information in the Reagent Module in TALS. This standard is made biweekly.

7.16 Second-Source Initial Calibration Verification Daily Working Standard (Hg H20 ICV), 2.4 µg /L.

Add 0.3 mL of the 400 µg/L ICV intermediate standard (see Section 7.15) to a 50 mL digestion tube filled with 30 mL of Reagent Blank. Prepare as a sample. Record this information in the Reagent Module in TALS.

7.17 Laboratory Control Sample (LCS), 3 µg/L

The LCS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Standard (Section 7.12) to 30 mL of Reagent Blank in a digestion tube. The LCS goes through the same digestion process as the samples.

7.18 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 3 µg/L

7.18.1 The MS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution (Section 7.12) to a digestion tube containing a second 30-mL aliquot of the selected sample.

7.18.2 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

7.18.3 The MS and MSD go through the same digestion process as the samples.

7.19 Reporting Limit (CRA) Check Standard (Hg H20 RL), 0.12 µg/L

The 0.12 µg/L calibration standard is analyzed as a sample to verify the reporting limit. Denoted as CRA in the run sequence.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	HDPE or glass	50 mL	HNO_3, pH < 2	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. See Table 1 for a summary of these requirements. This procedure meets all criteria for DoD QSM 5.0 or 5.1 unless otherwise stated. Any deviations or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC

9.2.1 Preparation Batch

A group of no more than 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.2.2 Method Blank (MB)

The method blank consists of Reagent Blank containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than ½ the reporting limit or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

For DoD V5 the method blank is controlled to < ½ LOQ or 10% of the amount measured in any sample or 10% of the regulatory limit, whichever is greater.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.2.3 Laboratory Control Sample (LCS)

The LCS is a blank to which a known concentration of the target analyte has been added. At least one aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Maximum control limits for LCS recoveries for Method 7470A are 80-120%. In-house control

limits based on three standard deviations of the mean of past results are used as long as they are at least as tight as the limits in the methods (see TestAmerica Denver Policy DV-QA-003P for further details on establishing control limits).

For DoD V5.0 or V5.1 the QSM Appendix C limits are required.

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.2.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a second aliquot of a selected field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked exactly as the MS) prepared and analyzed along with the sample and matrix spike. One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported for project-related samples only. Spiking levels are provided in Attachment 1. When the MS/MSD concentration is above the linear range; the MS/MSD and parent sample **MUST** be re-analyzed at a dilution. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM 5.0 or 5.1 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the

established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.2.5 Serial Dilution

Some programs require that a fivefold (1+4) dilution must be included in each analytical batch for each sample matrix.

Acceptance Criteria: The results must be within 10% of the expected value, assuming that the initial sample concentration is at least 25x the MDL concentration (or 50x the LOQ for DoD).

Corrective Action: If the control limit is not met, all associated sample results must be qualified and the failure addressed in the narrative.

9.2.6 Post-Digestion Spike

Some programs require the inclusion of a post-digestion spike in each analytical batch. The post-digestion spike is prepared by adding 0.3 mL of the 100 µg/L Daily Calibration Working Solution to 10 mL of sample digestate. Post-digestion spikes are performed as an additional check for matrix interference.

Acceptance Criteria: The percent recovery limits for the post-digestion

spike are 80 to 120%.

Corrective Action: If the acceptance criteria are not met, all associated sample results must be qualified.

9.2.7 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 3)

9.3 Instrument QC

9.3.1 Initial Calibration (ICAL)

9.3.1.1 Detailed information regarding calibration models and calculations can be found in Corporate Policy CA-Q-S-005, *Calibration Curves & Selection of Calibration Points*.

9.3.1.2 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. All calibration standards and calibration QC samples will be recorded in prep batches and prepared as samples. The instrument calibration date and time must be included in the raw data.

9.3.1.3 Calibrate using seven standards and a blank. The concentration levels are listed in Attachment 1.

NOTE: It is generally not acceptable to reject calibration points for this method.

9.3.1.4 The calibration curve must have a correlation coefficient of $\geq$ 0.995 for an unweighted linear regression or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

9.3.1.5 Record the microabsorbance for the 10 ppb standard in the instrument maintenance logbook.

9.3.2 Initial and Continuing Calibration Blank (ICB/CCB)

9.3.2.1 An initial calibration blank is tested immediately after the daily ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Client specific requirements take precedence. For example, DoD QSM 5.0 or 5.1 requires control of blanks to a

concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.3.2.2 Continuing calibration blanks are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than ½ the reporting limit. Some programs require that blanks be less than 2x the MDL or less than the LOD (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.3.3 Initial Calibration Verification (ICV), 2.4 µg/L

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV result must be within 10% of the true value.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

9.3.4 Reporting Limit Check Standard (CRA), 0.12 µg/L

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value. For DoDV5.1 the recovery is within 20%

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, and the instrument

recalibrated.

9.3.5 Continuing Calibration Verification (CCV), 3.0 µg/L

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run. The CCV must be a mid-range standard at a concentration other than that of the ICV.

Acceptance Criteria: The CCV result must fall within 20% of the true value.

For DoD V5 the CCV result must be within 10%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be re-analyzed once without modification to the instrument's operating conditions. If the re-analyzed CCV is found to be in control, the CCV analysis must be repeated a second time with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

9.3.6 Linear Range

TAL Denver does not report values greater than the highest standard (10 µg/L) used for calibration. Any sample concentration greater than 90% of the highest standard will be diluted. The calibration curve is validated by running 3 check standards, 0.12 µg/L (CRA) , 3 ug/L (CCV), and 2.4 µg/L (ICV), during the analytical run. No further linear range study is warranted.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

NOTE: Record the IDs of all pipettes and thermometers in the TALS batch record.

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure.

10.3.2 Transfer 30.0 mL of well mixed sample to a clean sample digestion tube. The calibration standards may be prepared in duplicate to ensure sufficient volume to complete the analytical sequence. Additional aliquots of CCV and CCB solution may have to be prepared for larger sample runs to ensure that CCV and CCB samples bracket every 10 samples in the analytical sequence.

10.3.3 Prepare an MB, LCS, MS, and MSD for each batch.

10.3.3.1 The MB consists of 30.0 mL of Reagent Blank.

10.3.3.2 The LCS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to 30 mL of Reagent Blank in a digestion tube.

10.3.3.3 The MS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second 30-mL aliquot of the selected sample.

10.3.3.4 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

10.3.4 Add 1.5 mL of concentrated H_2SO_4 and 0.75 mL of concentrated HNO_3 to the samples in the digestion tubes, mixing after each addition.

10.3.5 Add 4.5 mL of 5% potassium permanganate solution to each sample. For samples high in organic materials or chlorides, dilute the sample until the purple color persists for at least 15 minutes.

10.3.6 Add 2.4 mL of potassium persulfate solution, cap the vial, and heat for two hours at 90 - 95°C. Record the start and stop times and the initial and final temperatures on the bench sheet. Verify that a purple color persists or a black precipitate is present after the two hours of heating. If this is not true, repeat the digestion using a smaller aliquot of sample.

NOTE: Record both the observed and corrected temperatures on the bench sheet.

10.3.7 Allow the samples and standards to cool at room temperature.

10.4 Calibration

10.4.1 All calibration standards are digested together with samples, as described in Section 10.3, prior to analysis.

10.4.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration by starting the gas, lamp, heater, and sample pump (approximately 30 minutes of warm-up is required).

10.4.3 The mercury analyzer method uses external standard calibration. Use of an internal standard for this method is not appropriate.

10.5 Sample Analysis

NOTE: Because of differences between various makes and models of CVAA instrumentation, detailed push-button operating instructions are not provided here. Refer to the specific instrument-operating manual for detailed autosampler setup and operation protocols.

NOTE: The injection of samples and the addition of stannous chloride are done automatically by the instrument. Refer to the specific instrument manual for details.

10.5.1 When ready to begin analysis, add 1.8 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains).

10.5.2 Add additional Reagent Blank to the samples, QC samples and calibration standards to bring the final volume of each sample to 50 mL.

10.5.3 Aliquot each sample and calibration standard into a disposable test tube for analysis.

10.5.4 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples that are within 10% of the highest calibration standard.

10.5.5 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be diluted and reanalyzed.

10.5.6 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.5.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB.

10.5.8 The following analytical sequence must be used for Method 7470A. Refer to Quality Control Section 9.0 and Attachment 2 for quality control

criteria to apply to Method 7470A.

Instrument Calibration
ICV
ICB
CRA
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete the run.
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.5.9 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- **Recovery of the analyte in the matrix spike is <50%;**
- **The concentration of the analyte does not exceed the regulatory level; and**
- **The concentration of the analyte is within 20% of the regulatory level.**
- **The reporting and matrix spike levels for TCLP analyses are detailed in Attachment 1. Attachment 3 provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.**

10.5.10 To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

10.5.11 See Attachment 5 for guidelines for minimizing contamination of samples and standards. See Attachments 4 and 6 for guidance on troubleshooting and preventive maintenance.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves & Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

11.2 Accuracy

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spike concentration}} \times 100$$

11.3 Precision (RPD)

$$\textbf{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.4 Concentration = Hg concentration (μg/L) = $\frac{C \times V_1 \times D}{V_2}$

Where:

C = Concentration (μg/L) from instrument readout

D = Instrument dilution factor

V_1 = Final volume in liters after sample preparation

V_2 = Initial volume of sample digested in liters

11.5 Appropriate factors must be applied to sample values if dilutions are performed.

11.6 Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy (DV-QA-004P).

11.7 Documentation and Record Management

11.7.1 All sample data is uploaded to TALS. All sample preparation and analytical batch information, including the batch number(s), list of samples, preparation analyst and date, instrument analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes) is recorded in TALS.

11.7.2 Raw data is scanned or saved directly as a PDF and is attached to the analytical batch in TALS.

11.7.3 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

11.7.4 If dilutions are required due to insufficient sample, interferences, or other problems, the reporting limit and MDL are multiplied by the dilution factor and the data may require flagging.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Aqueous Acidic (Metals) - Corrosive - Waste Stream J

14.2.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

15.3 U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

15.4 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 5.0, 7/2013

16.0 Method Modifications:

Item	Method	Modification
1	EPA 7470A	Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
2	EPA 7470A	This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."
3	EPA 7470A	Methods 7470A and 7471A state that working mercury standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory has developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • **Successful proficiency testing PT results for samples that were prepared and analyzed within 24 hours, but on successive days (e.g., ERA WP-66);** • **Successful analysis of true NIST mercury standards within every analytical batch; and** • **A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."**
4	EPA 7470A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above ½ the reporting limit.

17.0 Attachments

Attachment 1: Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels
Attachment 2: Summary of Quality Control Requirements
Attachment 3: MSA Guidance
Attachment 4: Troubleshooting Guide
Attachment 5: Contamination Control Guidelines
Attachment 6: Preventative Maintenance
Table 1: DOD QSM 5.0/5.1 QC Criteria for CVAA/Mercury

18.0 Revision History

- Revision 9 dated September 19, 2019
  - Annual review

- Revision 8 dated August 31, 2018
  - Annual review
  - Removed vendor from Section 7.10.1
  - Updated standard concentration in Section 7.10.2
  - Updated standard concentration and spiking amount in Section 7.15.

- Revision 7 dated August 31, 2018
  - Annual review
  - Removed reference to use of an auto diluter in sections 6.1.2 and 10.5.4.
  - Added control criteria for DoDV5.1 in Section 9.3.4 for the CRA.
  - Updated Attachment 2 for DoDV5.1 CRA and DoDV5 CCV control limits.

- Revision 6 dated August 31, 2017
  - Annual review
  - Added reference to QSM 5.1 throughout the SOP
  - Added support equipment ID documentation requirement to section 6.0
  - Clarified batch definition to “no more than 20 samples” to section 9.2.1
  - Added reference to LCSD requirement to sections 9.2.3 and 9.2.4
  - Added current section 12.2 LOQV information

- Revision 5 dated August 31, 2016
  - Annual review
  - Removed all references to AFCEE
  - Added comment to section 7.6 to prepare Stannous Chloride Solution every two weeks
  - Added comment to sections 7.7 Sodium chloride-hydroxylamine hydrochloride solution, 7.8 Potassium permanganate, and 7.9 Potassium persulfate to prepare annually
  - Updated section 9.2.4 MS/MSD acceptance criteria and corrective actions
  - Added note to 10.3 to document IDs of pipettes and thermometers
  - Added note to 10.3.6 to record observed and corrected temperatures
  - Updated a three subsection of section 12 to reflect current practices
  - Update Attachment 2 for ICB/CCB for DoD to <LOD
  - Removed revision histories for 2010 and earlier (available upon request)

- Revision 4 dated July 31, 2015
  - Annual review
  - Added 0.1 standard to Section 7.13.2
  - Updated MB control limit to ½ LOQ for DoD
  - Removed 20% DoD control for CRI from Section 9.3.4
  - Added DoD V5 spiking requirement for MDLV to Section 12.1
  - Updated Section 12.2 DOC to use LCS’s
  - Added 0.1 standard to Attachment 1
  - Added TALS reference to Section 2.0
  - Added instructions to Section 7.5 regarding making reagent blank

  - Added TALS reagent IDs
  - Change LIMS to TALS throughout
  - Added Linear range section 9.3.6
  - Corrected Section 11.7 to match current practice
  - Removed figures one and two
  - Revised reagent Sections 7.6-7.9 to match current practice
  - Changed Sections 7.13-7.19 to reflect new procedure
  - Updated procedures in Section 10 to better meet traceability requirements
- Revision 3 dated July 31, 2014
  - Annual review
  - Added DoD V5 requirements to Sections 9.2.2, 9.2,3, 9.2.4, 9.3.2.1 and 9.3.5.
  - Added table 1 - DOD QSM 5.0 QC Criteria for CVAA/Mercury

- Revision 2 dated July 15, 2013
  - Annual review
  - Changes section 7.6 and 12 to reflect current practices
  - Remove reference to Standards Log to Reagent Module in the LIMS in section 7.11.6, 7.12.6, 7.13.4, 7.15 & 7.16.
  - Changed “RL” to “CRA” in sections 7.19, 9.3.4, 10.5.8
  - Added CRA (RL Standard) to Attachment 2
  - Removed Attachment 3 and re-number subsequent attachments
  - Clarified first bullet point under 10.5.9
  - Corrected references date for section 15.2
  - Added Texas TRRP to section 12.1

- Revision 1.2 dated July 13, 2012
  - Updated Sections 7.6 and 7.7 to state Hg reagents are used
  - Updated Sections 9.3.2.1 and 9.3.2.2 to control calibration blanks to ½ RL
  - Added Section 9.3.1.5 to record the counts for the 10 ppb high standard
  - Updated Sections 10.5.2 to bring samples to a final volume of 45 mL with 1% HNO_3
  - Formatting and grammatical changes

- Revision 1.1 dated February 03, 2012
  - Annual technical review
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated Section 9.1.2 and Attachment 2 for Method Blank acceptance criteria
  - Added dilution note to Section 10.3.4
  - Updated section 12.0 to reflect current laboratory practice
  - Removed Leeman instrument and replaced Nitrogen with Argon for Attachment 7

- Revision 1.0 dated 23 August 2011
  - Updated Section 7.15 ICV Intermediate Standard to 400ug/l
  - Updated Section 7.16 ICV Working Standard level to 4ug/l
  - Updated Section 9.2.3 ICV true value to 4ug/l
  - Updated Section 10.3.8 ICV and ICB run order

- Revision 0.5 dated 25 April 2011
  - Removed all references to the FIMS Analyzer
  - Sections 6.1 and 6.3 were updated to reflect the use of digestion blocks from water baths.

  - The reagent amounts were updated to reflect using a 30ml aliquot from 10ml.
  - Section 10.3.2 was updated to show a final volume of 40ml.

- Revision 0.4 dated 07 February 2011
  - Revised section 10 to reflect use of calibrated digestion tubes and calibration standard volumes
  - Revised supplies list
  - Revised section 6.2 to include reference to Master List of Documents, Software and Hardware
  - Added section 11.1 to reference corporate SOP CA-Q-S-005 “Calibration Curves”

Earlier revision histories have been archived and are available upon request.

Attachment 1

Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels (µg/L)

	Value at Instrument	Final Value
Standard Aqueous RL	0.12	0.2
Std 0	0	0
Std 1	0.06	0.1
Std 2	0.12	0.2
Std 3	0.3	0.5
Std 4	0.6	1.0
Std 5	1.5	2.0
Std 6	3.0	5.0
Std 7	6.0	10.0
ICV	2.4	4.0
CCV	3.0	5.0
LCS	3.0	5.0
Aqueous MS	3.0	5.0

Attachment 2

Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run.	90 - 110% recovery	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.3.3).
RL Check Standard (CRA)	Following the ICV	50-150% recovery, 80-120% for DoDV5.1	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICB	Beginning of every analytical run, immediately following the ICAL.	Absolute value must be < ½ RL, <LOD for DoD	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.3.2).
CCV	Every 10 samples and at the end of the run.	80 - 120% recovery, 90-110 for DoDV5	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.3.5).
CCB	Immediately following each CCV.	Absolute value must be < ½ RL, <LOD for DoD	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.3.2).
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ½ RL Sample results greater than 10x the blank concentration are acceptable.	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.2.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 80-120% recovery.	Terminate analysis; Correct the problem; Re-digest and reanalyze all samples associated with the LCS (see Section 9.2.3).
Matrix Spike	One per 10 samples preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 75-125% recovery	In the absence of client-specific requirements, flag the data (see Section 9.2.4).
Matrix Spike Duplicate	See Matrix Spike	In-house 3 standard deviation control limits, not to exceed 20% RPD	See Corrective Action for Matrix Spike.

Attachment 3

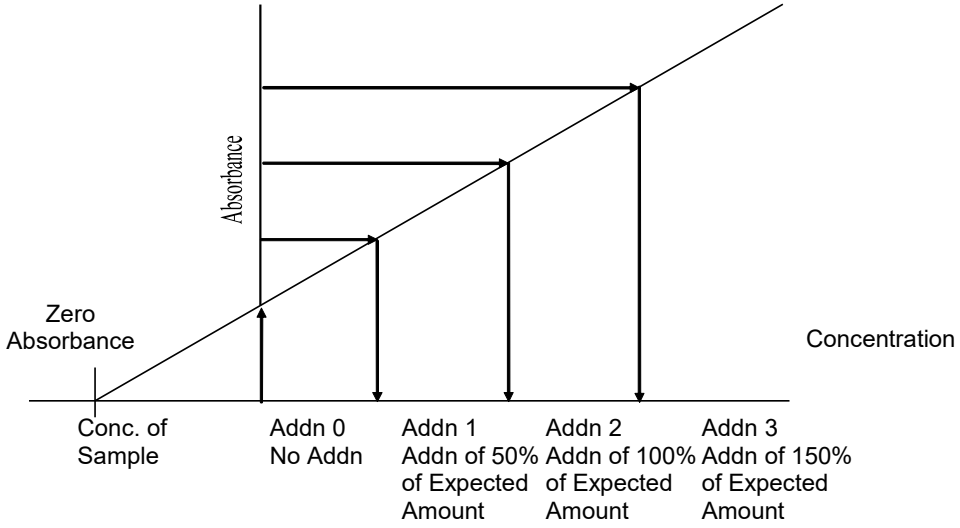
MSA Guidance

Method of Standard Addition (MSA)

Four equal volume aliquots of sample are measured and known amounts of standards are added to three of the aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration, and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of an analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. The absorbance (or response) is plotted on the vertical axis versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. The correlation coefficient (r) and the x-intercept (where y=0) of the curve are calculated. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 4

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

Attachment 5

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered gloves should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- If an unusually high sample is analyzed, segregate the glassware and soak with sulfuric acid prior to routine cleaning.

Attachment 6

Preventative Maintenance

A maintenance logbook is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time, and instrument number; describe the problem; and explain the corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational:

Cold Vapor Atomic Absorption (CETAC Analyzers)

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

Table 1
DOD QSM 5.0 & 5.1 QC Criteria for CVAA/Mercury

QSM 5.0 5.1 Table 7. Inorganic Analysis by CVAA/Mercury	
Requirement	**DoD QSM 5.0, QSM 5.1 and DOE QSAS 3.0**
Initial Calibration (ICAL)	Measure a minimum of 5 standards and a calibration blank daily and $r^2 \geq 0.99$ No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Run second-source standard once after each ICAL and prior to sample analysis. Must be within ± 10% of expected value. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.
Continuing Calibration Verification (CCV)	Run CCV after every 10 samples, and at the end of the analysis sequence: Reported analyte within ± 10% of expected value If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV. Or Immediately (within one hour) analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analytes(s) in all samples since the last acceptable CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank	Analyze calibration blank before analyzing samples, after every 10 field samples, and at the end of the analysis sequence. No analytes detected > LOD. Correct any problems, then re-prep and reanalyze the calibration blank and analyze all samples since the last acceptable calibration blank. Failures due to carryover may not require an ICAL.
Method Blank	One per prep batch. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater If criteria not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed.

Table 1
DOD QSM 5.0 and 5.1 QC Criteria for CVAA/Mercury
(Continued)

QSM 5.0 and 5.1Table 7. Inorganic Analysis by CVAA/Mercury	
DoD QSM 5.0, QSM 5.1 and DOE QSAS 3.0	
LCS	One per prep batch. Recovery must meet DoD QSM limits. If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.
Matrix Spike (MS)	One MS per prep batch. Use DoD acceptance criteria for LCS. If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be ≤ 20%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. If acceptance criteria are not met, apply J-flag. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

ICP Analysis for Trace Elements by SW-846 Method 6010C/D

Approvals (Signature/Date):

Doug Gomer 7/31/18
Doug Gomer Date
Technical Specialist

Doug Gomer 7/31/18
Doug Gomer Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 7/31/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 7/31/18
Richard Clinkscales Date
Laboratory Director

Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICPAES). This procedure references Methods 6010C and 6010D for hazardous waste (RCRA) testing.

1.2 The elements that can be determined by this procedure are listed in Attachment 1, together with the routine reporting limits. Additional elements may be analyzed under Method 6010C and 6010D provided that the method performance criteria presented in Section 12.0 are met.

1.3 The laboratory digests all water samples according to SOP DV-IP-0010.

1.4 Silver concentrations must be below 1.0 mg/L in aqueous sample digestates and 100 mg/kg in solid matrix sample digestates. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data. Samples with silver concentrations exceeding these levels must be re-prepared and reanalyzed using a smaller sample amount.

1.5 The digestion procedure for soil samples is described in SOP DV-IP-0015.

1.6 State or client specific requirements may take precedence over this SOP for water analyses. Review special instructions for each project before starting work.

2.0 Summary of Method

2.1 The laboratory uses simultaneous ICPAES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.

2.2 Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by a charge injection device (CID). The photo-currents from the charge injection device (CID) are processed and controlled by a computer system.

2.3 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

2.4 Refer to the appropriate SOPs for details on sample preparation methods: DV-IP-0010 for aqueous samples and DV-IP-0015 for soil samples.

3.0 Definitions

3.1 Dual View ICP – an ICP equipped with both radial and axial viewing capabilities.

3.2 Dissolved Metals - Those elements which pass through a 0.45-μm membrane. The sample is acidified after filtration.

3.3 Potentially Dissolved Metals - Potentially dissolved metals is the concentration of metals in solution after acidifying the sample with nitric acid to pH <2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-μm membrane filter. This definition is based on the Colorado surface water regulations.

3.4 Suspended Metals - Those elements which are retained by a 0.45-μm membrane.

3.5 Total Metals - The concentration determined on an unfiltered sample following vigorous digestion.

3.6 Total Recoverable Metals - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.7 Reporting Limit (RL) - The lowest concentration to which results are reported without qualification. Details concerning RLs are presented in Policy DV-QA-009P.

3.8 Reagent Water - Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

3.9 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by the following:

4.1.1 Overlap of a spectral line from another element.

4.1.2 Unresolved overlap of molecular band spectra.

4.1.3 Background contribution from continuous or recombination phenomena.

4.1.4 Stray light from the line emission of high concentration elements.

4.2 A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.3 Spectral Interferences

Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte signal. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections must be applied to the analyte to compensate for the effects of these unwanted emissions.

4.4 Physical Interferences

An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If internal standard recoveries are not acceptable (see Section 9.11), then dilution of the sample may be necessary to overcome the interferences.

4.5 Chemical Interferences

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed, can be minimized by buffering the sample, matrix matching, or standard addition procedures.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be

removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	Hazards	Exposure Limit[(2)]	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Thermo Fischer ICP 6500E Trace Analyzers are currently used.

Instruments with demonstrated equivalent performance can also be used

6.1.2 Radio Frequency Generator

6.1.3 Argon gas supply

6.1.4 Coolflow or appropriate water-cooling device.

6.1.5 Peristaltic Pump.

6.1.6 Autosampler.

6.2 Supplies

6.2.1 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.2 Class A volumetric flasks.

6.2.3 Autosampler tubes.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards used in calculations shall be entered into the TALS Reagent Module with all applicable information (e.g., components, concentrations, expiration, etc.).

7.2 Shelf-Life

7.2.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, a one-year expiration will be assigned by the laboratory.

7.2.2 The expiration date of intermediate concentration standards or working standards will be set at six months or less and cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.

7.2.3 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be replaced immediately.

7.3 Standards

7.3.1 Standards used for calibration and quality control purposes must be NIST traceable, where available. Multi-component custom blend standards must be verified against a second-source standard before they are first put into use (the only exception is standards purchased directly from NIST), as described in SOP DV-QA-0015. If the standard has been purchased previously it does not need to be verified, but the COA must be inspected to confirm that there have been no changes to the standard analyte levels.

7.3.2 Stock standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon, polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in the TestAmerica LIMS TALS.

7.3.3 Intermediate calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 4.4 of this procedure.

7.4 Reagent Blank / Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

Fill a 20-liter carboy with about 18 liters of reagent water. Slowly add 1 liter of concentrated nitric acid and 1 liter of concentrated hydrochloric acid. Adjust the total volume to 20 liters. Mix carefully. Record the acid lot number and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.5 Stock ICSA and ICSAB Standards

The following standards are purchased from commercial sources:

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
Icp stk ICSA	Fe Al, Ca, Mg	2,000 5,000
ANALYTES B	Ba, Be, Co, Cr, Cu, Mn, V Ag, Cd, Ni, Pb, Zn	50 100
ICP ISAB STD1	Li, Mo, Sb, Sr As, B, P Se K, Na	100 200 500 5,000

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
ICP ISAB STD2	Ti Sn	100 1,000
10000 Si	Si	10,000
10000 Th	Th	10,000
1000 Tl	Tl	1,000
1000 Zr	Zr	1,000
1000 S	S	1,000
1000 Bi	Bi	1,000

7.6 ICSA Working Standard (ICP ICSA)

A combined working ICSA standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSA and ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSA standard concentrations shown in Attachment 4.

7.7 ICSAB Working Standard

A combined working ICSAB standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
Icp stk ICSA	25
ANALYTES B	2.5
ICP ISAB STD1	2.5
ICP ISAB STD2	2.5
10000 Si	0.25
10000 Th	0.05
1000 Tl	2.5
1000 Zr	0.25
1000 S	0.25
1000 Bi	0.25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSAB standard concentrations shown in Attachment 4.

7.8 Calibration Check Standard (S1, S2)

The two calibration check standards are the same as the working ICAL standards (ICP ICAL1A and ICP ICAL2A) described in Section 7.12.

7.9 Laboratory Control Sample (LCS) Stock Standards

The LCS stock standards are purchased from commercial sources. The stocks are custom-made standards purchased at ready-to-use concentrations as follows:

LCS Stock Standards	Elements	Concentration (mg/L)
ICP SPK 3A	Ca, K, Mg, Na P Al, Ba, Bi, Se, Tl, U, As, Fe, Li, Sr, Th Co, Mn, Ni, Pb, V, Zn Cu Cr Cd Ag, Be	5,000 1,000 200 100 50 25 20 10 5
ICP SPK 2B	Sb, Zr B, Mo, Ti Sn Si (SiO_2) S	50 100 200 1,000 (2,140) 200

The soil and water LCSs are prepared according to the instructions in SOPs DV-IP-10 and DV-IP-0015. Final concentrations are shown in Attachment 2.

7.10 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

The same LCS stock standards described in Section 7.9 are also used to prepare matrix spikes and matrix spike duplicates. Final concentrations are shown in Attachment 2.

7.11 Post Digestion Spike (PDS) Standards (Analyte Addition Spike Standards)

The custom standards tabulated below are purchased from a commercial source. Add 0.06 mL of each to 6 mL (100X) of digestate or dilution of digestate.

PDS Stock	Elements	Conc. (mg/L)
ICP PDS 1	Ag, Be, Cd, Co, Cr, Cu, Mn, Ni, Sr, V Ba, Li, Pb, As, Se, Th, Tl, Zn U Al, Fe P Ca, K, Mg, Na,	5 5 10 20 50 100 200 2,000
ICP PDS 2	Mo, Ti, Zr B, Sb, Sn Si (SiO_2)	5 10 500 (1,070)

7.12 Initial Calibration (ICAL) Standards

7.12.1 Stock Calibration Standards

The following stock solutions are purchased from commercial sources.

Stock Standard	Elements	Conc. (mg/L)
Icp cal std 2	Mo, Ti, Zr Sn Si (SiO_2)	100 200 1,000 (2,140)
Icp cal std 3	Ag, Al, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Sr, V, Zn Li, P Fe Ca, Na Mg K	100 200 500 1,000 4,000 10,000
Al, Ca, Fe, Na, S, Th Stocks	Al, Ca, Fe, Na, S, Th	10,000
As, Pb, Sb, Se, Tl, U, Bi Stocks	As, Pb, Sb, Se, Tl, U, Bi	1,000

7.12.2 Working Initial Calibration Standard (ICP ICAL1A)

Add 10.0 mL each of Icp cal std 2 and Icp cal std 3 to a 1L volumetric flask partially filled with reagent blank solution. Add 2 mL of the As, Pb, Sb, Se, and Tl stocks. Dilute to the mark with reagent blank solution.

7.12.3 Working Initial Calibration Standard (ICP ICAL2A)

Add 10 mL of the Al and Fe and 50 mL of the Na 10,000 mg/L stock solutions; 1 mL of the Th and 20 mL of the U 1,000 mg/L stock solutions; 2 mL of the 1,000 mg/L Bi solution and 1 mL of the 10,000 mg/L S solution to a 1,000-mL volumetric flask partially filled with reagent blank and dilute to the mark with reagent blank.

7.13 Initial Calibration Verification (ICV)

7.13.1 ICV Stock Standards

The following stock solutions are purchased from commercial sources:

Stock Standard	Elements	Conc. (mg/L)
Icp ICVL A	Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn Se, Tl Ca, Na Mg K	25 50 200 1,000 2,000

Stock Standard	Elements	Conc. (mg/L)
Icp ICVL B	Ag, Mo, Sb, Ti, Zr Sn P, Si (SiO_2)	25 50 200 (428)
Icp ICVH	Al, Na Fe U Th	4,000 8,000 500 300
Bi, S Stocks	Bi, S	1,000

7.13.2 Working High Initial Calibration Verification (ICP ICVH)

Add 1.0 mL of the ICVH Stock, 0.05 ml Bi and 0.4 mL of the Sulfur to a 100 mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

Note: For Method 6010D the ICV working solutions must be prepared daily.

7.13.3 Working Initial Calibration Verification (ICP ICV)

Add 1.0 mL of each of the Icp ICVL A and Icp ICVL B stock solutions to a 100-mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

Note: For Method 6010D the ICV working solutions must be prepared daily.

7.14 Reporting Limit Standard

7.14.1 RL Stock Standards

The low level ICV/CCV verification stock standards are custom-made commercial standards used to make the working RL standard:

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
ICP LLCCV-1	K Na Ca, Mg Al, Bi, Fe U Ni Zn As, Cu, Se, Tl, Th Ba, Cr, Co, Li, Mn, Ag, Sr, V Pb Cd Be	300 100 20 10 6 4 2 1.5 1 1 0.9 0.5 0.1

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
ICP LLCCV-2	P Si B Sn Mo Zr Sb Ti	300 50 10 10 2 1.5 1 1
100 mg/L S	S	100

7.14.2 Reporting Limit Working Standard (ICP CRI)

RL Standard	Vol. of Stock Added (mL)
ICP-LLCCV-1	1
ICP-LLCCV-2	1
100 mg/L S	0.1

Adjust to volume (100 mL) using the reagent blank solution. The working RL standard must be prepared daily.

7.15 High Continuing Calibration Verification (ICP CCVH)

Perform a 2x dilution of the working ICP ICAL2A solution (Section 7.12.3) with reagent blank solution.

7.16 Continuing Calibration Verification (ICP CCV)

Perform a 2x dilution of the working ICP ICAL1A solution (Section 7.12.2) with reagent blank solution.

7.17 Low Level ICV/Low Level CCV (ICP LLCCV)

The low level ICV/CCV verification stock standards are custom-made commercial standards as follows:

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
ICP LLCCV-1	K	300
	Na	100
	Ca, Mg	20
	Al, Bi, Fe	10
	U	6
	Ni	4
	Zn	2
	As, Cu, Se, Tl, Th	1.5
	Ba, Cr, Co, Li, Mn, Ag, Sr,	1
	V	1
	Pb	0.9
	Cd	0.5
	Be	0.1
ICP LLCCV-2	P	300
	Si	50
	B	10
	Sn	10
	Mo	2
	Zr	1.5
	Sb	1
	Ti	1

7.17.1 Low Level ICV \ Low Level CCV, Working Standards

RL Standard	Vol. of Stock Added (mL)
ICP-LLCCV-1	1
ICP-LLCCV-2	1

Adjust to volume (100 mL) using the reagent blank solution.

7.18 Linear Range Verification Standard (LR)

The LRA standard is prepared from single element stock standards of each metal obtained from a commercial source. The stock standards are each purchased at a concentration of 1,000 mg/L except for Iron and Silicon which are each at a concentration of 10,000 mg/L. The LRA is prepared by taking the appropriate volume of each stock and adding it to a 500 mL volumetric flask partially filled with reagent blank and diluted to the mark after all elements have been added. The volume of each stock solution of each metal and volume used, along with final concentrations of each are listed in the following table.

Elements	Stock Conc. (mg/L)	Volume of stock (mL)	Final Conc. (mg/L)
Cd,	1,000	1.0	2
Co, Mo, Se, Tl	1,000	2.5	5
As, B, Cr, Cu, Mn, Ni, Pb, Sr, Ti, V, Zn	1,000	5.0	10
Ba	1,000	6.0	12
Fe	10,000	25	500
Si (SiO_2)	10,000	2.5	50 (107)

7.19 Reagents

7.19.1 Concentrated nitric acid (HNO_3), trace metals grade or better.

7.19.2 Concentrated hydrochloric acid (HCl), trace metals grade or better.

7.19.3 Reagent water must be produced by a Millipore DI system or equivalent, with a minimum resistivity of 1.0 Mohm/cm at 25 ˚C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation [1]	Holding Time [2]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2;	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool ≤ 6 °C [3]	180 Days	N/A

[1] Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required for most programs. Preservation must be verified prior to analysis.

[2] Inclusive of digestion and analysis.

[3] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot volume for both analyses must be refrigerated.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. DoD QSM 5.0 or 5.1 QC Acceptance Criteria for ICP analyses are presented in Attachment 11. The criteria must be met unless otherwise documented in the project documents.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.3 Method Blank

The blank is de-ionized water taken through the procedure as if it were a sample. For soil samples analyzed under the DoD QAPPs, the method blank consists of < 1 mm glass beads that have been processed in the same manner as the samples. A method blank is required with every batch of 20 or less samples.

Acceptance Criteria: The method blank must not contain any analyte of interest above ½ the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

9.4 Laboratory Control Sample (LCS)

The LCS is prepared as described in Section 7.9. One LCS is required with each analytical batch.

Acceptance Criteria: The recovery of the LCS must be within historical control limits. Historical control limits are based on three standard deviations of past results, and must be 80 - 120% or tighter. In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the report narrative. The process of establishing control limits is described in more detail in the Policy DV-QA-003P. The control limits are stored in TALS.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS/MSDs are prepared as described in Section 7.10. One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require the MS/MSDs to be run at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DoD QSM 5.0 or 5.1 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in

order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client’s needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

9.6 Method of Standard Additions (MSA)

9.6.1 This technique involves constructing a calibration curve in the sample matrix itself to compensate for any sample interferents that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. Attachment 8 provides more guidance on performing MSA analyses.

9.6.2 EPA Method 1311 (Section 8.4) requires that the MSA be used as the calibration method if the MS or MSD recoveries for TCLP extracts are less than 50% and the sample result is within 80 - 100% of the regulatory level. Attachment 4 provides a list of the regulatory limits. Although the MSA calibration technique may be used with only the sample and a single spiked point, Method 1311 specifies that three spiked points must be used along with the sample.

9.6.3 TALS does not currently have the capability to report results from an MSA-based analysis. If an MSA must be performed, the sample results must be calculated using the MSA spreadsheet (stored at R:\QA\Edit\Forms\Metals\MSA Worksheet - Water) and reported in an NCM. All of the associated samples must then be recalculated against the MSA spreadsheet. The completed spreadsheet must be saved and attached to the analytical batch in TALS along with the raw data.

9.6.4 A manual “N’ flag must also be added to all of the affected samples in TALS, indicating the presumptive evidence for the analyte. This flag

signals the Project Manager and indicates that narration of the result is required.

9.7 Serial Dilution Test

A dilution test is performed for each batch of samples. The purpose of this test is to ensure that neither positive nor negative interferences are biasing the analytical results. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

Acceptance Criteria: If the analyte concentration is sufficiently high (minimally, a factor of 50 times the MDL), an analysis of a 1:5 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within ± 10% of the original determination. For DoD QSM 5.0 or 5.1 the serial dilution is required if the MS or MSD fails and the parent concentration is greater than 50x the LOQ prior to dilution. 6010D requires the parent sample to be at least 25x higher than the RL to be calculable and sets the recovery limit at 20%.

Corrective Action: If the two results do not agree within the required limits, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and the failure is addressed in the case narrative to alert the client that a matrix affect may be present. For DoD QSM 5.0 or 5.1 a J-flag is added to the parent sample for the specific analyte if the acceptance criteria are not met.

9.8 Post Digestion Spike (PDS)

Whenever the MS/MSD recoveries are unacceptable, a PDS spike must be performed. The PDS spike is prepared as described in Section 7.10. Some programs require a PDS analysis whenever the serial dilution test fails. Other programs (e.g., DoD QSM 5.0 or 5.1) require a PDS to be included in every batch. Check project requirements. For programs where a PDS is required, the same sample that was used for the serial dilution test should be used for the PDS.

Acceptance Criteria: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80 - 120% for Method 6010C and 75 - 125% for 6010D.

Corrective Action: If the spike is not recovered within the specified limits, a matrix effect is confirmed. For DoD QSM 5.0 or 5.1 a J-flag is added to the parent sample if the sample concentration is less than 50x the LOQ prior to dilution. Any failures are flagged and should be described in the report case narrative.

9.9 Interference Check Analysis (ICSA / ICSAB)

The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Sections 7.5, 7.6, and 7.7 for the preparation of the ICSA and ICSAB solutions. Attachment 4 lists the final concentrations. All analytes are spiked into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run.

Acceptance Criteria: The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements must be ≤ 2x RL. The DoD and AFCEE programs have their own criteria based on the version used. For DoD QSM 5.0 or 5.1 the non-spiked analytes must be less than the absolute value of the LOD unless they are verified impurities. For 6010D the non-spiked analytes must be less than the absolute value of the RL.

Corrective action: If the ICSA results for the non-interfering elements do not meet these limits, the field sample data must be evaluated as follows: If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted. The sample data may also be accepted if the affected element was not required. If the interfering elements are not present in the field sample at a concentration which would result in an absolute value > 2x RL, then the field sample data can be accepted. If the interfering element is present in the field sample at a level which would result in a false analyte signal > 2x RL, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA. If the data do not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed.

9.10 Monitoring Internal Standard Results

Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

Acceptance Criteria: If the internal standard counts fall within ± 30% of the counts observed in the ICAL blank, then the data are acceptable.

Corrective Action: If the internal standard counts in the field samples are outside of the control limits, the field samples must be diluted and reanalyzed;

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0010 and DV-IP-0015).

10.4 Calibration

10.4.1 Instrument Start Up

Set up the instrument with the operating parameters recommended by the manufacturer. Complete any required preventative maintenance and record in the ICPAES Preventative Maintenance Log. Preventive maintenance recommendations are listed in the TestAmerica Denver Quality Assurance Manual. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4.2 Initial Calibration (ICAL)

The calibration curve is established on each day of operation using a blank and one standard. The preparation of the ICAL standards is described in Section 7. The final concentrations of the ICAL standards are presented in Attachment 6. The validity of the calibration curve is confirmed by analysis of the ICV, CCV, ICB, RL Check standard and Low Level ICV/CCV) which are run immediately after the ICAL. Some programs also require a high-level verification check (see Section 10.4.9).

10.4.3 Initial Calibration Verification (ICP ICVH and ICP ICV)

Calibration accuracy is verified using a second-source standard (ICP ICVH and ICP ICV) that is at or below a concentration near the mid-point of the working range. The ICV is analyzed immediately after the ICAL. The preparation of this standard is described in Section 7. The concentration of the ICV standard is presented in Attachment 6.

Acceptance Criteria: The ICV result must fall within 10% of the true value for that solution. The relative standard deviation must be < 5% (the laboratory is using at least two exposures for all ICP analyses).

Corrective Action: If the ICV fails to meet acceptance limits, the standard may be reanalyzed without modification to the instrument operating conditions. Two consecutive, acceptable analyses are required before the analytical run may continue. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.4 Mid Level Continuing Calibration Verification (CCV)

The preparation of the CCV solution is described in Section 7. The final concentrations of the CCVs are presented in Attachment 6. Note that the CCV is made at a different concentration than the ICV to meet NELAC requirements. CCVs are analyzed after the ICV, after every ten samples, and at the end of the analytical run.

Acceptance Criteria: The CCV must be within 10% of the expected value. The relative standard deviation must be < 5%.

Corrective Action: If the CCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue*. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed.

10.4.5 6010C - Low Level Initial Calibration (LLICV) and Continuing Calibration Verification (LLCCV)

The preparation of the LLCCV solution is described in Section 7. The low-level CCV needs to be analyzed at the beginning and end of every run sequence. If low level samples are expected then the low-level CCV should also be run every ten samples.

Acceptance Criteria: The LLCCV must be within +/-30% of the expected value to meet Method 6010C requirements.

Corrective Action: If the LLCCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue*. If the calibration cannot be verified within theses specified limits, the analysis of samples containing the affected analytes at similar concentrations cannot continue until the cause is determined and the LLCCV standard successfully analyzed. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed. TestAmerica will not hold samples with concentrations greater than 10x the reporting limit to the 30% acceptance criteria.

10.4.6 Initial Calibration Blank (ICB)

System cleanliness is verified by analyzing an ICB after the first CCV. The preparation of the ICB is described in Section 7.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD QSM 5.0 or 5.1 requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the ICB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.7 RL Calibration Check Standard (ICP CRI)

Calibration accuracy at the RL is verified by analyzing a standard prepared at a concentration at or below the laboratory's standard reporting limit. The preparation of this standard is described in Section 7. Alternate RL standard concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents Module in TALS.

Acceptance Criteria: For routine work the acceptance limits are ± 50% of the expected value. For **6010D and DoD QSM** the acceptance limits are ± 20%.

Corrective Action: If the RL Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.8 Lower Limit of Quantitation Check (LLQC)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits, quarterly and on an as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the LLICV/CCV is that this standard is carried through the entire preparation and analytical procedure. Prepare 7 aliquots spiked at the LLOQ.

Acceptance Criteria: LLQC is verified when all analytes are detected within ± 30% of their true value with an RSD ≤ 20%.

Corrective Action: If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.9 High-Level Calibration Check Standard

The method 6010C defines the linear working range used for daily analysis based on the LDR studies performed every six months, in which case this standard is not required. However, some programs require verification of the high end of the linear range at different frequencies. The DoD QSM 5.0 OR 5.1 requires that the linear range must be verified on a daily basis. For DoD QSM 5.0 or 5.1 and Method 6010D samples, the spike level of the highest standard analyzed defines the linear range for that day.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the High-Level Calibration Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analyst must run a standard at a lower concentration until the criteria is met for this calibration or the sample

results cannot exceed the level of the highest calibration standard.

10.4.10 Continuing Calibration Blank (CCB)

CCBs, prepared as in Section 7.4, are analyzed after each CCV.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD. Method 6010D sets the CCB upper limit at the RL.

Corrective Action: If the CCB is greater than these limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, instrument maintenance should be considered, the calibration re-verified, and all samples analyzed since the last successful CCB must be reanalyzed.

10.5 Sample Analysis

10.5.1 Replicate Readings

The laboratory averages the results from two exposures for Axial and Dual View ICP for each standard, field sample, and QC sample due to sample volume limitations of the autosampler tube.

10.5.2 Rinse Time between Samples

Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless, following the protocol outlined in 12.7, it can be demonstrated that a shorter rinse time may be used.

10.5.3 The following analytical sequence is used:

Instrument Calibration
High Standard Verification
ICV
LLICV (6010C only)
CCV
ICB
RL Verification Standard
LLQC (as needed)
ICSA
ICSAB
LRA
CCV

CCB
LLCCV (6010C only)
10 samples
CCV
CCB
LLCCV (6010C)
10 samples
CCV
CCB
LLCCV (6010C)
Repeat sequence with 10 samples between CCV/CCB pairs
CCV
CCB
LLCCV (6010C)

10.5.4 Full method-required QC must be available for each wavelength used in determining reported analyte results. Guidelines are provided in the appendices for minimizing contamination of samples and standards (Attachment 10) and troubleshooting (Attachment 9).

10.5.5 Dilutions for High Levels of Elements of Interest

For 6010, results must fall within the linear range. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. Dilutions must be prepared using the reagent blank solution to maintain the correct acid concentration.

10.5.6 6010D Mid-Run Recalibration

During the course of an analytical run, the instrument may be recalibrated to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of a CCV and CCB before any further samples may be analyzed.

10.5.7 Dilutions for High Levels of Interfering Elements

Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted until the interferent is at or below the working range. An NCM will be written in these instances.

10.6 Instrument Maintenance

See Section 20 in the QAM.

10.7 Troubleshooting

See Attachment 9.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves & Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 The procedure for performing the calculation of ferric iron is detailed in the Work Instruction WI-DV-0092, *Calculation Methods*.

11.3 ICV percent recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}} \right) \times 100\%$$

11.4 CCV percent recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}} \right) \times 100\%$$

11.5 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{SSR - SR}{SA} \right) \times 100\%$$

Where:

SSR	=	Spike Sample Result
SR	=	Sample Result
SA	=	Spike Added

The relative percent difference (RPD) of a matrix spike/matrix spike duplicate pair is calculated according to the following equation:

$$RPD = \left| \frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right| \times 100$$

Where:

MS	=	determined spiked sample concentration
MSD	=	determined matrix spike duplicate concentration

11.6 The final concentration for a digested aqueous sample is calculated as follows:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
$V1$	=	Final volume in liters after sample preparation
$V2$	=	Initial volume of sample digested in liters

11.7 The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
V	=	Final volume in liters after sample preparation
W	=	Weight in kg of wet sample digested
S	=	Percent solids/100

NOTE: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

11.8 The LCS percent recovery is calculated according to the following equation:

$$\%R = \left(\frac{\text{LCS Found Value}}{\text{LCS True Value}} \right) \times 100\%$$

11.9 The IEC's are calculated according to the following equation:

$$IEC = \left(\frac{\text{observed concentration}}{\text{observed concentration of the interfering element}} \right)$$

11.10 The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)

S = Dilution test result (Instrument reading × 5)

Appropriate factors must be applied to sample values if dilutions are performed.

11.11 Documentation and Record Management

11.11.1 All sample data is uploaded to TALS. All sample preparation and analytical batch information, including the batch number(s), list of samples, preparation analyst and date, instrument analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes) is recorded in TALS.

11.11.2 Raw data is scanned or saved directly as a PDF and is attached to the analytical batch in TALS.

11.12 Reporting

11.12.1 Reporting units are ug/L for water samples and mg/kg for solid samples.

11.12.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.12.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.12.4 All associated data are entered or uploaded into the LIMS as required.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.12.5 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process. Any manually transcribed data must be reviewed in its entirety by the second level data reviewer.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly. DoD QSM 5.0 or 5.1 requires the MDLV spike level to be 2 - 4 times the calculated MDL.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Instrument Detection Limit Study

12.3.1 Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each wavelength used for analysis.

12.3.2 Run seven blanks on three non-consecutive days.

12.3.3 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.3.4 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.3.5 For Method 6010D the IDL solutions:

- Should be prepared with each of the different matrices analyzed on the instrument;
- Should be prepared with 10 replicates for each matrix;
- Should have all the replicates for each matrix analyzed in a single day.

12.4 Linear Dynamic Range (LDR)

12.4.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

12.4.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.4.3 The LDR is determined by analyzing successively higher standard concentrations of the analyte. A minimum of three standards is required for the initial and on-going studies, and one of the levels must be close to the upper end of the range. The highest concentration must be within 10% of the stated concentration.

12.4.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions. Certain programs do not allow the use of LDRs for reporting purposes and instead require all sample results to fall below the highest daily standard analyzed.

12.4.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.5 Background Correction Points

12.5.1 To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength of interest and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations.

12.5.2 Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

12.6 Interelement Corrections (IECs)

12.6.1 ICP interelement correction (IEC) factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

12.6.2 When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs.

12.6.3 Refer to the facility-specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. Note that the USACE program requires a control limit of 2x |MDL|, which is feasible when verified MDLs are used.

12.6.4 To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element." Method 6010D requires that the IEC standards include Al, B, Ba, Ca, Cu, Fe, Mg, Mn, Mo, Na, Ni, Se, Si, Sn, V, and Zn.

12.6.5 Dual-View ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the CID detector instruments as reflected by the ICSA response.

12.7 Rinse Time Determination

12.7.1 Rinse times must be determined annually.

12.7.2 To determine the appropriate rinse time for a particular ICP system, a standard containing the highest concentration level that would be reported for samples is aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system.

12.7.3 For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level).

12.7.4 Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

12.7.5 The ICP instruments use an intelligent rinse program. The intelligent rinse lengthens the rinse time whenever a sample result for a known problem analyte is above a set concentration.

12.8 Demonstration of Capabilities

12.8.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

12.8.2 IDOCs and on-going proficiency demonstrations are conducted as follows: Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

12.8.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.9 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with federal, state, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Acid solutions from ICP drain - Waste Stream J

14.2.2 Metals waste potentially contaminated with Cat 1 radioactive materials – Waste Stream RJ

Note: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition and all promulgated updates, EPA Office of Solid Waste, through January 2008.

15.1.1 Method 6010C, Revision 3, Update IV, February 2007.

15.1.2 Method 6010D, Revision 4, Update V, July 2014.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6010C	This procedure uses mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.
2	EPA 6010C	The alternate run sequence presented in Section 10.5.3 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol

Item	Method	Modification
		requirements.
3	EPA 6010C	Method 6010 states that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific "concentration range around the calibration blank." Because of the lack of definition for "concentration range around the calibration blank," the laboratory has adopted the procedure in EPA CLP ILMO4.0 for determining IECs,
4	EPA 6010C	Section 9.9 of Method 6010C states: "If less than acceptable accuracy and precision data are generated, additional quality control tests are recommended prior to reporting concentration data for the elements in this method." The dilution test helps determine if a chemical or physical interference exists. Because the laboratory sometimes does not have prior knowledge if the MS/MSD will be within criteria, the analyst may select to perform a dilution test on one sample in each preparation batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. In this procedure, matrix interference is determined by evaluating data for the LCS, MS/MSD, and serial dilutions. The laboratory must request documented, clear guidance when an unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.0 Attachments

Attachment 1 Metals Analyzed by ICP and Reporting Limits
Attachment 2 Matrix Spike and Aqueous Laboratory Control Sample Levels
Attachment 3 Low Level ICV and CCV Spiking Levels
Attachment 4 Interference Check Sample Concentrations
Attachment 5 TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels
Attachment 6 6500 Initial Calibration &Continuing Calibration Verification Standards
Attachment 7 Summary of Quality Control Requirements
Attachment 8 MSA Guidance
Attachment 9 Troubleshooting Guide
Attachment 10 Contamination Controls
Attachment 11 DoD QSM 5.0 or 5.1 QC Acceptance Criteria

18.0 Revision History

Revision 7, dated 31 July 2018
- Annual review
- Added Sections 7.14.1 and 2 to use the ICVL/CCVL to make the RL Standard
- Added Silicon, Silica, and Boron to the list of elements in Attachment 6.

Revision 6, dated 31 July 2017
- Annual review
- Added reference to QSM 5.1 throughout SOP where applicable
- Updated spiking amounts in Section 7.12.2 to be based off 1L instead of 500ml

- Added LCSD requirement to Section 9.5 for DoD when not enough volume for MS/MSD
- Added Section 11.12 regarding reporting requirements
- Added current Section 12.2 regarding LOQVs

Revision 5, dated 31 July 2016
- Annual review
- Minor formatting and language corrections throughout
- Removed references to AFCEE and USACE
- Added Section 3.8 reagent water definition
- Added six-month expiration for intermediate standards in Section 7.2.2
- Added clarification to Section 7.3.2 regarding new standard verification
- Added new Section 7.18 explaining daily LR standard
- Removed cooling requirement for water samples in Section 8.0
- Added information to MS/MSD Section 9.5 to reflect current policy
- Changed Section 9.9 to Section 9.6, renumbered other sections accordingly
- Added information to Section 9.6 regarding the MSA requirement for TCLP extract samples
- Created subsections 9.6.1 - 9.6.4
- Added language to Section 10.4.9 to clarify daily linear range requirements
- Added Section 11.2 referencing the ferric iron work instruction
- Added requirement to review all manually transcribed data at second level review (Section 11.11.3)
- Archived pre-2011 revision histories

Revision 4, dated 31 December 2015
- Added requirements for Method 6010D to the SOP
- Minor grammar and formatting corrections throughout
- Added list of IEC test analytes to Section 12.5.4
- Added Section 10.5.6 regarding mid-run recalibration
- Added Section 12.2.5 defining 6010D IDL studies

Revision 3, dated 31 July 2015
- Annual review
- Updated Section 7.4 for how to make the 5% HNO_3/5% HCl solution
- Updated Section 12.2 for MDLV spike level to 2-4x MDL
- Updated Section 12.8.2 to use LCSs instead of ICVs for the DOC
- Reformatting throughout
- Removed reference to silica holding time
- Added Maintenance and troubleshooting sections
- Replaced Section 11.10 to match current practice
- Removed Section 12.2
- Removed Sections 1.3.1 and 1.3.2
- Added new Section 1.6
- Removed reference to glass beads in Section 6.2
- Corrected Reagent and Standard formulae throughout to agree with current practice

Revision 2, dated 31 July 31 2014
- Annual review
- Updated Section 6.1.3 to specify purity of argon gas
- Added statement to section 9.1.2 to reference DoD QSM 5.0 criteria in Attachment 11
- Removed references to preparation of oil/oily samples throughout the document as the

lab no longer supports this digestion method
- Added references for prep methods to section 15
- Added DOD QSM 5.0 QC acceptance criteria as Attachment 11

Revision 1, dated 15 July 2013
- Annual review
- Removed section 1.7
- Added section 3.8
- Corrected formatting
- Added section 11.12
- Removed Attachment 8, renumbered attachments and fixed references to attachments throughout the document

Revision 0.3, dated 13 July 2012
- Annual Review
- Clarified soil preservation for ICP only analysis, Section 8
- Updated section 9.1, 10.1, 10.2, and 12.1 to reflect current practice
- Updated sections 10.4.6 and 10.4.10 to control calibration blanks to ½ the RL

Revision 0.2, dated 30 June 2011
- Added reference to DV-IP-0017 “Microwave Digestion” throughout document
- Added section 6.3 “Computer Software and Hardware”
- Removed Uranium from the ICSA/ICSAB tables in sections 7.4, 7.5, and 7.6
- Updated sections 7.14 and 7.15 to reflect current practices
- Updated the Acceptance Criteria in sections 9.4, 9.6, and 9.10
- Referenced the TestAmerica Denver Quality Assurance Manual in section 10.4.1
- Updated section 11 to reference corporate SOP CA-Q-S-005, “Calibration Curves” and Arizona Calibration Training spreadsheet
- Added IEC calculation to section 11

Earlier revision histories have been archived and are available upon request.

Attachment 1

Metals Analyzed by ICP and Reporting Limits

ELEMENT	Symbol	CAS #	6010 Analyte	Reporting Limit (µg/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	100	10
Antimony[trace]	Sb	7440-36-0	X	10	1
Arsenic[trace]	As	7440-38-2	X	15	1
Barium	Ba	7440-39-3	X	10	1
Beryllium	Be	7440-41-7	X	1	0.1
Bismuth	Bi	7440-69-9		100	10
Boron	B	7440-42-8	X	100	10
Cadmium[trace]	Cd	7440-43-9	X	5	0.5
Calcium	Ca	7440-70-2	X	200	20
Chromium	Cr	7440-47-3	X	10	1
Cobalt	Co	7440-48-4	X	10	1
Copper	Cu	7440-50-8	X	15	2
Iron	Fe	7439-89-6	X	100	10
Lead[trace]	Pb	7439-92-1	X	9	0.8
Lithium	Li	7439-93-2	X	10	5
Magnesium	Mg	7439-95-4	X	200	20
Manganese	Mn	7439-96-5	X	10	1
Molybdenum	Mo	7439-98-7	X	20	2
Nickel	Ni	7440-02-0	X	40	4
Phosphorus	P	7723-14-0	X	3,000	300
Potassium	K	7440-09-7	X	3,000	300
Selenium[trace]	Se	7782-49-2	X	15	1.3
Silicon	Si	7631-86-9		500	50
Silver[trace]	Ag	7440-22-4	X	10	1
Sodium	Na	7440-23-5	X	1	100
Strontium	Sr	7440-24-6	X	10	1
Sulfur	S	7704-34-9	X	200	2
Thallium[trace]	Tl	7440-28-0	X	15	1.2
Thorium	Th	7440-29-1		15	15
Tin	Sn	7440-31-5	X	100	10
Titanium	Ti	7440-32-6	X	10	1
Uranium	U	7440-61-1		60	20
Vanadium	V	7440-62-2	X	10	2
Zinc	Zn	7440-66-6	X	20	2
Zirconium	Zr	7440-67-7		15	1

Attachment 2

Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (µg/L)	Matrix Spike Level (µg/L)
Aluminum	2,000	2,000
Antimony	500	500
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	50	50
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO_2)	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Sulfur	2,000	2,000
Thallium	2,000	2,000
Thorium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	500	500

Attachment 3

Low Level ICV/CCV

ELEMENT	LCS Level (µg/L)
Aluminum	100
Antimony	10
Arsenic	15
Barium	10
Beryllium	1
Bismuth	100
Boron	100
Cadmium	5
Calcium	200
Chromium	10
Cobalt	10
Copper	15
Iron	100
Lead	9
Lithium	10
Magnesium	200
Manganese	10
Molybdenum	20
Nickel	40
Phosphorous	3,000
Potassium	3,000
Selenium	15
Silicon	500
Si (as SiO_2)	1070
Silver	10
Sodium	1,000
Strontium	10
Thallium	15
Thorium	15
Tin	10
Titanium	10
Uranium	60
Vanadium	10
Zinc	20
Zirconium	15

Attachment 4

Interference Check Sample Concentrations

Element	ICSA (µg/L)	ICSAB (µg/L)
Aluminum	500,000	500,000
Antimony	-	1,000
Arsenic	-	2,000
Barium	-	500
Beryllium	-	500
Bismuth	-	1,000
Boron	-	2,000
Cadmium	-	1,000
Calcium	500,000	500,000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200,000	200,000
Lead	-	1,000
Lithium	-	1,000
Magnesium	500,000	500,000
Manganese	-	500
Molybdenum	-	1,000
Nickel	-	1,000
Phosphorous	-	2,000
Potassium	-	50,000
Selenium	-	5,000
Silicon	-	10,000
Silica	-	21,400
Silver	-	1,000
Sodium	-	50,000
Strontium	-	1,000
Sulfur	-	1,000
Thallium	-	10,000
Titanium	-	1,000
Vanadium	-	500

Attachment 4

Interference Check Sample Concentrations (cont'd)

Element	ICSA (µg/L)	ICSAB (µg/L)
Zinc	-	1,000
Tin	-	10,000
Thorium	-	10,000
Uranium	2,000	2,000
Zirconium	-	1,000

Attachment 5

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (µg/L)	Regulatory Limit (µg/L)	Spike Level (µg/L)
Arsenic	500	5000	4000
Barium	10000	100000	12000
Cadmium	100	1000	1100
Chromium	500	5000	5200
Lead	500	5000	5500
Selenium	250	1000	3000
Silver	500	5000	1050
Copper	100	N/A	2250
Zinc	200	N/A	2500

Attachment 6

6000 Dual View Calibration, ICV & CCV Standards

Element	Calibration Level	ICV (µg/L)	CCV (µg/L)
Aluminum Lo	1,000	250	500
Aluminum Hi	100,000	40,000	50,000
Antimony	2,000	250	1,000
Arsenic	2,000	250	1,000
Barium	1,000	250	500
Beryllium	1,000	250	500
Bismuth	2,000	500	1000
Boron	1,000	250	500
Cadmium	1,000	250	500
Calcium	10,000	2,000	5,000
Chromium	1,000	250	500
Cobalt	1,000	250	500
Copper	1,000	250	500
Iron Lo	5,000	250	2,500
Iron Hi	100,000	80,000	50,000
Lead	2,000	250	1000
Magnesium	40,000	10,000	20,000
Manganese	1,000	250	500
Molybdenum	1,000	250	500
Nickel	1,000	250	500
Phosphorous	2,000	2,000	1,000
Potassium	100,000	20,000	50,000
Selenium	2,000	500	1,000
Silicon	10,000	2,000	5,000
Si (as SiO2)	21,400	4,280	10,700
Silver	1,000	250	500
Sodium Lo	10,000	2000	5,000
Sodium Hi	500,000	40,000	250,000
Strontium	1,000	250	500
Sulfur	10,000	4,000	5,000
Thallium	2,000	500	1,000
Thorium	10,000	3,000	5,000
Tin	2,000	500	1,000
Vanadium	1,000	250	500
Uranium	20,000	5,000	10,000
Zinc	1,000	250	500
Zirconium	1,000	250	500

Attachment 7
Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90-110% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
RL Standard	At the beginning of the run	Results must within 50%	Terminate analysis; Correct the problem; Recalibrate.
LLICV/CCV	At the beginning of the run and after every 10 samples	Recovery must be within 30%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable LLCCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within +/- ½ RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within +/- ½ RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.10	See Section 9.10
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10
Dilution Test	One per prep batch.	For samples > 10x LOD (after dilution)' dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.

See Section 10.5.3 for run sequence to be followed.

Attachment 7

Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to ½ the RL. Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < ½ RL may not require redigestion or reanalysis (see Section 9.3)	Re-run once in a clean tube. If > ½ RL, re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.4)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	75 – 125% recovery or tighter in-house control limits.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	One per sample preparation batch of up to 20 samples. 10% frequency for some programs (see 9.5)	75 – 125 % recovery; RPD ≤ 20% or tighter in-house control limits.	See Corrective Action for Matrix Spike.

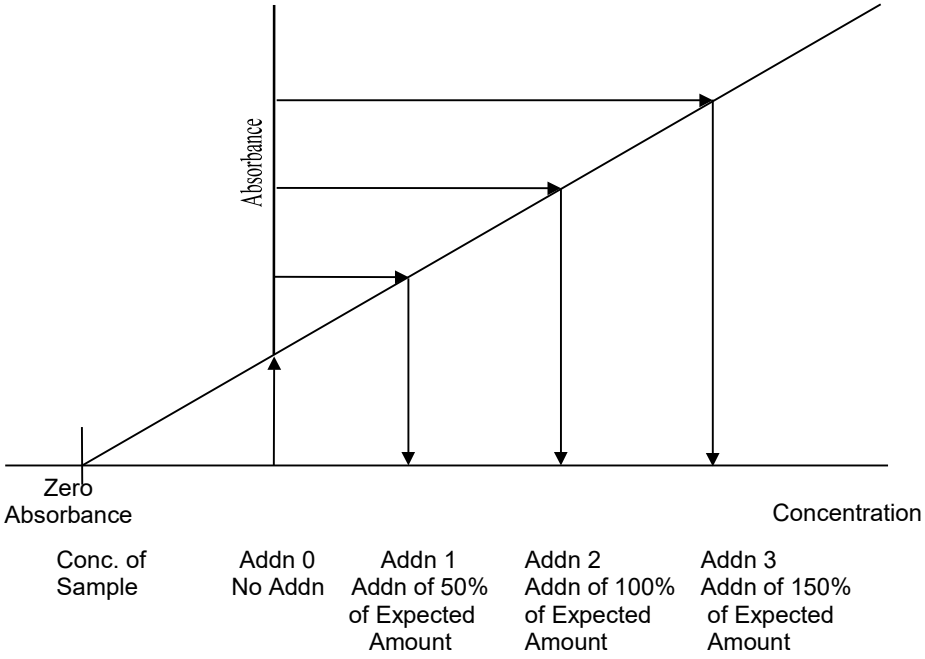
Attachment 8

MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the absolute value of the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear (r=0.995 or greater) over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 9

Troubleshooting Guide

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Replace RF generator
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

Attachment 10

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered gloves should not be used in the metals laboratory because the powder contains silica and zinc as well as other metallic analytes.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

Attachment 11
DoD QSM 5.0 OR 5.1 QC Criteria for Analysis by ICP

QSM 5.0 OR 5.1 Table 8. Inorganic Analysis by ICP	
Requirement	**DoD QSM 5.0 OR 5.1 and DOE QSAS 3.0**
Linear Dynamic Range (LDR) or high-level standard check	Run an LDR or high-level check standard at least once every 6 months. When calibrating with a single standard and a blank, the daily LDR standard must be analyzed at a concentration greater than any samples analyzed that day. Data cannot be reported above the high calibration range without an established/passing high-level check standard. Must be within ± 10% of expected value. Dilute samples within the calibration range or re-establish/verify the LDR.
Initial Calibration (ICAL)	Measure a minimum of one high standard and a calibration blank, daily. If more than one standard used, then $r^2 \geq 0.99$ ($r \geq 0.995$), otherwise no acceptance criteria. The ICAL must pass before running any samples. NOTE: The laboratory currently performs duplicate burns for the ICPAES method.
Initial Calibration Verification (ICV)	Run second-source standard once after each ICAL and prior to sample analysis. All reported analytes must be within ± 10% of expected value. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.
Continuing Calibration Verification (CCV)	Run CCV after every 10 field samples, and at the end of the analysis sequence. All reported values within ± 10% of expected value If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV. Or Immediately (within one hour) analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analytes(s) in all samples since the last acceptable CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-Level Calibration Check Standard (Low-level ICV)	Run low-level standard at a concentration ≤ LOQ daily after one-point ICAL. All reported analytes must be within ± 20% of expected value. Correct any problems, then reanalyze or repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.

Attachment 11
DoD QSM 5.0 OR 5.1 QC Criteria for Analysis by ICP
(continued)

QSM 5.0 OR 5.1 Table 8. Inorganic Analysis by ICP	
Requirement	**DoD QSM 5.0 OR 5.1 and DOE QSAS 3.0**
Initial and Continuing Calibration Blank (ICB.CCB)	Analyze calibration blank before analyzing samples, after every 10 field samples, and at the end of the analysis sequence. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. (13ICP) If not accepted by client, ICB/CCB must be <LOD. If criteria not met, correct problem If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed. Correct any problems and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCB failures due to carryover may not require an ICAL.
Interference Check Solution (ICS)	Run the ICS at the beginning of an analytical run (after ICAL and prior to sample analysis). ICS-A: Absolute value of concentration for all non-spiked analytes must be < LOD (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of expected value. (Note: ICS-AB not needed if instrument can read negative responses.) Correct any problems and reanalyze ICS. Do not analyze samples without a valid ICS. NOTE: TAL Denver has a letter from the ICSA standards manufacturer for many of the elements.
Method Blank	One per prep batch. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common lab contaminants not detected > LOQ. (2CLC) For ICP, common lab contaminants are: Al, Ca, Fe, K, Mg, Na, Si, Zn (Ba for TCLP) If criteria not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed.
LCS	One per prep batch. Recovery must meet DoD QSM limits. If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.

Attachment 11
DoD QSM 5.0 OR 5.1 QC Criteria for Analysis by ICP
(continued)

QSM 5.0 OR 5.1 Table 8. Inorganic Analysis by ICP	
Requirement	**DoD QSM 5.0 OR 5.1 and DOE QSAS 3.0**
Matrix Spike (MS)	One MS per prep batch. Use DoD acceptance criteria for LCS. If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be ≤ 20%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. If acceptance criteria are not met, apply J-flag. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
Dilution Test	One per prep batch if MS or MSD fails. Only applicable for samples with concentrations >50x LOQ (prior to dilution). For samples with lower concentrations perform PDS. Five-fold dilution must agree within ± 10% of the original result. Apply J-flag if acceptance criteria not met and explain in the case narrative.
Post-Digestion Spike (PDS) Addition	Perform Recovery Test when dilution test fails or analyte concentration in all samples is <50 x LOQ. Recovery must be within 80-120 % of expected result. If test fails, then run samples by MSA or apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 80 - 120%.
Method of Standard Additions	When dilution or post digestion spike fails and if required by the project. Document use of MSA in case narrative.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B

Approvals (Signature/Date):

Doug Gomer 10/31/18
Doug Gomer Date
Technical Specialist

Doug Gomer 10/31/18
Doug Gomer Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 10/31/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 10/31/18
Richard Clinkscales Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on EPA Method 6020A and 6020B.

1.2 Method 6020A and 6020B lists twenty-three elements approved for analysis by ICP-MS. The laboratory has implemented analysis by these methods for : Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mg, Mn, Ni, Se, Ag, Sr, Tl, Th, V, and Zn. Additional elements may be included provided that the method performance criteria presented in Sections 9 and 12 are met. Project approval may be required from the controlling agencies for compliance testing beyond the elements included in the promulgated methods and for those elements which may require state-specific accreditation.

1.3 The procedure is applicable to the analysis of acid digested waters, sediments, sludges and soils. Standard reporting limits are listed in Attachment 1 for water and soil. The preliminary acid digestion for aqueous samples is described in SOP DV-IP-0014 for Methods 3005A and 3020A and the digestion procedure for solids is given in SOP DV-IP-0015 for Method 3050B.

2.0 Summary of Method

2.1 Aqueous digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into a radiofrequency (RF) plasma. There the sample is decomposed and desolvated.

2.2 The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a mass resolution better than or equal to 0.9 amu (see Section 3) peak width at 10% of the peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

2.3 A collision/reaction cell utilizing He and (optionally) H_2 gases is used to remove molecular interferences. As the ion beam passes through the cell chamber, a diffuse cloud of He or H_2 gas is injected into its path. Collisions between the ions and the atoms in the gas deflect and remove interferences. See Section 4.2.3 for more information.

2.4 Interferences must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference corrections must include compensation for background ions contributed by the plasma gas, reagents, and the constituents of the sample matrix. Recommended elemental equations, which correct for many of these interferences, are listed in Attachment 2. Interference equations may vary or be unnecessary depending on the instrument setup and choice of collision/reaction gas.

2.5 Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices. Internal standard assignments are listed in Attachment 4.

3.0 Definitions

3.1 **Atomic Mass Unit (amu) –** Obsolete term replaced by “unified atomic mass unit (u)” or “dalton (Da)”, which denotes a small unit of mass that is used to express atomic and molecular masses. It is defined to be 1/12 of the mass of one atom of carbon-12.

3.2 **Batch –** The batch is a set of up to 20 samples of the same or similar matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. See Policy DV-QA-003P for further details.

3.3 **Dissolved Metals -** Those elements which pass through a 0.45-μm membrane filter (sample is acidified after filtration).

3.4 **Total Metals -** The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5 **Total Recoverable Metals -** The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6 **Instrument Detection Limit (IDL) -** See Section 12.3.

3.7 **Sensitivity -** The slope of the analytical curve (i.e., the functional relationship between raw instrument signal and the concentration).

3.8 **Tuning Solution -** This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9 **Initial Calibration Verification / Quality Control Standard (ICV) -** A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.10 **Continuing Calibration Verification (CCV) -** A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11 **Interference Check Standard (ICS) -** A solution containing both interfering and analyte elements of known concentration that is used to correction factors.

3.12 **Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB) -** A multi-element standard of known concentrations that is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13 **Reagent Blank -** High purity (> 18 megohm-cm) water containing the same acid matrix as the calibration standards that is carried through the entire digestion process.

3.14 **Calibration Blank -** High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples. Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15 **Method Detection Limit (MDL) -** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

3.16 **Low Level ICV (LLICV) / Continuing Calibration Verification (LLCCV) -** A multi-element standard of known concentrations prepared to monitor and verify the instrument performance at the reporting limit (RL).

3.17 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Elemental Isobaric Interferences

Elemental isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software and by the careful selection of isotopes for analysis.

4.2 Isobaric Molecular Interferences

4.2.1 Polyatomic interferences are derived from the plasma gas, reagents or sample matrix. Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Attachment 3 lists isobaric interferences which might possibly affect required analytes. These molecular interferences are minimized by use of the collision cell utilizing He and/or H_2 gases. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.2.2 Chloride in samples can produce low recoveries for antimony and silver. If chloride interference is a concern, 1% HCl can be added during digestion, but calibration standards must be adjusted to include 1% HCl also. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

4.2.3 Collision cell interference removal works both by causing the interfering molecular ion to dissociate and by reducing the kinetic energy of the ion. The latter is termed Kinetic Energy Discrimination (KED), and is the primary mechanism for interference removal. Polyatomic ions are larger than elemental ions and so collide with the helium atoms in the collision

cell more frequently than the smaller elemental ions. Each collision reduces the energy of the ion, so the molecular ions lose energy more quickly. At the end of the collision cell a positive voltage plate prevents passage of the now low energy molecular ions. Thus, the interference is eliminated because the molecular ions do not reach the detector.

4.3 Doubly Charged Ion Interferences

Doubly charged elemental ion interferences are possible in cases where the second ionization potential of the element is significantly below the first ionization potential for argon (15.7 eV). If a doubly charged ion is formed, it will cause a response at half of its elemental mass, potentially causing interference. Most elements have high enough second ionization potentials that formation of doubly charged ions is not an issue. The percentage of doubly charged ions being formed in the plasma is monitored on a daily basis during the instrument tuning process.

4.4 Physical Interferences

4.4.1 Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.4.2 Internal standards should be added at a level to give approximately 100,000 – 20,000,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 50 amu of the mass of the measured analyte.

4.4.3 Matrix effects are monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020A, the internal standard recoveries in samples can not fall below 70% of the intensity of the calibration standard. For method 6020B, the internal standard recoveries in samples cannot fall below 30% while the requirement for DoD is 30-120%. If they fall outside the applicable window, a five-fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample is reanalyzed.

4.4.4 Memory effects or carry-over can occur when there are large relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use.

It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The ICP-MS plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) capable of providing resolution less than or equal to 0.9 amu at 10% peak height and 1.0 amu at 5% peak height in the mass range from 6-253 with a data system that allows corrections for isobaric interferences and the application of the internal standard technique. The ICP-MS must be equipped with a collision cell for the removal of molecular interferences.

6.1.2 A four-channel peristaltic pump.

6.1.3 Autosampler with autosampler tubes.

6.1.4 Appropriate water cooling device.

6.2 Supplies

6.2.1 Argon gas: High purity grade (99.99%).

6.2.2 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.3 Class A volumetric flasks.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards must be entered into the TestAmerica LIMS (TALS) Reagent Module. Reagents that are not used for calculating results may either be recorded in the Reagent Module or may be entered into batch worksheets.

7.1 Storage and Shelf-Life

7.1.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Standards stored at concentrations as received from the vendor and mid-level dilutions must be replaced prior to the expiration date assigned by the vendor. If no expiration date is provided, the stocks and mid-level standards may be stored for up to one

year. They must be replaced sooner if verification from an independent source indicates a problem.

7.1.2 Working standards, i.e., all standards at concentrations ready to analyze on the ICP-MS (except tuning mixes, ICSA and ICSAB mixes, which are received at ready-to-use concentrations) are prepared fresh daily.

7.1.3 For more information on standard storage and shelf-life, see SOP DV-QA-0015.

7.2 Standards

Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in TALS.

7.2.1 Tuning Solution

The parent tuning solution is purchased as a custom multi-element mix. The elements and concentrations of the constituents are shown in Attachment 7. Prepare the working tuning Solution as detailed below.

7.2.1.1 Obtain a clean 1 L volumetric flask

7.2.1.2 Place 500 mL of reagent water and 10 mL of conc. HNO3 in the flask

7.2.1.3 Pipette 1mL of the Tuning Solution Stock into the flask

7.2.1.4 For the Agilent 7700 also add 50 µL of a 500 mg/L Mg solution and 30 µL of a 1000 mg/L Be solution.

7.2.1.5 Dilute to volume with reagent blank (See Section 7.3). Stopper and mix.

7.2.2 P/A factor solution

7.2.2.1 The Pulse/Analog (P/A) solution is used to monitor the correlation between the Pulse counting and Analog modes of the electron multiplier. The diluted solution must be prepared at different concentrations depending on the current instrument conditions. Multiple dilutions may be required to cover the required intensity range for all elements.

7.2.2.2 The P/A solution may be commercially purchased as a custom multi-element mix. See Attachment 7 for a list of the constituents and concentrations.

7.2.2.3 Prepare and use the P/A solution as recommended by the instrument manufacturer. The P/A solution should be analyzed daily.

7.2.3 Calibration Standard

Stock calibration standards are purchased as custom multi-element mixes or as single element solutions. Each day of analysis, the standards are diluted to working levels using reagent blank (see Section 7.3). The concentrations are given in Attachment 10. Prepare the Daily Working Calibration Standard as shown.

7.2.3.1 Daily Working Calibration Standard for Instruments 077 and 078 (ms 77 cal std)

7.2.3.1.1 Obtain a clean 100 mL volumetric flask.

7.2.3.1.2 Place 50 mL of reagent blank in the flask.

7.2.3.1.3 Pipette 0.5 mL of the MS CALSTD-1 stock standard into the flask.

7.2.3.1.4 Pipette 0.5 mL of the MS CALSTD-2 stock standard into the flask.

7.2.3.1.5 Pipette 0.5 mL of the MS CALSTD-3 stock standard into the flask.

7.2.3.1.6 Pipette 0.5 mL of the MS BRC CALSTD stock standard into the flask.

7.2.3.1.7 Pipette 0.5 mL of a 20 mg/L Zr standard into the flask.

7.2.3.1.8 Pipette 0.5 mL of a 20mg/L Sr standard into the flask.

7.2.3.1.9 Pipette 0.5 mL of a 200 mg/L Li standard into the flask.

7.2.3.1.10 Dilute to volume with reagent blank. Stopper and mix.

7.2.4 Initial Calibration Verification (ICV) Standard

The ICV stock is from a source different than the source for the calibration standards. Each day of analysis, the ICV standards are prepared new in reagent blank to the concentrations shown in Attachment 10. Prepare the ICV as shown below:

7.2.4.1 Initial Calibration Verification Standard for Instruments 077 and 078 (MS 77 ICV)

7.2.4.1.1 Obtain a clean 50 mL volumetric flask.

7.2.4.1.2 Place 25 mL of reagent blank in the flask.

7.2.4.1.3 Pipette 0.1 mL of the MS ICV StockA Standard into the flask.

7.2.4.1.4 Pipette 0.1 mL of the MS ICV StockB Standard into the flask.

7.2.4.1.5 Pipette 0.1 mL of the MS ICV Alt HP Standard into the flask.

7.2.4.1.6 Pipette 0.1 mL of the MS ICV BRC HP Standard into the flask.

7.2.4.1.7 Pipette 0.1 mL of a 20 ug/L Sr standard into the flask.

7.2.4.1.8 Pipette 0.1 mL of a 200 ug/L Li standard into the flask.

7.2.4.1.9 Dilute to volume with reagent blank. Stopper and mix.

7.2.5 Continuing Calibration Verification (CCV) Standard

The CCV is prepared from the same source as the calibration standards. The CCV standards are prepared fresh each day of analysis in reagent blank. The concentration is shown in Attachment 10.

7.2.5.1 Continuing Calibration Verification for Instruments 077 and 078 (ms 77 ccv)

7.2.5.1.1 Obtain a clean 100 mL volumetric flask.

7.2.5.1.2 Place 50 mL of the Daily Working Calibration Standard in the flask.

7.2.5.1.3 Dilute to volume with reagent blank. Stopper and mix.

7.2.5.2 Continuing Calibration Verification for Instrument 024 (MS CCV)

7.2.5.2.1 Obtain a clean 100 mL volumetric flask.

7.2.5.2.2 Place 50 mL of reagent blank in the flask.

7.2.5.2.3 Pipette 0.25 mL of the MS CALSTD-1 stock standard into the flask.

7.2.5.2.4 Pipette 0.25 mL of the MS CALSTD-2 stock standard into the flask.

7.2.5.2.5 Pipette 0.25 mL of the MS CALSTD-3 stock standard into the flask.

7.2.5.2.6 Pipette 0.25 mL of a 20 mg/L W standard into the flask.

7.2.5.2.7 Dilute to volume with reagent blank. Stopper and mix.

7.2.6 Reporting Limit (RL) Standards

The reporting limit standards are prepared fresh daily from the same stock as the calibration standards using the reagent blank. The analyte concentrations must be less than or equal to the respective reporting limits. Multiple solutions may be required in order to satisfy all of the project and client specific reporting limits. Alternate reporting limit concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents module in TALS. Prepare the Reporting Limit Standard for the Agilent 7700 as detailed below.

7.2.6.1 RL Standard for Instruments 077 and 078 (ms 77 RL)

7.2.6.1.1 Obtain a clean 50 mL volumetric flask.

7.2.6.1.2 Place 30 mL of reagent blank in the flask.

7.2.6.1.3 Pipette 0.5 mL of the ms 77 cal std solution into the flask.

7.2.6.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.7 Daily Linear Range Standard

The Linear Range standard is prepared from the same stock as the calibration standards using reagent blank.

7.2.7.1 Daily Linear Range Standard for Instruments 077 and 078 (MS 77 LR STD)

7.2.7.1.1 Obtain a clean 500 mL volumetric flask.

7.2.7.1.2 Place 50 mL of reagent blank in the flask.

7.2.7.1.3 Pipette 1.0 mL of a 1,000 mg/L As standard into the flask.

7.2.7.1.4 Pipette 2.5 mL of a 1,000 mg/L Ba standard into the flask.

7.2.7.1.5 Pipette 1.0 mL of a 1,000 mg/L Be standard into the flask.

7.2.7.1.6 Pipette 1.0 mL of a 1,000 mg/L Cd standard into the flask.

7.2.7.1.7 Pipette 1.0 mL of a 1,000 mg/L Co standard into the flask.

7.2.7.1.8 Pipette 2.5 mL of a 1,000 mg/L Cr standard into the flask.

7.2.7.1.9 Pipette 2.5 mL of a 1,000 mg/L Cu standard into the flask.

7.2.7.1.10 Pipette 1.0 mL of a 1,000 mg/l Li standard into the flask.

7.2.7.1.11 Pipette 5.0 mL of a 1,000 mg/L Mn standard into the flask.

7.2.7.1.12 Pipette 1.0 mL of a 1,000 mg/L Mo standard into the flask.

7.2.7.1.13 Pipette 2.5 mL of a 1,000 mg/L Ni standard into the flask.

7.2.7.1.14 Pipette 2.5 mL of a 1,000 mg/L Pb standard into the flask.

7.2.7.1.15 Pipette 0.5 mL of a 1,000 mg/L Sb standard into the flask.

7.2.7.1.16 Pipette 1.0 mL of a 1,000 mg/L Se standard into the flask.

7.2.7.1.17 Pipette 1.0 mL of a 1,000 mg/L Sn standard into the flask.

7.2.7.1.18 Pipette 1.0 mL of a 1,000mg/l Sr standard into the flask.

7.2.7.1.19 Pipette 0.5 mL of a 1,000 mg/L Tl standard into the flask.

7.2.7.1.20 Pipette 1.0 mL of a 1,000 mg/L U standard into the flask.

7.2.7.1.21 Pipette 1.0 mL of a 1,000 mg/L V standard into the flask.

7.2.7.1.22 Pipette 2.5 mL of a 1,000 mg/L Zn standard into the flask.

7.2.7.1.23 Pipette 0.05 mL of a 10,000 mg/L Th standard into the flask.

7.2.7.1.24 Dilute to volume with reagent blank. Stopper and mix.

7.2.8 Internal Standard (IS) Solution (77 I.S. / MS I.S. INT)

The internal standard solution is added continuously by peristaltic pump through a mixing tee. The concentrations and components are specified in Attachment 4. Prepare the IS solution as follows:

7.2.8.1 Obtain a clean 250 mL volumetric flask.

7.2.8.2 Place 100 mL of reagent blank in the flask.

7.2.8.3 Pipette 1.2 mL of the 1,000 mg/L Ge Standard into the flask.

7.2.8.4 Pipette 0.4 mL of the 1,000 mg/L Ho Standard into the flask.

7.2.8.5 Pipette 0.4 mL of the 1,000 mg/L In Standard into the flask.

7.2.8.6 Pipette 0.75 mL of the 1,000 mg/L Sc Standard into the flask.

7.2.8.7 Pipette 1.5 mL of the 1,000 mg/L ^{6}Li Standard into the flask.

7.2.8.8 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9 Interference Check Standard Solutions (ICSA / ICSAB)

The interference check standard solution (ICSA) and the spiked interference check standard solution (ICSAB) are prepared as follows:

7.2.9.1 ICSA Standard (ms 77 icsa / MS ICSA)

7.2.9.1.1 Obtain a clean 100 mL volumetric flask.

7.2.9.1.2 Place 50 mL of reagent blank in the flask.

7.2.9.1.3 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9.2 ICSAB Standard (MS 77 ICSAB / MS ICSAB)

7.2.9.2.1 Obtain a clean 100 mL volumetric flask.

7.2.9.2.2 Place 50 mL of reagent blank in the flask.

7.2.9.2.3 Pipette 0.5 mL of the MS CALSTD-1 stock standard into the flask.

7.2.9.2.4 Pipette 0.5 mL of the MS CALSTD-2 stock standard into the flask.

7.2.9.2.5 Pipette 0.5 mL of the MS CALSTD-3 stock standard into the flask.

7.2.9.2.6 For the Agilent 7700 instruments pipette 0.5 mL of the MS BRC CALSTD stock standard into the flask.

7.2.9.2.7 For the Agilent 7700 instruments pipette 0.5 mL of a 20 mg/L Sr standard into the flask.

7.2.9.2.8 For the Agilent 7700 instruments pipette 0.5 mL of a 200 mg/L Li standard into the flask.

7.2.9.2.9 For the Agilent 7500 instrument pipette 0.5 mL of a 20 mg/L W standard into the flask.

7.2.9.2.10 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.2.11 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.10 6020A only -Low Level Initial Calibration Verification and Low-Level Continuing Verifications (ms 77 LLCCV / MS LCCV)

The low level ICV / low level CCV solution is prepared from the same source as the calibration standards. The low level standard is prepared fresh each day of analysis in reagent blank. The concentration is shown in Attachment 10. Prepare the low level standard solution as follows:

7.2.10.1 Obtain 2 clean 100 mL volumetric flask.

7.2.10.2 Place 50 mL of reagent blank in each flask.

7.2.10.3 Prepare a working standard by adding 0.5 ml of a 1000mg/l Li and 0.1ml of a 20mg/l Sr to the first flask and bring to volume.

7.2.10.4 Pipette 1.0 mL of the MS LLCCV1 stock standard into the second flask.

7.2.10.5 Pipette 1.0 mL of the MS LLCCV 2A stock standard into the second flask.

7.2.10.6 Pipette 1.0 ml of working standard for Sr and Li into the second flask.

7.2.10.7 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.3 Reagents

7.3.1 **Reagent Water –** Water free of the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.3.2 **Reagent Blank - Agilent 7700, 2% HNO3/0.5% HCl –** Carefully dilute 40 mL of concentrated HNO3 and 10 mL of HCl in 2.0 L of reagent water. This solution is used to dilute samples and it is used for the initial and continuing calibration blanks.

7.3.3 **Reagent Blank - Agilent 7700, 5% HNO3/5% HCL (Zr only) –** Carefully dilute 100 ml of concentrated HNO_3 and 100 ml if HCL in 2.0 L of reagent water. This solution is used to dilute samples and it is used for calibration blanks.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Water[2]	HDPE	50 mLs	HNO_3, pH < 2	180 Days	SW-846
Soil	Glass	4 oz	Cool $\leq$ 6°C[3]	180 Days	SW-846

[1]Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Holding Times are calculated from the date the sample was collected.

[2]Water samples collected for dissolved elements are filtered immediately on-site by the sampler before adding preservative.

[3]Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration per SW-846. Samples which will be used to aliquot for both analyses must be refrigerated. Therefore the laboratory routinely refrigerates samples to be analyzed by Methods 6020A or 6020B.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply. Quality control requirements are summarized in Attachment 9.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

For aqueous and soil samples, the method blank consists of reagent water that has been processed in the same manner as the samples. For soil samples analyzed under DoD QAPPs, the method blank consists of <1 mm glass beads that have been processed in the same manner as the samples. One method blank must be processed with each preparation batch.

Acceptance Criteria: Method blank results are acceptable if the concentration for each analyte of interest is less than ½ the reporting limit (RL). For DoD QSM 5.0 the control limit is less than ½ LOQ. In the absence of project specific reporting limits, if the blank is less than 10% of the lower limit of quantitation check sample concentration, less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater, then the method blank is considered acceptable.

Corrective Action: If the method blank does not meet the acceptance criteria, the source of contamination should be investigated to determine if the problem can be minimized or eliminated. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following

circumstances, may be reported as qualified (qualifier flags or narrative comments):

- The same analyte was not detected in the associated samples;
- The method blank concentration is less than 1/10 of the measured concentration of any sample in the batch;
- The method blank concentration is less than 1/10 the specified regulatory limit; or
- The analyte is a common laboratory contaminant (e.g., copper, zinc, iron, or lead) less than 2 times the RL. Note that some programs do not recognize common lab contaminants or have a more stringent criterion (e.g., DoD QSM 5.0 allows common laboratory contaminants up to the RL).

If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative and the client must be notified.

9.3 Laboratory Control Sample (LCS)

The LCS consists of reagent water that is spiked with the analytes of interest at the project specific action level or, when lacking specific action levels, at approximately the mid-point of the calibration range (summarized in Attachment 10). For soil samples analyzed under DoD QAPPs, the LCS consists of <1 mm glass beads that have been spiked with the analytes of interest and processed in the same manner as the samples. One LCS must be processed for each preparation batch.

Acceptance Criteria: LCS control limits are based on three standard deviations of past laboratory results or program specific requirements. These limits must not exceed 80-120%. The control limits are maintained in TALS. For DoD QSM 5.0 the laboratory must use QSM Appendix C Limits for batch control if project limits are not specified.

Corrective Action: If the LCS recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reanalyzed One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. If project requirements allow this exception, the data may be accepted with qualifiers, an NCM must be generated, and the failure narrated in the final report.

9.4 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

The MS is prepared by taking a second aliquot of a selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 10). An MSD is prepared by taking a third aliquot of the selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 10). The MS and MSD are processed in the same manner as the samples. One MS/MSD pair must be processed for each preparation batch. Some programs (e.g., DoD) require that matrix spikes can be performed only on project samples, and that the samples to be used are identified on the chain of custody form. The spike concentration should be the same level as the LCS.

Acceptance Criteria: Control limits are based on historical data or project specific requirements. Historical control limits are based on three standard deviations of past laboratory results. These limits are not to exceed 75-125% recovery, and 20% relative percent difference (RPD). The control limits are maintained in TALS. For DoD QSM 5.0 the laboratory must use QSM Appendix C limits for batch control if project limits are not specified.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the

analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision.

9.5 Interference Check Solutions (ICSA/ICSAB)

The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Attachment 5. The interference check solutions must be analyzed at the beginning of every analytical run and once every 12 hours thereafter. The results of solution "A" and solution "AB" should be monitored for possible interferences.

Acceptance Criteria: The non-spiked analytes in the A solution must be less than 2x the RL. The results for the trace elements (B portion) must be ± 20% of the expected value. In addition, the internal standard recoveries for both the ICSA and AB must be within 70-150% for Method 6020A, 30-150% for Method 6020B and 30-120% for DoD. Some programs have control limits for the non-spiked elements in the ICSA. Please check the client specific requirements. For DoD QSM 5.0 the ICSA for non-spiked elements is controlled to less than the absolute value of the LOD unless they are a verified impurity.

Corrective Action: If the ICSAB results exceed the 20% limit or the ICSA is out for DoD QSM 5.0, then the analysis sequence must be terminated. For DoD QSM 5.0 if the ICSA is outside of the control limits for the non-spiked elements the sequence must also be terminated. The problem must be investigated and fixed. The ICSA and all affected samples must be re-analyzed.

NOTE: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (e.g., ICPAES), acceptance criteria will be applied at that level and the data accepted.

9.6 Internal Standards Evaluation for Samples

The IS recovery in samples cannot fall below 70% or be above 150% of the intensity of the calibration blank for 6020A and 30-150% for 6020B. If sample IS recoveries fall outside of these criteria, a five-fold (1:4) dilution must be performed, the dilution analyzed, and the same acceptance criteria applied. For DoD QSM 5.0 the internal standard for samples is controlled to 30-120%.

9.7 Serial Dilution

One serial five-fold dilution should be analyzed per preparation batch. If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 50 times above the MDL), the serial dilution must agree to within 10% of the original analysis. If not, an interference effect is suspected, which must be described in an NCM and included in the final report narrative. Samples identified as blanks should not be used for serial dilution. For DoD QSM 5.0 the serial dilution is evaluated if the parent sample concentration is greater than 50x the

LOQ prior to dilution. If the acceptance criteria are not met then the parent sample is flagged "J". Method 6020B sets the calculation level at 25x RL and the required limit at 20%.

9.8 Post-Digestion Spike Addition (PDS)

A PDS is performed for each batch. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80 - 120% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected. Typically the concentration of the PDS is 200 µg/L for each element except silver which is spiked at 50 µg/L. For DoD QSM 5.0 if the parent sample concentration is less than 50x the LOQ prior to dilution then the PDS must recover within 80-120%. If the recovery is outside of the control limits for a given element then the parent sample is flagged "J". Method 6020B allows limits of ± 25%.

9.9 Linear Range Verification (LRA/LRC)

The LDRs should be verified whenever, in the judgment of the analyst, a change in the analytical performance caused by either a change in instrument hardware or operating conditions would dictate the necessity to re-establish them. 6020B and DoD QSM require verification of linear ranges in each analytical run. As described in Section 7.2.7, a lower concentration is used for the daily check than is used for the quarterly determination.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the Linear Range Verification standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analyst must run a standard at a lower concentration until the criteria is met or the samples cannot exceed the level of the highest calibration standard.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure identified after the work has been completed must also be documented in an NCM, with a cause and corrective action described.

10.3 Instrument Maintenance

See Section 20 in the QAM

10.4 Instrument Troubleshooting

See Attachment 11

10.5 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0014 and DV-IP-0015).

10.6 Calibration

10.6.1 Instrument Start Up

Set up the instrument according to manufacturer's operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning. It is recommended that the instrument be flushed with the ICSA solution to help condition the cones and improve stability. Allow the instrument time to rinse completely before tuning the instrument.

10.6.2 Oxide\Doubly Charge Performance Check

With the sample probe in the Tune solution verify that the oxides and doubly charged ions are less the 3% by running the Tune report.

10.6.3 Instrument Tuning / Mass Calibration

Tune the instrument with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 5 times. Mass calibration and resolution checks using the tuning solution must be completed at the beginning of every day. If either of the following conditions fails the instrument setup must be re-evaluated and the solution rerun.

Mass Calibration Check – The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

Mass Resolution Check - The resolution at 5% peak height should be approximately 0.75amu.

NOTE: Method 6020B states to use the manufacturer's instructions for the tune. Since the laboratory may perform analysis for Method 6020A on the same instrument, the same requirements are applied to both.

10.6.4 Initial Calibration

The ICP-MS is calibrated each day of operation using a blank and a single standard (see Section 7.2.3). At least three integrations are employed. The validity of the calibration is determined by the subsequent calibration verifications, which are performed at concentrations as described in the next sections.

10.6.5 Low-Level Initial Calibration Verification (LLICV/ICVL)

A low-level ICV standard at or below the reporting limit (see Section 7.2.10) is analyzed after the initial calibration for Method 6020A. This is a standard obtained from the same vendor used for calibration.

Acceptance Criteria: The ICVL recovery must be within 70-130%.

The ICVL can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICVL results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.6 Mid-Level Second-Source Initial Calibration Verification (ICV)

A 40 μg/L ICV standard (see Section 7.2.4) is analyzed immediately after the initial calibration. This is a standard obtained from a different vendor than the standard used for calibration.

Acceptance Criteria: The ICV recovery must be within 90-110%. The ICV can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICV results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.7 Calibration Blank

An initial calibration blank (ICB) is analyzed after the ICV. Continuing calibration blanks (CCBs) are analyzed after each continuing calibration verification. The appropriate reagent blank is used for the blanks.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as zinc and iron must be less than the RL. In addition, the internal standard recoveries must be within 70-150% of the associated calibration blank for Method 8020A or

30-150% for Method 6020B. Client specific requirements take precedence. DoD QSM 5.0 requires control of blanks to a concentration less than or equal to the LOD with the internal standard recoveries of 30-120%.

Corrective Action: If the calibration blank exceeds acceptance limits, then the possibility of instrument contamination should be examined, particularly the possibility of carry-over from high level samples. The blank can be reanalyzed, and if successful, analysis can continue. However, samples tested after high-level samples should be retested. If the reanalysis is not successful, then the analysis should be terminated. After the problem is corrected, recalibrate and reanalyze all samples tested since the last acceptable CCB.

10.6.8 Reporting Limit (RL/CRI) Verification Standard

Because the ICP-MS calibration does not include multiple calibration levels, an independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. The RL verification standard (see Section 7.2.6) is analyzed after the daily ICB.

Acceptance Criteria: For Method 6020A, the results should be within 50% of the expected value. Some programs may require tighter controls. For Method 6020B and DoD QSM 5.0 the control limits are 80-120%.

Corrective Action: If the RL verification fails to meet acceptance limits, data for the associated samples must be assessed. For example, if the results are high, consider blank contamination, and if the results are low, consider MDL verifications. At a minimum, sample results must be qualified in the final report. For DoD QSM 5.0, if the low-level standard does not meet the limits when spiked at the required project RL, the run sequence must be terminated.

10.6.9 Lower Limit of Quantitation Check (LLQC, LLOQ)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits, quarterly and on an as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the RL is that this standard is carried through the entire preparation and analytical procedure. Prepare 7 aliqouts.

Acceptance Criteria: LLQC is verified when all analytes are detected within ± 35% of their true value. The RSD should be ≤20%

Corrective Action: If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.6.10 6020A Only - Low-Continuing Calibration Verification (LLCCV/CCVL) Standard

A low-level CCV standard is analyzed after every set of ten samples and at the end of the analytical sequence.

Acceptance Criteria: The CCVL recovery must be within 70-130%. In addition, the IS recovery must be within method limits. If CCVL results are not within these limits, the CCVL can be reanalyzed, but it must be successful twice in succession. If the calibration cannot be verified within these specified limits, the analysis of samples containing the affected analytes at similar concentrations cannot continue until the cause is determined and the CCVL standard successfully analyzed.

For the state of Washington the CCVL must work on the first attempt.

Corrective Action: If the CCVL fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCVL. If the associated samples are at levels greater than 10X the level of the CCVL the data may be considered acceptable but the failure must be documented with an NCM and addressed in the case narrative.

For the state of Washington the previous 10 samples must be reanalyzed. A recalibration is not necessary if the two CCVL's following the failure are successful but the samples must still be rerun.

10.6.11 Continuing Calibration Verification (CCV) Standard

A 50 μg/L CCV standard (see Section 7.2.5) is analyzed after every set of ten samples or every 2 hours, whichever is most frequent, and at the end of the analytical sequence.

Acceptance Criteria: The CCV recovery must be within 90-110%. In addition, the IS recovery must be within 70-150% for Method 6020A or 30-150% for Method 6020B. If the CCV results are not within these limits, the CCV can be reanalyzed, but it must be successful twice in succession or further corrective action must be taken.

For the state of Washington the CCV must work on the first attempt

Corrective Action: If the CCV fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCV.

For the state of Washington the previous 10 samples must be reanalyzed. A recalibration is not necessary if the two CCV's following the failure are successful but the samples must still be rerun.

10.7 Sample Analysis

10.7.1 Report the average of at least three integrations for all field and QC samples analyzed.

10.7.2 Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

10.7.3 Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element. See Attachment 3 for examples.

10.7.4 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte. DoD QSM 5.0 requires that samples be diluted and reanalyzed if they are above the daily linear range check standard. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the RL. (The sample should be diluted to the approximate midrange of the analytical curve.) See Section 9.9 for the linear range verification requirements.

10.7.5 The analytical run sequence should be performed as follows to meet all quality control criteria:

Instrument initialization / Warm-Up
Tune instrument
Perform mass calibration
Perform resolution check
Validate tuning criteria
Calibration blank
Calibration standard

ICV
ICB
LLICV
RL verification standard
LLQC(as needed)
ICSA
ICSAB
LRA
CCV
CCB
LLCCV (6020B)
10 Samples (which can include all sample types)
CCV
CCB
LLCCV (6020B)
Reslope
CCV
CCB
LLCCV

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 ICV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}} \right) \times 100\%$$

11.3 CCV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}} \right) \times 100\%$$

11.4 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{SSR - SR}}{\text{SA}} \right) \times 100\%$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When the sample concentration is less than the detection limit, use SR = 0 for the purpose of calculating %R.

11.5 The relative percent difference (RPD) between sample duplicates is calculated according to the following equation:

$$RPD = \left| \frac{DU1 - DU2}{\frac{1}{2}(DU1 + DU2)} \right| \times 100$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

11.6 The final concentration for an aqueous sample is calculated as follows:

$$\text{Result}\,(\mu\text{g/L}) = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration from instrument readout, ppb

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

11.7 The concentration determined in digested solid samples when reported on a dry weight basis is as follows:

$$\text{Result}\,(\mu\text{g/kg}) = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration from instrument readout, ppb

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight, in g, of wet sample digested

S = Percent solids/100

11.8 Sample data are reviewed by the analyst (Level 1 data review) and documented on the data review checklist (See SOP DV-QA-0020). The data package is then submitted for level 2 review by another analyst or data reviewer. Second level review is documented on the same checklist initiated by the analyst. The data review process is explained in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is

present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-002. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD, DOE and TX TRPP projects, an MDL verification is performed quarterly.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard.

12.2.1 Prepare an MDLV standard at 2-4 times the calculated MDL concentration.

12.2.2 Analyze the MDLV standard immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

12.2.3 The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio > 3, under routine instrument conditions.

12.2.4 If the first MDLV is not detected, re-prepare the MDLV standard at twice the original concentration and analyze. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.3 Instrument Detection Limit Study

Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each analyte used for analysis in accordance with Policy DV-QA-014.

12.3.1 Pour out seven undigested calibration blanks and run them on three non-consecutive days.

12.3.2 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.3.3 Method 6020B requires an initial verification of the IDL using 10 replicates in a single analytical sequence but no longer requires quarterly verification. Reverification is required after major maintenance, such as changing the detector.

12.3.4 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.4 Linear Dynamic Range (LDR)

12.4.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte used on each instrument. The linear

range is the concentration above which results cannot be reported without dilution of the sample.

12.4.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.4.3 The LDR is determined by analyzing successively higher standard concentrations of the analytes of interest. A minimum of three standards are required for the initial and on-going studies, and one of the levels must be at the upper end of the range. The calculated concentrations must be within 10% of the stated concentrations.

12.4.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions.

12.4.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.5 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.5.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.5.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.5.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.5.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.5.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.1 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A

new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 **Aqueous Acidic (Metals) -** Corrosive - Waste Stream J

14.2.2 **Expired reagents and standards –** Contact the Waste Coordinator.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 6020A: *Inductively Coupled Plasma - Mass Spectrometry*, Revision 1, February 2007.

15.1.2 Method 6020B: Inductively Coupled Plasma - Mass Spectrometry, Revision 2, July 2014.

15.1.3 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.4 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.5 Method 3050B, Acid Digestion of Sediments, sludges and soils, Rev. 2,

Dec. 1996.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/20/2010

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5, July 2013.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6020A	Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.
2	EPA 6020A	Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.
3	EPA 6020A	Corrective action for a PDS failure will be limited to flagging the PDS indicating the failed analyte and the recovery rather than diluting and reanalyzing the sample.
4	EPA 6020A	Internal standard recoveries are based on the intensities of the internal standards in the most recent calibration blank rather than the intensities of the internal standards in the initial calibration standard.
5	EPA 6020A	Method 6020A states that the dilution test is applicable if the matrix sample is at least 50x the reporting limit. TestAmerica uses the tighter limit of 50x the MDL.
6	EPA 6020B	Method 6020B states to tune the instrument according to manufacturer's instructions. TestAmerica continues to use the tune requirements in Method 6020A in order to be able to run samples by either method under the same tune.
7	EPA 6020B	Method 6020B does not include the analysis of the ICS-AB. TestAmerica continues to analyze this QC sample due to various program requirements.
8	EPA 6020A/B	The tuning criteria listed in method 200.8 for mass resolution is used to satisfy the requirements of method 6020A/B

17.0 Attachments

Attachment 1: Standard Reporting Limits for Water and Soil
Attachment 2: Recommended Elemental Equations
Attachment 3: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes
Attachment 4: Internal Standards and Corresponding Metals
Attachment 5: Interference Check Sample Components and Concentrations
Attachment 6: Suggested Mass Choices
Attachment 7: Tuning Solution and P/A Solution
Attachment 8: Suggested Tuning and Response Factor Criteria
Attachment 9: Summary of Quality Control Requirements
Attachment 10: Calibration, Calibration Verification, and Spike Concentrations
Attachment 11: Troubleshooting

18.0 Revision History

Revision 8, Dated 31 October 2018
- Annual Review
- Updated copyright section

Revision 7, Dated 31 October 2017
- Annual Review
- Updated section 10.6.3 Mass Resolution criteria to method 200.8
- Added Section16 item 8 for Mass Resolution criteria

Revision 6, Dated 26 October 2016
- Updated sections 7.2.3.1.9, 7.2.4.1.8, 7.2.9.2.8 and 7.2.10 for how Sr and Li are prepared in the standards
- Updated Section 10.6.9 and Attachment 9 to reflect 6020B limits and use of in-house limits
- Updated Sections 10.6.10 and 10.6.11 for the state of Washington requirements
- Updated Section 15 for consistency with other SOPs to reflect reference for SW-846

Revision 5, Dated 28 September 2016
- Added Li and Sr as analytes to the SOP
- Revised Section 1.2 to more clearly identify elements addressed by this SOP
- Added prep method numbers to Sections 1.3 and 15
- Combined Footnotes 1 and 3 for table in Section 8.0 to eliminate redundancy
- Removed references to AFCEE throughout SOP; no longer utilize AFCEE protocols
- Expanded MS/MSD failure corrective action in Section 9.4 to reflect current policy
- Added note in Section 10.6.3 regarding Method 6020B requirements for tune
- Added LLICV acceptance criteria for Method 6020B, different from 6020A.
- Removed references to Instrument 24 (Agilent 7500) throughout
- Added requirements for IDLs by 6020B to Section 12.3
- Moved Section 12.5 to new Section 9.9
- Revised Sections 12.6 and 12.7 to reflect current practice
- Added Method exceptions 6 and 7 for Method 6020B where the laboratory performs a more stringent practice than required.

Revision 4, Dated 31 December 2015
- Added method 6020B

- Added 6020B limits to Section 9.7 and 9.8, SD and PDS
- Corrected references

Revision 3, Dated 30 April 2015

- Annual Review
- Language and formatting changes throughout
- Corrected tuning sample requirement from 4 replicates to five replicates
- Changed 12.4.2 to 12.5
- Added Section 2.1
- Added new Section 2.3 to describe reaction cell
- Added new Section 4.2.3
- Added batch definition
- Deleted Attachment 2
- Deleted Attachment 4
- Added new Attachment 1 standard reporting limits
- Section 4.3.2 enlarged expected IS intensities
- Integrated Sections 4.4 and 4.5 into 4.2
- Created new Section 4.3 Doubly charged ions
- Added new Section 6.2.3 volumetric flasks
- Section 7.2.1.4 corrected Mg addition to standard
- Section 7.2.2 replaced P/A section with same section from DV-MT-0025
- Added P/A section to Attachment 7
- Section 7.1.3 added reference to standards SOP
- Added all new standard prep information into Sections 7.2.3 – 7.2.9
- Section 9.3 changed spike level from midpoint of LR to midpoint of cal curve
- Added note to Section 9.4
- Changed timing of ICSA to beginning of analytical run and every 12 hours
- Changed concentration limits for SD in Section 9.7 to 50x MDL
- Added method modification 5 to address SD limit
- 10.6.7 added DoD 5.0 language to corrective action
- Removed Section 10.3
- 10.7.5 added DoD requirement to dilute above daily LR
- 11.5 Corrected RPD calculation
- 11.7 changed to dry weight correction
- 12.2.1 changed MDLV spike level to 2-4x MDL

Revision 2, dated 09 April 2014

- Annual review
- Updated Section 7.2 for standards to reference TALS for how to make
- Added Section 7.4.4 for 5%HNO3/5%HCL for Zirconium
- Added Section 10.4 for Maintenance
- Added Section 10.5 for Troubleshooting
- Updated Sections 9 and 10 to include requirements for DoD QSM 5.0
- Added reference to DoD QSM 5.0.

Revision 1, dated 15 July 2013

- Annual review
- Corrected formatting
- Added section 3.16
- Added reference to data review in section 10.7
- Added documentation information in section 11.8
- Added detail to note associated with section 14.2

- Updated reference in section 15.2
- Removed Attachment 13

Revision 0.3, dated 13 July 2012

- Revised standards preparation procedures in Section 7
- Added section 7.2.2
- Split acid diluent into two solutions depending upon instrument
- Updated standard mixes used to prepare standards; instrument specific mixes as needed
- Clarified requirements for preservation of soil samples for ICPMS only analysis, Section 8
- Revised list of common lab contaminants in method blank corrective action (Section 9.2)
- Added section 10.5.2: oxide/doubly charged performance check
- Updated Sections 9.2 and 10.5.7 to control method blanks and calibration blanks to ½ the RL
- Updated Sections 9.1, 10.1, 10.2 and 12.1 to reflect current practice.

Revision 0.2, dated 08 July 2011

- Added Section 4.4 on polyatomic interferences
- Added Instruments to Section 6.1
- Section 10.5.1 Added to condition cones with the ICSA solution
- Added Section 10.6.3 to reflect soil dilution practices
- Section 11.4 Corrected the RPD calculation
- Added section 11.1 referencing corporate SOP CA-Q-S-005 “Calibration Curves”
- Added section 12.2 “MDL Verification (MDLV)”
- Added Attachment 13 “ICP-MS Technical Data Review Checklist”

Attachment 1

Standard Reporting Limits for Water and Soil

Element Name	Element Symbol	Water (ug/L)	Soil (ug/Kg)
Aluminum	Al	50	5,000
Antimony	Sb	2.0	200
Arsenic	As	5.0	600
Barium	Ba	1.0	200
Beryllium	Be	1.0	100
Cadmium	Cd	1.0	100
Chromium	Cr	2.0	200
Cobalt	Co	1.0	100
Copper	Cu	2.0	250
Iron	Fe	50	5,000
Lead	Pb	1.0	150
Lithium	Li	50	5,000
Manganese	Mn	1.0	250
Molybdenum	Mo	2.0	200
Nickel	Ni	2.0	150
Selenium	Se	5.0	500
Silver	Ag	5.0	100
Strontium	Sr	10	100
Thallium	Tl	1.0	100
Thorium	Th	5.0	200
Tin	Sn	10	2,500
Tungsten	W	5.0	500
Uranium	U	1.0	100
Vanadium	V	5.0	500
Zinc	Zn	10	1,000
Zirconium	Zr	0.5	---

Attachment 2

Recommended Elemental Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

Where M = Total ion count rate at the specified mass.

Attachment 3

Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS, SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

Attachment 3 (cont.)
Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Attachment 4
Internal Standards and Corresponding Metals

IS	ICP-MS 077/078
^{6}Li	Be
Sc	Li, Na, Mg, Al, K, Ca
Ge	Sr, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se
In	Mo, Ag, Cd, Sn, Sb, Ba
Ho	Tl, Pb, Th, U, W

Attachment 5
Interference Check Sample Components and Concentrations

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	110.0
Ca	100.0	110.0
Fe	100.0	110.0
Mg	100.0	110.0
Na	100.0	110.0
P	100.0	100.0
K	100.0	110.0
S	100.0	100.0
C	200.0	200.0
Cl	1000.0	1000.0
Mo	2.0	2.1
Ti	2.0	2.0
As	0.0	0.1
Sb	0.0	0.1
Be	0.0	0.1
Ba	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Pb	0.0	0.1
Li	0.0	1.0
Mn	0.0	0.1
Ni	0.0	0.1
Nb	0.0	0.2
Pd	0.0	0.1
Pt	0.0	0.1
Se	0.0	0.1
Sr	0.0	0.1
Tl	0.0	0.1
Th	0.0	0.1
Sn	0.0	0.1
Ag	0.0	0.1
U	0.0	0.1
V	0.0	0.1
W	0.0	0.1
Zn	0.0	0.1

Attachment 6
Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
"7"	Lithium
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
93	Niobium
105	Palladium
195	Platinum
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"88"	Strontium
"**205**", 203	Thallium
232	Thorium
192	Tungsten
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
139	Lanthanum
118	Tin
238	Uranium
35, 37	Chlorine
98, 96, 92, **97**, 94, "95"	Molybdenum
72	Germanium (IS)
165	Holmium (IS)
115	Indium (IS)
6	Lithium (6+) (IS)
45	Scandium (IS)

Attachment 7: Tuning Solution and P/A Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below is a suggested solution covering a typical mass calibration range. Instrument manufacturer recommendations should be followed for tuning solutions.

The P/A solution is used to monitor the correlation between the Pulse and Analog parts of the electron multiplier. This solution is prepared at different concentrations depending on the current instrument conditions. The parent standard concentration is shown below.

Element	Tuning Concentration (µg/L)	P/A Concentration (mg/L)
Al		5
As		20
Ba	10	5
Be	10	20
Bi		5
Cd		20
Ce	10	
Co	10	5
Cr		5
Cu		5
Ge		10
In	10	5
Ir		5
^{6}Li		5
Li	10	
Lu		5
Mg	10	10
Mn		5
Mo		10
Na		5
Ni		10
Pb	10	10
Pd		10
Rh	10	
Ru		10
Sb		10
Sc		5
Sn		10
Sr		5
Tb		2.5
Th		50
Ti		50
Tl	10	50
U	10	50
V		50
Y	10	2.5
Zn		20

Attachment 8:

Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>2,000
Rh	>20,000
Pb	>10,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

Attachment 9:

Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
LLOQ (6020B only)	With initial setup, Quarterly and on an as needed basis	65 - 135% recovery or in-house limits	Terminate analysis; correct the problem; recalibrate.
LLICV (6020A only)	Beginning of every analytical run.	70 - 130% recovery. 6020A IS, 70-150% rec.	Terminate analysis; correct the problem; recalibrate.
ICV	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result is < ½ RL.	Terminate analysis; correct the problem; recalibrate.
LLCCV (6020A only)	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	70 - 130% recovery. 6020A IS, 70-150% rec.	See Section 10.6.10. Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCV	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	90 - 110% recovery.	Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.

Attachment 9: Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
CCB	Immediately following each CCV.	The result must be < ½ RL. .	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA	Beginning and every 12 hours.	Monitor for possible interferences.	See Section 9.5
ICSAB	Immediately following each ICSA.	Monitor for possible interferences.	See Section 9.5
Method Blank	One per lot of 20 field samples or fewer.	The result must be < ½ RL. Sample results greater than 10x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Re-run once. If > ½ RL, redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Serial Dilution	One per batch of 20 field samples or fewer.	90 - 110% recovery	See Section 9.7 for additional requirements.
Post-Digestion Spike	One per batch of 20 field samples or fewer.	80-120% recovery	See Section 9.8.
Laboratory Control Sample	One per batch of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.3
Matrix Spike	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.6 for additional requirements.

Attachment 10
Calibration, Calibration Verification, and Spike Concentrations

Element	Initial Calibration (µg/L)	ICV (µg/L)	CCV (µg/L)	LCS (µg/L)	MS/MSD (µg/L)	Post Digestion Spike (ug/L)
Aluminum	10000	40	50	400	400	20000
Antimony	100	40	50	40	40	200
Arsenic	100	40	50	40	40	200
Barium	100	40	50	40	40	200
Beryllium	100	40	50	40	40	200
Cadmium	100	40	50	40	40	200
Chromium	100	40	50	40	40	200
Cobalt	100	40	50	40	40	200
Copper	100	40	50	40	40	200
Iron	10000	4000	5000	400	400	20000
Lead	100	40	50	40	40	200
Lithium	1000	400	500	100	100	200
Manganese	100	40	50	40	40	200
Molybdenum	100	40	50	40	40	200
Nickel	100	40	50	40	40	200
Selenium	100	40	50	40	40	200
Silver	100	40	50	40	40	50
Strontium	100	40	50	40	40	200
Thallium	100	40	50	40	40	200
Thorium	100	40	50	40	40	--
Tin	100	40	50	40	40	200
Tungsten	100	40	50	40	40	200
Uranium	100	40	50	40	40	200
Vanadium	100	40	50	40	40	200
Zinc	100	40	50	40	40	200
Zirconium	100	40	50	40	40	--

This procedure has been developed for twenty elements. Additional elements may be included in the calibration solution at the above levels. Levels may be adjusted to meet specific regulatory or client programs.

Attachment 11

ICP-MS Troubleshooting Guide

Problem	Possible Cause/ Solution
High Calibration Blanks	Inspect historical blank data to determine root cause Inspect, clean or replace torch Inspect, clean or replace pump tubing or sample tubing Inspect, clean or replace nebulizer Remake blank solution Recalibrate instrument
Instrument Drift	Make sure instrument has warmed properly Condition cones to aid stability Reslope to correct for changing cone conditions during run Stop run, clean cones and start over with a new calibration
Erratic Readings, High RSDs	Check nebulizer pressure Check sample flow around the pump, adjust tension on pump tubing to ensure smooth flow Check for clogs in the uptake tubing, nebulizer, or valve Clean or replace nebulizer
Low Sensitivity	Clean cones Adjust lens voltages Remove and clean lens, remove and clean or replace reaction cell
Bad Tune: Bad Mass Cal	Adjust lens voltages, remove and clean lens
Bad Tune: High Oxides	Inspect, clean, or replace torch, nebulizer, and spray chamber

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

Approvals (Signature/Date):

Amelia Byl 10/31/2018
Amelia Byl Date
Technical Specialist

Doug Gomer 10/29/18
Doug Gomer Date
Health & Safety Manager/Coordinator

Roxanne Sullivan 10/29/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 10/29/18
Richard Clinkscales Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from water samples, TCLP leachates, and SPLP leachates, using a separatory funnel. This SOP based on SW-846 Method 3510C, EPA 608, EPA 610, EPA 614, AK102, NWTPH-Dx, and Oklahoma DRO method.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, "Concentration of Organic Extracts", for details concerning the concentration and cleanup of extracts.

2.0 Summary of Method

A measured volume of sample, is placed in a separatory funnel. The pH is adjusted as required for the efficient extraction of specific compounds. The organic compounds are extracted with three portions of methylene chloride. The water phase is discarded. The organic phase is dried using sodium sulfate.

3.0 Definitions

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in "take an aliquot of a sample for testing or analysis." In the context of this SOP, "aliquot" is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

3.6 **Reagent Water** (aka ELGA water – water generated from ELGA water polishing units): Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.3 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented in an NCM.

4.4 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Especially take note of the possibility of phthalate contamination from gloves. Gloves should be changed out frequently and whenever they come in contact with solvent. Glassware should be handled in a fashion that keeps gloves away from the interior and mouth of the glassware.

4.5 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenol may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510C. Method 3510C is preferred over Method 3520C for the analysis of these classes of compounds. However, the recovery of phenols is optimized by using Method 3520C and performing the initial extraction at the acid pH.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The use of separatory funnels to extract samples using methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity. Either a face shield must be worn over safety glasses or goggles must be worn when it is performed.

5.3.2 Glass centrifuge tubes can break in the centrifuge if proper care is not taken. This can lead to a hazardous material spill and endanger employees. Do not exceed the manufacturer's recommended maximum RPM for glass containers. Normally speeds greater than 2700 rpm are not advisable.

5.3.3 The procedure calls for the use of an electric rotator. The rotator is equipped with a safety latch that does not allow the rotator to rotate even if the power switch is turned on. The separatory funnels are secured to the rotator using straps. During the procedure it will be necessary to loosen the straps in order to un-stopper the separatory funnels. Whenever the straps are loose, the safety latch must be fastened to prevent the rotator from rotating.

5.3.4 Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive Poison	2 mg/m3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, and runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit			

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use.

6.1 Supplies

- Separatory funnel, 2-liter with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel, 500-mL with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel rack and mechanical rotator.

- Balance, ≥ 1400 g capacity, accurate to ± 1 g, calibration checked daily per SOP DV-QA-0014.
- pH indicator paper, wide range.
- Class A Graduated Cylinder, sizes ranging from 50 mL to 1 L.
- Media bottles, 300 mL with Teflon-lined caps or capped with aluminum foil.
- Media bottles, 100 mL with Teflon-lined caps or capped with aluminum foil.
- Disposable pipettes, various volumes.
- Stemless glass funnel.
- Glass wool, baked at 400 °C for four hours.
- Mechanical pipette, 1 mL, positive displacement, with disposable tips, calibrated per SOP DV-QA-0008.
- Aluminum foil.
- Paper towels.

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent Water

TestAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.2 Methylene Chloride

Each lot of solvent is tested following SOP CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Acids and Bases

7.3.1 1:1 Sulfuric Acid (H2SO4), *TALS Reagent ID "1:1 H2SO4"*

Place an ice water bath on a stir plate. Place a container with a magnetic stir bar in the bath. While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36N) to 1 part water from the ELGA purification system. Assign a 1 year expiration date from the date made or the vender expiration date, whichever is shorter.

7.3.2 10N Sodium Hydroxide (NaOH), *TALS Reagent ID "10N_NaOH"*

Purchased at ready-to-use concentration from commercial vendors. Assign a 1 year expiration date from the date opened or the vender expiration date, whichever is shorter.

7.3.3 1N Hydrochloric Acid (HCl), *TALS Reagent ID "1N_HCl"*

Dilute 100 mL of stock reagent grade, concentrated HCl to 1000 mL with reagent water.

7.4 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400 °C oven for at least four hours. Store in tightly closed container.

7.5 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours.

7.6 Standards

Please reference SOP DV-OP-0020 and WI-DV-0009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix and Method	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	Amber Glass	1000 mL	Cool, ≤ 6°C	7 Days	40 CFR Part 136.3
Water for Method AK 102	Amber Glass	1000 mL	Cool, ≤ 6°C and pH ≤ 2 with HCl	14 Days if properly preserved. 7 Days if un-preserved.	Method AK 102
Water for Method Oklahoma DRO	Amber Glass	1000 mL	Cool, ≤ 6°C and pH ≤ 2 with HCl	7 Days	Oklahoma Dept. of Environmental Quality
Water for Method NWTPH-DX	Amber Glass	1000 mL	Cool, ≤ 6°C and pH ≤ 2 with HCl	7 Days	NWTPH-Dx
Water for Method 8082 or 8082A	Amber Glass	1000 mL	Cool, ≤ 6°C	None [2]	SW-846 Chapter 4, Revision 4, Feb 2007
Water for Method 8081 or 8082 by Large Volume Injection	Amber Glass	250 mL	Cool, ≤ 6°C	7 Days	40 CFR Part 136.3
Water for Method 8270SIM by Large Volume Injection	Amber Glass	250 mL	Cool, ≤ 6°C	7 Days	40 CFR Part 136.3
TCLP Leachates	Glass	200 mL for 8270 100 mL for 8081 100mL for 8141	Cool, ≤ 6°C	7 Days from the start of the leach	SW-846 1311
SPLP Leachates	Glass	1000 mL	Cool, ≤ 6°C	7 Days from the start of the leach	SW-846 1312

[1] Exclusive of analysis.

[2] Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require a 1 week hold time for method 8082 and 8082A. The states of California, South Carolina, Pennsylvania, and Connecticut require a 1 week hold time.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.3 Method Blank (MB)

9.3.1 One method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

9.3.2 The method blank for batches of aqueous samples for Large Volume Injection (prep method 3510C_LVI) consists of 250mL of reagent water free of any of the analyte(s) of interest.

9.3.3 The method blank for batches of aqueous samples for all other methods consists of 1 L of reagent water free of any of the analyte(s) of interest.

9.3.4 The method blank for batches of TCLP leachates for methods 8081 and 8141 consists of 100 mL of leach fluid.

9.3.5 The method blank for batches of TCLP leachates for method 8270 consists of 200 mL of leach fluid.

9.3.6 The method blank for batches of SPLP leachates consists of 1 L of leach fluid.

9.4 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

9.4.1 At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

9.4.2 The LCS for batches of aqueous samples for Large Volume Injection (prep method 3510C_LVI) consists of 250mL of reagent water to which the analyte(s) of interest are added at known concentrations.

9.4.3 For aqueous sample batches for all other methods, the LCS consists of 1 L of reagent water to which the analyte(s) of interest are added at known concentration.

9.4.4 For methods 8081 and 8141 TCLP leachates, the LCS consists of 100 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

9.4.5 For method 8270 TCLP leachates, the LCS consists of 200 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

9.4.6 For SPLP leachates, the LCS consists of 1 L of leach fluid to which the analyte(s) of interest are added at known concentration.

9.4.7 Method 608, 614, 610 requires a LCS at a 10% frequency. In other words one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

9.4.8 Method AK102 requires LCS and a LCSD for every batch for every spike compound.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.5.2 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared unless Method Comments indicate otherwise. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

9.5.3 Method 608, 610, and 614 requires one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples. If there is insufficient sample volume for matrix spikes, then a LCSD must be performed.

9.5.4 Method NWTPH-Dx requires a matrix spike and a matrix spike duplicate for every 10 samples. If insufficient sample volume is available for MS/MSD, a NCM must be written and a LCS and LCSD must be performed for every 10 samples.

9.6 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.4 Critical Procedural Considerations

10.4.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.4.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other separatory funnel than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.5 Assemble and clean the glassware immediately before use.

NOTE: Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

10.5.1 Place a stopcock in each separatory funnel. For 1-liter extractions use a 2000 mL separatory funnel. For 250 mL, 200 mL and 100 mL extractions, use a 500 mL separatory funnel. Place a stopper for each separatory funnel on a clean sheet of aluminum foil that is marked with individual positions for each stopper. This is done to prevent cross-contamination.

NOTE: Samples logged with method 3510_LVI are for Large Volume Injection methods and require 250 mL initial volumes. Samples logged for 8270 with a TCLP pre-prep require 200mL initial volumes. Samples logged for 8081 and 8141 with a TCLP pre-prep require 100 mL initial volumes.

10.5.2 For each separatory funnel, plug a glass funnel with baked glass wool and add baked sodium sulfate. Place the funnel on a media bottle and place the media bottle below the separatory funnel.

10.5.3 Rinse each separatory funnel once with methylene chloride. Be sure that all surfaces come into contact with the solvent. Drain the methylene chloride into the media bottle through the sodium sulfate.

10.5.4 Rinse the sodium sulfate with additional methylene chloride if the first rinse did not completely saturate the sodium sulfate.

10.5.5 Allow the methylene chloride to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional methylene chloride to the rinse if necessary.

10.5.6 Discard the methylene chloride.

10.5.7 Label each media bottle with the sample ID or batch QC ID.

10.6 Prepare LCS and Method Blank Samples

NOTE: For SW-846 methods if there is not a MS/MSD pair in the batch then perform a LCS/LCSD. Methods 608, 610, and 614 require a LCS and LCSD in batches of 11 samples or more or if there are no Matrix Spikes in batches of 10 or less.

10.6.1 For aqueous sample batches logged for Large Volume Injection, (3510_LVI), pour 250 mL of reagent water into the separatory funnels marked for the LCSs and the MB.

10.6.2 For all other aqueous sample batches, pour 1 liter of reagent water into the separatory funnels marked for the LCSs and the MB.

10.6.3 For 8270 TCLP leachates, use a 250 mL or 500 mL Class A graduated cylinder to measure out 200 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.4 For 8081 and 8141 TCLP leachates, use a 100 mL or 250 mL Class A graduated cylinder to measure out 100 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.5 For SPLP leachates, use a 1000 mL Class A graduated cylinder to measure out 1000 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.7 Measure the initial sample pH of the samples.

10.7.1 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.

10.7.2 If the sample is logged for AK102_103, Okla_DRO, or NWTPH_Dx the samples should have been field preserved. See Section 8. If the samples are not preserved, an NCM should be written.

10.8 Aliquot the samples

10.8.1 For 8270 TCLP leachates, use a 250 mL or 500 mL Class A graduated cylinder to measure out 200 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.8.2 For 8081 and 8141 TCLP leachates, use a 100 mL or 250 mL Class A graduated cylinder to measure out 100 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.8.3 For SPLP leachates, use a 1 Liter Class A graduated cylinder to measure out 1000 mL of the leachate. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.8.4 For water samples, it should be noted that TestAmerica Denver routinely aliquots gravimetrically. This is done to prevent cross-contamination due to volumetric glassware and to provide a more accurate initial volume measurement. However, some clients and regulatory programs require the laboratory to aliquot samples volumetrically. The Method Comments and QASs must be read before samples are aliquotted to check for this requirement. If samples are to be aliquotted volumetrically, use Class A volumetric glassware only and proceed to Section 10.8.6

10.8.5 Weigh the bottle (250 mL amber bottles for 3510C_LVI or 1000 mL amber bottles for all other aqueous samples) and record the gross weight to the nearest gram. If there is any indication that the sample's density is not 1 g = 1 mL, then measure

the density of the sample using a calibrated pipette and an analytical balance. The weight of the sample extraction will be corrected for the density later. See Section 11 for the calculation. For example, normally a 1 liter bottle weighs 500 g when empty and when filled completely can only hold 1060 mL, therefore a full bottle weighing more than 1560 g is an indication that either the sample density is greater than 1g or the sample bottle contains a lot of sediment. Document any sample with a density greater than 1 g in an NCM.

10.8.6 Inspect the samples for large amounts of sediment that may interfere with the extraction of the sample by causing excessive emulsions or clogging the stop-cock.

10.8.6.1 If the sample contains so much sediment that the entire sample volume cannot be extracted, decant the sample into the separatory funnel (or a 1 L graduated cylinder if volumetric aliquotting is required), careful not to transfer the sediment. Write a NCM to document the sediment and that it prevented the entire sample volume from being extracted and the sample container from being solvent rinsed. This is considered a deviation and must be documented in a NCM.

10.8.6.2 If the sample does not contain a significant amount of sediment, then the entire sample volume will be used in the extraction.

10.8.6.3 For the 600 method series: if there is no more than an inch of sediment in the bottom of the sample bottle, shake the sample well and determine if the sediment resettles in approximately 1 minute. If not, the density of the sediment is likely to be low enough to stay suspended and not block the sidearm.

10.8.6.4 For the 600 method series: if the density of the sediment is high and likely to cause a problem in the extraction or if there is more than an inch of sediment contact the PM so that the client's input can be obtained. Not extracting the entire sample and rinsing the bottle with the extraction solvent is a method deviation. If the client concurs that the sample can be decanted write an NCM to describe the deviation from the procedure.

10.8.7 Place the sample containers in front of the separatory funnel labeled for that sample. A second analyst should then check the labels to make sure the correct sample is being extracted. This check is documented in the Organic Extraction Checklist (WI-DV-0009)

10.9 If volumetric aliquotting is required, transfer the entire sample into a Class A graduated cylinder and record the volume on the benchsheet. If the sample bottle contains more than 1000 mL, a 100 mL Class A graduated cylinder can be used to complete the measurement. The entire sample volume must be used. Record the volume to the nearest 10 mL. Then pour the sample into the labeled separatory funnel. Place the used

graduated cylinder in front of the appropriate separatory funnel so it can be solvent rinsed later.

NOTE: A 1000 mL Class A graduated cylinder is not accurate enough to measure to the nearest 1 mL. Therefore all samples that are aliquoted using a 1000 mL Class A graduated cylinder will have the initial volume recorded to the nearest 10 mL. This accuracy is sufficient.

10.10 If volumetric aliquotting is not required, pour the sample directly into the separatory funnel. Place the empty sample container in front of the appropriate separatory funnel so it can be solvent rinsed.

10.11 Add Surrogates to All Field Samples and QC Samples

10.11.1 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch Reference work instruction WI-DV-0009 to determine the appropriate standard and the appropriate volume required.

10.11.2 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.11.3 Add the appropriate volume of the appropriate working surrogate standard to the separatory funnel for each sample, MB, LCS, and MS/MSD. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-0009 to determine the appropriate standard and the appropriate volume required.

10.12 Add Spikes to all LCS's and MS/MSDs

10.12.1 Add the appropriate volume of the appropriate working spike standard to the separatory funnels for the MS/MSD, LCS and/or LCSD samples. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-0009 to determine the appropriate standard and the appropriate volume required.

10.13 Add approximately 6g (1 teaspoon) of NaCl to all samples and all QC samples. This is done to give the reagent water used in the MBs and LCSs some ionic strength to more closely mimic the matrix of actual water samples and to aide in the extraction of the more polar target compounds. Record the lot number of the sodium chloride on the bench sheet.

NOTE: Per the South Carolina QAS, do NOT add NaCl to South Carolina samples or associated QC. South Carolina samples should be prepared separately from other samples in order to meet this requirement.

10.14 Adjust pH of Field Samples and QC Samples

Adjust the sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid (or 1 M hydrochloric acid for Methods AK102, Okla_DRO and NWTPH_Dx) or 10 N sodium hydroxide, as necessary. Record the adjusted pH and the lot number of the acid or base on the bench sheet. For TCLP leachates by method 8270, usually 1 mL of 1:1 sulfuric acid is sufficient.

NOTE: TCLP Leachates may have pH of < 5. In those cases, the pH should be adjusted per the table below.

Method	Initial Extraction pH	Secondary Extraction pH
All 8270 methods *except* SIM.	1 – 2	If samples are TCLP leachates extract at 14. If samples are water extract at 11 - 12
All 8270 SIM methods	As Received	None
All 8081, 8082 and 608 methods.	5 - 9	None
All 8141 and 614 methods	5-8	None
All 8015 methods	As Received	None
All 8310 and 610 methods	As Received	None
AK102_103 Okla_DRO NWTPH_Dx	If samples are preserved between pH 1 – 2, then acidify the MB and LCS. Otherwise extract as received and document insufficient preservation in an NCM.	None

10.15 For 1 Liter samples, add 60 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. For 250 mL or smaller samples, add 30 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. Cap the container and shake gently to rinse all internal surfaces of the bottle. Pour the methylene chloride from the sample container into the appropriate separatory funnel. If a graduated cylinder was used to aliquot volumetrically, rinse the cylinder and add that rinse to the separatory funnel as well. Record the lot number of the methylene chloride on the bench sheet. If the sample contained significant sediment and the entire sample contents could not be extracted, do not rinse the empty sample container, but instead add the solvent directly to the separatory funnel. If the solvent rinse of the sample container cannot be performed, prepare a NCM.

10.16 For water samples that were aliquotted gravimetrically, reweigh the bottle and calculate the initial sample volume by subtracting the empty bottles weight from the full bottles weight, assuming a density of 1 g = 1 mL. If there is any indication that the samples density is not 1 g = 1 mL then measure the density of the sample and correct the calculated initial volume accordingly using the formula in Section 11. Document abnormal sample density in an NCM. For example, normally a 1 liter bottle when filled completely can only hold 1060 mL, therefore an initial volume greater than 1060mL is an indication that the density is not 1 g. Document any sample with a density greater than 1 g in an NCM.

10.17 If the initial volume is less than 80% of the nominal volume, the sample reporting limits and method detection limits will be elevated substantially. Document this in a NCM.

10.18 Stopper and rotate the separatory funnel for 3 minutes with periodic venting to release excess pressure. Document the extraction date and time on the benchsheet.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken a few seconds. Vent into hood away from people and other samples. A face shield or goggles must be worn during venting.

10.19 Allow the organic layer to separate from the water phase for at least 5 minutes or until complete visible separation has been achieved. This can take up to 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, use mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, pouring the solvent layer and emulsion back through the top of the separatory funnel (pour-back), or centrifugation. The emulsion could also be filtered through the glass funnel by adding additional sodium sulfate to remove all water in the emulsion. This technique should only be used after other techniques have failed to make complete phase separation and only after the last shake.

NOTE 1: If an emulsion forms, the analyst does not have to wait a complete 5 minutes before attempting to break the emulsion with pour-backs and centrifuge. Start employing the mechanical techniques right away to achieve phase separation.

NOTE 2: As much as 15 to 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

10.20 Drain the lower methylene chloride layer into the sodium sulfate filled glass funnel. Allow the methylene chloride to drain completely into the media bottle. Rinse the sodium sulfate with a small amount of methylene chloride to ensure that all compounds of interest are collected in the media bottle. Record the lot number of the sodium sulfate on the bench sheet. If the sodium sulfate becomes saturated with water, add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

10.21 Repeat the extraction two more times for a total of 3 extractions. Collect all three methylene chloride extracts in the same media bottle. For the 2nd and 3rd extractions it is not necessary to wait 5 minutes to allow the solvent to separate from the water; a 3 minute wait time should be sufficient.

10.22 For the base/neutral and acid extractable method 8270, adjust the pH of the samples according to chart in Section 10.14. For 8270 TCLP leachates an excess of base is required to effectively extract pyridine, therefore at least 7 mL of base should be used to ensure the pH is 14. Then extract the sample 3 more times. For these extractions, it is not necessary to wait 5 minutes to allow the solvent to separate from the water; a 3 minute wait time should be sufficient.

NOTE: For 8270 water extractions please note that typically 2 mL of acid is needed to achieve a pH 1-2; 5 mL of base is typically required to achieve the pH of 11-12.

10.23 Cap the media bottle with a Teflon-lined cap or aluminum foil and submit for concentration and possible clean-up steps.

10.24 Dispose of the solvent-saturated water remaining in the separatory funnel in the appropriate waste container. See Section 14.

10.25 Initial weights and volumes of samples are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-0009).

10.26 Troubleshooting

10.26.1 If the sample appears very dark or viscous or in any way un-like water, stop and test the sample's miscibility before attempting to extract the sample by this procedure. Place a few milliliters of sample in a vial with methylene chloride. Cap and shake. If the sample is miscible in methylene chloride, the sample should be re-logged as a waste matrix with a prep method of 3580A.

10.27 Maintenance

10.27.1 Approximately every 6 months, the centrifuge should be lubricated.

10.27.2 Contact the Facilities Manager immediately if the rotator is observed to be making un-familiar noises or rotating in a "jerking" manner.

11.0 Data Analysis and Calculations

11.1 Initial Volume calculation

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g/mL)}$$

11.2 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on DV-F-0045 Organic Extraction Department Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the

IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Solid waste/sodium sulfate – Waste Stream D

14.2.3 Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.4 Acidic aqueous sample waste saturated with methylene chloride – Waste Stream Y.

14.2.5 Neutral aqueous sample waste saturated with methylene chloride – Waste Stream X or Waste Stream Y.

14.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for

Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, Organochlorine Pesticides and PCBs.

15.3 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 610, Polynuclear Aromatic Hydrocarbons.

15.4 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 614, Organophosphorous Pesticides.

15.5 Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

15.6 Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

15.7 NWTPH-Dx "Semi-Volatile Petroleum Products Method for Soil and Water.

15.8 Oklahoma Department of Environmental Quality Methods 8000/8100 (Modified) Diesel Range Organics (DRO) Revision 4.1 Date 10/22/97

16.0 Modifications:

16.1 Modifications from SW-846 Method 3510C

16.1.1 Section 7.1 of the method calls for initial sample volume to be determined volumetrically either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

16.1.2 Section 7.5 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.1.3 Section 7.6 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.1.4 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.1.5 The source method calls for samples to be extracted for method 8141 at the pH they are received. This procedure calls for the extraction to be performed at a pH between 5 and 8. This is done per guidelines found in Section 2 and Section 8 of SW-846 8141B.

16.2 Modifications from 40 CFR Method 608 and 610

16.2.1 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.2.2 Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.2.3 Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.2.4 Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL methylene chloride aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

16.2.5 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.3 Modifications from 40 CFR Method 614

16.3.1 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.3.2 Section 10.2 of the method calls for the extraction to be performed with at 15% v/v methylene chloride in hexane solvent. This procedure uses methylene chloride for the extraction. SOP DV-OP-0007 calls for the methylene chloride extract to be concentrated and exchanged to hexane.

16.3.3 Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.3.4 Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.3.5 Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL solvent aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

16.3.6 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.4 Modifications from Method AK 102

16.4.1 Section 9.1.1.1 of the method calls for using no more than 1 liter of sample and to determine the volume either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

16.4.2 Section 9.1.1.6 of the method says to allow the water and solvent layers to separate for approximately 10 minutes. This SOP calls for the allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.4.3 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.5 Modifications from Method NWTPH-Dx

16.5.1 The method calls for determining the initial volume of the sample my marking the meniscus on the bottle and later determining the volume of tap water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

16.5.2 The method calls for shaking the separatory funnel for one minute. This SOP calls for the separatory funnel to be shaken for at least three minutes.

16.5.3 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.6 Modifications from Oklahoma DRO

16.6.1 The method calls for aliquotting 800 mL to 900 mL of the sample volumetrically. This SOP calls for the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via

glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

16.6.2 The method calls for extracting using 50 mL of solvent. This SOP calls for the extraction to be done using at least 60 mL of solvent.

16.6.3 The method calls for shaking the separatory funnel for two minutes. This SOP calls for the separatory funnel to be shaken for at least three minutes.

16.6.4 The method calls for a method blank and LCS to be analyzed every 10 samples. This SOP calls for a method blank and LCS to be analyzed every batch of 20 samples.

16.6.5 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

17.0 Attachments

Table 1. Determinative Methods Using Separatory Funnel Extractions

18.0 Revision History

- **Revision 18.0, October 31, 2018**
  - Updated the last sentence in the copyrighted information section.
  - Updated MDL SOP number.
  - Added note to section 10.13 per South Carolina requirement.
  - Corrected references to WI-DV-009 to correct document ID, WI-DV-0009 throughout.
- **Revision 17.0, December 31, 2017**
  - Adjusted the volumes in the note in section 10.22 to reflect current practice.
- **Revision 16.0, August 16, 2017**
  - Updated and rearranged section 10.8.6 – 10.15 to account for adjustment of procedure. Surrogate and spike standards will now be added to samples in extraction vessel, rather than sample container.
- **Revision 15.0, June 30, 2017**
  - Paragraph with reference to QAM for basic definitions was added to Section 3.0
  - Removed previous Section 9.2 Initial Performance Studies, renumbered remaining Sections
  - Updated current Section 9.3.1 – removed “At least” for the one MB requirement
  - Added DoD requirements regarding MS/MSD (LCSD) to Section 9.5.2
  - Added current Section 10.2 regarding NCM documentation
  - The note was added to section 10.22 indicating the typical amounts of acid or base needed to achieve dual pHs for 8270 water extractions.
  - Added Section 11.2 regarding data review

- Added current Section 12.2 LOQV information
- Fixed numbering and section references throughout SOP
- Removed Method 610 from Table 1. Lab no longer performing method.

- **Revision 14.0, June 30, 2016**
  - Added Section 3.6 – definition of reagent water
  - Revised the table in Section 8 to reflect the nominal leachate volume for method 8141.
  - Updated sections 9.4.4, 9.5.4, 10.4.1, and 10.5.4 to include 8141 TCLP.
  - Added Section 10.2 - recording of support equipment IDs
  - Added note to Section 10.4 regarding the rotation of glassware
  - Added reference to method 8141 TCLP leachates to Section 10.7.2
  - Removed references to preparation of Wyoming Leachates throughout. Lab no longer performs Wyoming Leach method
  - Added Sections 10.7.6.3 and 10.7.6.4 to provide guidance for 600 method series in relation to sediment and decanting issues.
  - Updated Section 12 to be consistent with other SOPs
  - Renumbered paragraphs throughout due to the removal of the WY Leachate prep

- **Revision 13, August 31, 2015**
  - Annual Technical Review.
  - Removed the Notes from Section 2 and Section 10.9 regarding South Carolina. The laboratory no longer holds South Carolina certification for this method.
  - Added detail to Section 10.12 and 10.20 on how much acid and base is normally required to adjust the pH of leachates for method 8270.

- **Revision 12.0, August 31, 2014**
  - Revised Section 2 to remove references to initial volume. The procedure is used on waters and leachates with a variety of initial volumes. That detail is documented later in the procedure and was therefore removed from the summary found in Section 2.
  - Added a comment to Section 9.1.2 that states: “This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated.”
  - Section 9 was revised to remove Acceptance Criteria and Corrective Action details. This information is found in the analytical procedures.
  - Removed the Note following Section 10.4.2 that instructs the analyst to check the samples for sodium thiosulfate preservation. TestAmerica Denver does not analyze drinking water samples by this procedure and therefore this preservation is not needed.
  - All references to 8270 by LVI were removed. TestAmerica Denver does not extract samples by this procedure for 8270 by LVI. Instead the samples are extracted by 3520C under DV-OP-0008.
  - The table in Section 10.12 was revised to make it easier to read and locate the correct Method.
  - Troubleshooting and Maintenance sections were added per DoD QSM 5.0 requirements.

- **Revision 11.0, August 19, 2013**
  - Added statement to Section 2.0 that LVI must not be used on SC samples

- **Revision 10.0, May 14, 2013**
  - The procedure was revised to instruct the analyst to allow the organic and aqueous phases to separate for a minimum of 5 minutes after the first extraction and 3 minutes after subsequent extractions.
  - The procedure was revised to increase the amount of sodium chloride added to samples and QC from 3g to 6g.
  - Section 5 was revised to include the hazards of glasswool and to instruct the analysts to handle it only in a fumehood.
  - Section 8 was revised to change the hold-time calculation for leachates from the start of the leaching procedure instead of the completion of the leaching procedure. This was done to ensure the holding times are contiguous.
  - Section 10.13 was revised to instruct the analyst to extract 250 mL to 100 mL samples with 30 mL of solvent instead of 15 mL of solvent. This was done to increase extraction efficiency while still reducing solvent usage.
  - Sections 2.0, 9.1 and 10.1 were updated to reflect current practice.

- **Revision 9.0, January 15, 2013**
  - Section 10.9 was updated to include note to eliminate use of salt in South Carolina samples.

- **Revision 8.0, September 25, 2012**
  - This procedure was updated to include instructions on how to extract 8270 water samples for Large Volume Injection.

- **Revision 7.0, January 31, 2012**
  - Annual Technical Review
  - Updated Section 6.2 to describe the requirements for computer software and hardware
  - Updated Section 7.0 to describe requirements for Reagents and Standards.
  - Updated Section 8.0 to state PCBs by method 8082 have no holding time as per SW-846 Update 4 and that samples for analysis by NW-TPH have a 7 day hold time, even if acid preserved.
  - Updated Section 9.1.4 and Section 10.1 to accurately describe the NCM notification system.
  - Updated Section 10.4 and 10.6 to state the appropriate size of the graduated cylinders to be used to measure out 100 mL and 200 mL of leachate.
  - Updated Sections 10.6.6 and 10.14 to give guidance to the analyst when a density check of a sample is required.
  - Updated Section 10.9 to give more detail on how much sodium chloride should be added to the samples.
  - Updated Section 16 to include the method modification of the sodium chloride addition.
  - Updated Table 1 to reflect the current analytical SOPs.
  - Corrected grammatical and formatting errors

- **Revision 6.0 dated January 10, 2011**

- Added note to Section 6 that sodium sulfate should be stored in tightly closed container.
- Revised Section 7 to reference DV-OP-00020 for information about surrogate and spike standards.
- Corrected Section 7.1 to indicate that the reagent water should be 18 to 18.2 Mohm/cm.
- Revised procedure to include details on the extraction of Wyoming Leachates.
- Added references to methods NWTPH-Dx, and Oklahoma DRO.
- Added Section 6.2 computer software and hardware.
- Section 8 was revised to give more detail on the preservation and hold times for methods AK102, AK103, NWTPH-Dx, and Oklahoma DRO.
- Revised Section 9 to include more detail on QC requirements for methods AK102_103, NWTPH-Dx, and Oklahoma DRO.
- Revised Section 10 to clarify that when 1 liter graduated cylinders are used to measure the initial volume of the water samples, that the volume should be recorded to the nearest 10 mL.
- Revised Section 10 to instruct that if samples for methods AK102_103, NWTPH-Dx, and Oklahoma DRO are received preserved, then the MB and the LCS samples should also be acidified with HCl. Otherwise the samples are extracted as received.
- Revised Section 16 to include more detail on modification from methods AK102_103, NWTPH-Dx, and Oklahoma DRO
- Revised the procedure to call for the 2nd fraction of 8270 TCLP leachates to be extracted at a pH of 14 instead of the pH 11 to 12 used in water samples. This was done to help the recovery of pyridine.

Earlier revision histories have been archived and are available upon request.

TABLE 1.

Determinative Methods Using Separatory Funnel Extractions

Method Description	*Determinative Method*	*SOP*
Diesel Range Organics & Jet Fuels	SW-846 8015, California LUFT Method, Alaska Methods AK102 & AK103 SW-846 8015C	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B EPA Method 608	DV-GC-0020 DV-GC-0016
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A EPA Method 608	DV-GC-0021 DV-GC-0016
Organophosphorus Pesticides	SW-846 8141A, & EPA Method 614	DV-GC-0017
Polynuclear Aromatic Hydrocarbons (PAH)	SW-846 8310	DV-LC-0009
Semi-volatiles by GC/MS	SW-846 8270 SW-846 8270D	DV-MS-0011 DV-MS-0012
PAH by GC/MS SIM	SW-846 8270	DV-MS-0002

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Concentration and Clean-up of Organic Extracts [SW-846, 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, ASTM Method D7065-11, and EPA 600 Series Methods]

Approvals (Signature/Date):

Amelia Byl 11/30/2018
Amelia Byl — Date
Technical Specialist

Doug Gomer 11/30/18
Doug Gomer — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 11/30/18
Roxanne Sullivan — Date
Quality Assurance Manager

Richard Clinkscales 11/30/18
Richard Clinkscales — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This standard operating procedure (SOP) provides instructions for the concentration, and if necessary, cleanup, of solvent extracts of organic compounds from water samples, soil samples, TCLP leachates, and SPLP leachates. This SOP is based on SW-846 Methods 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3630C, 3660B, 3665A, ASTM Method D7065-11, and EPA 600 Series methods.

1.2 The determinative methods and extraction methods used in conjunction with this procedure are listed in Attachment 1.

1.3 This procedure does not include the extraction steps. See the following SOPs for the applicable extraction procedures:

DV-OP-0006: Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series

DV-OP-0008: Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C, and Method 625 and ASTM Method D7065-11

DV-OP-0010: Soxhlet Extraction of Solid Samples, SW-846 3540C

DV-OP-0015: Microwave Extraction of Solid Samples, SW-846 3546

DV-OP-0016: Ultrasonic Extraction of Solid Samples, SW-846 3550B and 3550C

DV-OP-0021: Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS

NOTE: This SOP does not include the concentration steps of extracts for Herbicides by method 8151A or Herbicides by method 8321. See DV-OP-0011 and DV-LC-0014 respectively.

2.0 Summary of Method

Sample extracts are concentrated to a specific final volume using an S-EVAP, N-EVAP, or Turbo-Vap. Some methods require a solvent exchange. If necessary, various clean-up techniques are performed before the extract is sent for analysis.

3.0 Definitions

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time:** The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch:** A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. See WI-DV-0032

3.4 **Quality Assurance Summary (QAS):** Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/Method Comments field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, more than one layer of extract, etc.) must be documented.

4.3 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.4 Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by 8270D_SIM_LL must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, stem-less glass funnels, and Snyder columns will be clearly marked and segregated for this purpose.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the

method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 In order to limit the emission of methylene chloride, TestAmerica Denver uses a solvent recovery system. The system condenses and collects methylene chloride that has been evaporated off the sample extracts while on the S-EVAP.

5.1.1.1 Each analyst must inspect the system before using it to ensure the collection tubes are in good condition, the in-process tanks are not full, and the chiller is operating correctly.

5.1.1.2 While concentrating methylene chloride or methylene chloride / acetone extracts on the S-Evap, the analyst must check the level of the solvent collected in the in-process tanks at a frequency to ensure the tank will not be overfilled. A tank will not be filled more than 90%. The analyst may use a timer set at 30 minute intervals to help remind the analyst to check the level of the solvent collected in the in-process tanks.

5.1.1.3 The solvent recovery system will never be used for the collection of ether due to the potential danger to analysts if the system were to fail during operation.

5.1.2 Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Acetonitrile	Flammable Irritant Poison	40 ppm TWA	Exposure may cause cyanide poisoning resulting in reddening of the skin and eyes and pupil dilation. Effects of overexposure are often delayed due to the slow formation of cyanide ions in the body. May cause nose and throat irritation, flushing of the face, tightening of the chest. Also may cause headache, nausea, abdominal pain, convulsions, shock.
Hexane	Flammable	50 ppm TWA	Causes irritation to eyes, skin and respiratory tract. Aspiration hazard if swallowed. Can

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
	Irritant		enter lungs and cause damage. May cause nervous system effects. Breathing vapors may cause drowsiness and dizziness. Causes redness and pain to the skin and eyes.
Methanol	Flammable Irritant Poison	200 ppm TWA	Methanol evaporates at room temperature. Inhalation, ingestion and/or eye and skin contact can all possibly cause light-headedness, nausea, headache, and drowsiness. Prolonged exposure can lead to permanent blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Mercury	Corrosive Irritant Highly Toxic	0.05 mg/m^3 TWA	May be fatal if inhaled. May cause respiratory tract irritation. May be harmful if absorbed through skin. May cause skin irritation.
Methylene Chloride	Irritant Carcinogen	25 ppm TWA 125 ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

NOTE 1: All glassware used in this procedure is cleaned following SOP# DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use. Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

NOTE 2: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA method 8270D_SIM_LL and PAHs by method 8270C_SIM_LL must be concentrated in glassware designated for that method. K-D flasks, glass funnels, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

6.1 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.2 Kuderna-Danish (K-D) flasks.

6.3 Concentrator tubes for K-D flasks, un-graduated, approximately 10 mL.

6.4 Concentrator tubes for K-D flasks, graduated at 1mL, calibration checked before use following the steps detailed in DV-QA-0008.

6.5 Snyder columns, 3-ball with ground glass joints at top and bottom

6.6 Manual, adjustable positive-displacement pipette and bottle-top re-pipettor, used to dispense 1 to 20 mL. Calibration is checked following the steps detailed in DV-QA-0008.

6.7 Extract Storage Vials – variety of sizes, clear and amber

6.8 Pasteur pipettes – 6 inch and 9 inch in length.

6.9 Stem-less glass funnels

6.10 Glass wool, baked at 400°C for four hours.

6.11 Boiling Chips – contaminant free, approximately 10/40 mesh Teflon®, PTFE. For concentrating extracts to a final volume greater than 1mL.

6.12 Boiling Chips – contaminant free, carborundum #12 granules, for concentrating extracts to a 1mL final volume. These boiling chips are sufficiently small as to not add any error to the 1mL final volume.

6.13 Solvent Recovery System – includes re-circulating chiller, set no higher than 12°C, cooling condensers, Teflon® PTFE tubing and In-Process Tanks with quick-connect attachments

6.14 S-Evap, thermostat controlled water bath

6.15 N-Evap, thermostat controlled water bath with regulated nitrogen supply

6.16 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Methylene Chloride

Each lot of solvent is tested following CA-Q-S-001 or before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, that have not been previously tested per CA-Q-S-001, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.2 Hexane

For solvents packaged in bottles, each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.3 Methanol, HPLC Grade

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Acetone

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.5 Acetonitrile

Each lot of solvent is tested following CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.6 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400°C oven for at least four hours.

7.7 Sulfuric Acid, Concentrated

For use in PCB extract clean-up.

7.8 Florisil Solution, (FlorisilSol)

Add 900mL of hexane to a Class A graduated cylinder. Add 100 mL of Acetone to the same graduated cylinder for a final volume of 1000 mL. Pour the mixture into a 1 L amber bottle.

7.9 Florisil Cartridges,

Purchased ready to use. 1000 mg in 6 mL tube. Stored in a desiccator after opening. Restek part number 24034 or equivalent.

7.10 Anhydrous Silica Gel, 60-100 mesh, (SiGel60-100UA)

Sigma Aldrich part number 23799-1KG or equivalent

7.11 Activated Anhydrous Silica Gel, 60-100 mesh, (Active SilGel)

Bake Silica Gel from Section 7.10 above at 400°C for at least 4 hours. Store in a desiccator.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample extracts waiting to be concentrated are stored refrigerated at 0°C - 6°C in glass bottles or flasks and capped with Teflon-lined lids or aluminum foil. Final sample extracts are stored in glass vials with Teflon-lined lids. See Table 3 for details on storage vial types. Final concentrated extracts are stored refrigerated at 0°C - 6°C. Extracts have a holding time of 40 days from the date of extraction to the date of analysis.

8.2 All sample extracts, before or after concentration, are stored separately from standards.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs.* This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of reagent water, and for batches of soil samples, consists of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank for batches of TCLP and SPLP leachates consists of leach fluid. The method blank is processed and analyzed just as if it were a field sample.

9.5 Laboratory Control Sample (LCS)

9.5.1 At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at known concentration. For soil sample batches, the LCS consists of Ottawa sand to which the analyte(s) of interest are added at a known concentration. For TCLP and SPLP leachates, the LCS consists of leach fluid to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

9.5.2 EPA Methods 608, 610, 614, and 625 require a LCS at a 10% frequency. In other words, one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.6.2 EPA Methods 608, 610, 614, and 625 require one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples.

9.6.3 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Critical Procedural Considerations

NOTE: Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

10.3.1 As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.3.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces should be

cleaned or disposed of before coming into contact with the sample.

10.3.3 According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to WI-DV-0009 for the appropriate final solvents and final volumes.

10.4 Refer to WI-DV-0009 to determine if the extract is to be concentrated by the Kuderna-Danish / N-Evap method described in Section 10.5 and 10.6, or the Turbo-Vap method described in Section 10.6.6

10.5 Concentration by the Kuderna-Danish Method (S-evap)

10.5.1 Refer to WI-DV-0009. If the extract is to be concentrated to a 1 mL final volume, use a 1 mL graduated concentrator tube. For extracts that are to be concentrated to any other final volume, use an un-graduated concentrator tube.

10.5.2 Assemble the Kuderna-Danish concentrator by attaching the appropriate concentrator tube to the 500 mL K-D flask with a clip. Make sure the attachment is firm at the joint. While wearing cut-resistant gloves, tighten the joint with your fingertips and thumb. Do NOT over-tighten. Refer to Attachment 3 for configuration of the Kuderna-Danish concentrator.

NOTE: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by method 8270D_SIM_LL and PAHs by method 8270C_SIM_LL must be concentrated in glassware designated for those methods. K-D flasks, concentrator tubes, and Snyder columns will be clearly marked and segregated for this purpose.

10.5.3 Rinse the apparatus with methylene chloride. Discard the rinse solvent into the appropriate waste container. Care should be taken to ensure all surfaces of the glass are coated with solvent.

10.5.4 If the extract is to be concentrated to a 1 mL final volume, add 2-3 carborundum granules to the K-D concentrator. If the extract is to be concentrated to a final volume greater than 1 mL, add 1-2 Teflon® boiling chips to each K-D concentrator.

10.5.5 If the sample extracts have not been filtered through sodium sulfate at the time of extraction, or if the sample extract have visible water, then the extracts must be dried at this point. Plug a glass funnel with baked glass wool and add approximately 1 teaspoon of baked sodium sulfate. Rinse the funnel and the sodium sulfate with methylene chloride and place it on top of the K-D. During the quantitative transfer in section 10.5.6 the extract will be filtered through the sodium sulfate.

NOTE 1: Glass wool dust is a carcinogen and therefore glass wool should only be handled in a hood to avoid inhaling any glass particles. Once covered with sodium sulfate, it can be removed from the hood.

NOTE 2: If the extract contains more water than can be easily removed by filtering through 1 teaspoon of sodium sulfate, either more sodium sulfate can be used or a solvent-rinsed separatory funnel can be used to separate the water out of the extract. A NCM should be prepared if this is necessary.

10.5.6 Quantitatively transfer the sample extract to the K-D flask. Transfer the sample label to the K-D flask. Perform a quantitative transfer of the extract by rinsing the sample extract container with methylene chloride and adding the rinse solvent to the K-D. If the extract is being filtered through sodium sulfate, be sure to rinse the sodium sulfate well to ensure no target compounds are left on the sodium sulfate. Allow the solvent to drain from the sodium sulfate into the K-D flask then discard the sodium sulfate.

10.5.7 Turn a three-ball Snyder column upside down and rinse with methylene chloride, then rinse the bottom joint with methylene chloride. Attach the Snyder column to the top of the K-D concentrator as shown in Attachment 3.

10.5.8 Place the K-D concentrator on a s-evap water bath so that the tip of the receiver tube is submerged. The water level should not reach the joint between the concentrator tube and the K-D flask. Refer to WI-DV-0009 for the correct water bath temperature. Record both the observed and the corrected temperature on the benchsheet.

10.5.9 For extracts that are methylene chloride or 50/50 methylene chloride/acetone, attach the solvent recovery system tube to the top of the Snyder column. At the appropriate rate of distillation, the balls will actively chatter but the chambers should not flood.

NOTE 1: For extracts for analysis for low-level NDMA by method 8270D_SIM_LL and PAHs by 8270C_SIM_LL, the solvent recovery system will not be used to avoid possible contamination.

NOTE 2: At this time, a timer may be set for 30 minute intervals as a reminder to check the in-process solvent tanks.

10.5.10 If the method does not require a solvent exchange, skip to Section 10.5.12. If the method requires a solvent exchange, continue on to Section 10.5.11.

10.5.11 If the method requires a solvent exchange at this time, detach the solvent recovery system tube from the top of the Snyder column and add the appropriate exchange solvent through the top of the Snyder column. The exchange solvent should be added when the extract has concentrated to a level that it forms a quarter-sized pool of solvent in the bottom of the K-D. Refer to WI-DV-0009 for details of exchange solvents and volumes. Mark the K-D flask and sample label to indicate the exchange has been performed. There is no need to re-attach the solvent recovery system at this time as the majority of the methylene chloride has already been evaporated and collected.

10.5.12 Continue to concentrate the sample on the s-evap water bath back down to 10-15 mL, or just below the K-D and concentrator tube joint. At this point the boiling sample is just barely splashing above the top of the receiver tube.

NOTE: It is very important not to concentrate to dryness as analytes will be lost. Some of the analyses, especially for 8270 and 8015, are especially temperature sensitive and the sample should be taken off the water bath as soon as possible to avoid losing analytes. The 8081 surrogate TCMX is fairly volatile and can be lost if the extract is allowed to concentrate too low either before or after hexane exchange. If the analyst has concerns that the extract might have concentrated too low, they should notify their supervisor and/or write a NCM.

10.5.13 Remove the K-D concentrator from the water bath. Rinse the Snyder column down with a minimal amount of solvent. If the extract was exchanged, use the exchange solvent to perform the rinse, otherwise use methylene chloride.

10.5.14 Allow the extract to cool to room temperature, about 10 minutes.

10.5.15 After the extract is allowed to cool, if the level of the extract is above the level of the concentrator tube joint, add a fresh boiling chip and return the K-D concentrator to the water bath.

10.5.16 After the extract is cool, remove the snyder column. Remove the clip holding the K-D flask and concentrator tube together. Use a Kim-wipe to dry the water off of the joint area so that water does not get into the extract. Remove the concentrator tube from the K-D flask and rinse the lower K-D flask joint into the concentrator tube with methylene chloride or the appropriate exchange solvent.

10.6 Nitrogen Evaporation (N-Evap) to Final Concentration.

10.6.1 N-evap needles should be cleaned weekly by soaking overnight in methylene chloride. This is documented in the N-evap needle log-book.

10.6.2 At the beginning of each shift, the N-evap needles should be wiped clean with a Kim-wipe soaked in methylene chloride to remove any potential contamination. If a needle comes in contact with an extract, then it needs to be cleaned before being used on the next extract.

10.6.3 Place the concentrator tube on the nitrogen evaporator. The temperature of the water bath should be at least 5°C below the boiling temperature of the solvent being evaporated (See Attachment 2). Lower the needle down to the sample so that a small dimple forms on the surface of the solvent. The stream of nitrogen should be gentle enough that it does not cause the extract to splash. Record both the observed and the actual temperature on the benchsheet.

10.6.4 During the course of the evaporation, rinse the sides of the concentrator tube with approximately 1 mL of clean solvent. The rinse should occur when the solvent gets close to the final volume. Concentrate the solvent to just below the

final volume and remove from the nitrogen evaporator.

10.6.5 Transfer the extract into the appropriate vial. Refer to WI-DV-0009 for the appropriate final volume and correct vial.

10.6.5.1 If the extracts are to have a final volume of 1 mL, they should be in 1 mL graduated concentrator tubes. Using a Pasteur pipette, or a solvent wash bottle, add the appropriate solvent to the tube until the extract meniscus reaches the 1 mL gradation. Then using the Pasteur pipette transfer the extract to a labeled 2 mL amber glass vial.

10.6.5.2 For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable positive-displacement pipette or bottle-top re-pipettor. Document the pipette ID used on the batch record. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract into the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

NOTE 1: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

NOTE 2: Some extracts might not concentrate down to the required final volume. If the extract is very dark and viscous, or an oil layer or precipitate starts to form, a higher final volume can be used. This should be documented in an NCM.

10.6.6 After the extract has been transferred to the appropriate vial, rinse the concentrator tube with methylene chloride before washing per DV-OP-0004. This is important to remove any residual contamination.

10.7 TurboVap Method

10.7.1 Turn on the TurboVap and adjust the water temperature to 40°C. Turn the nitrogen supply on. Record both the observed and the actual temperature on the benchsheet.

10.7.2 Switch the endpoint sensor to “Manual”.

10.7.3 Adjust the water bath level. The water level should be at least 1 inch above the extract level.

10.7.4 Turn on the nitrogen gas and adjust the gas pressure to approximately 12 psi. Lower pressure may be used if needed to prevent samples from splashing out of the TurboVap tubes.

10.7.5 Rinse the TurboVap tube with methylene chloride or the solvent the extract is in. Discard the waste.

10.7.6 Transfer the sample to the TurboVap tube. For 8141 soils extracted by soxhlet, dry the extract first by filtering through a funnel with baked sodium sulfate. Rinse the sample extract container with clean solvent and transfer to the TurboVap tube. Do not fill the TurboVap tubes over the fill line or approximately ¾ full.

10.7.7 Place the TurboVap tube into the TurboVap and turn on nitrogen to the position the tube is in.

10.7.8 Close the lid. You should be able to see the sample extracts swirling in the tubes.

NOTE: If the extract splashes when the nitrogen flow starts, transfer a portion of the extract back into the original extract container, or lower the gas pressure.

10.7.9 As the extract concentrates, transfer the remainder of the extract in to the appropriate Turbovap tube. Rinse the sample container with a few milliliters of methylene chloride or appropriate solvent and transfer to the Turbovap tube.

10.7.10 During the concentration rinse the Turbovap tube walls with a few milliliters of solvent 1 or 2 times.

10.7.11 If a solvent exchange is required, concentrate to about 5 mL and add the exchange solvent. After the exchange solvent is added, swirl the extract to make sure the extract is well mixed. Concentrate back down to slightly less than the appropriate volume. Refer to Attachment 3 for details of exchange solvents and final volumes.

10.7.12 Transfer the extract into the appropriate vial.

10.7.12.1 Currently, the TurboVap is only used to concentrate extracts with final volumes greater than 1 mL. Ask the supervisor for guidance if a project requires a 1 mL final volume by TurboVap.

10.7.12.2 For extracts with a final volume greater than 1 mL, the vials should be calibrated using the manual, adjustable pipette or bottle-top re-pipettor. Document the pipette ID used on the batch record. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract to the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

10.7.12.3 Rinse the Turbovap tube with methylene chloride 2-3 times before washing. Turbovap tubes are not baked. They are cleaned in

accordance with DV-OP-0004. If the Turbovap tubes need to be used again before they are dry, rinse with acetone to dry the Turbovap tube.

10.8 Cleanup Techniques

NOTE: If any sample in a batch requires a clean-up, the batch QC must also undergo the same clean-up technique.

10.8.1 Florisil Cartridge Cleanup

Florisil can be used to remove low-medium molecular weight polar hydrocarbon interfering compounds from pesticide extracts. The laboratory will use Florisil cleanups whenever water extracts have any color, whenever soil extracts have any color darker than a Post-It® Note, or whenever there is clear evidence of interferences, such as significant interfering peaks in the RT range for the target pesticide compounds or failing sample surrogate recoveries. Extracts that are to be analyzed for kepone will not be florisil cleaned, because florisil will remove kepone from the extract.

NOTE: Florisil cartridge performance checks are conducted for every lot of Florisil before use. Add 1.0 mL of the Florisil check solution described in Attachment 4 to a pre-rinsed Florisil cartridge. Following the procedure described below, load and elute the 1mL of check solution through the Florisil cartridge. Bring the final volume back down to 1.0 mL in hexane. The test sample must show 80-115 % recovery of the controlled analytes with < 5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. The non-controlled analytes will be monitored for problems, but do not have to pass the 80-115% limits. If the check fails, repeat the test. If the re-check fails, contact QA for guidance.

10.8.1.1 Clean the manifold and ports

Prior to each use, the top and underside of the manifold lid must be wiped down with hexane and a Kim-wipe to prevent any cross-contamination. The manifold ports must be left open and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to ensure it does not spread contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

10.8.1.2 Place one Florisil cartridge into the vacuum manifold for each extract. Make sure all valves are closed.

10.8.1.3 Add approximately 6 mL of hexane to each cartridge by filling the tube.

10.8.1.4 Slowly open the valves to allow a few drops of hexane to pass through, then close the valve and allow the hexane to soak the cartridge for at

least 5 minutes.

10.8.1.5 Slowly open the valves again and allow the hexane to drain through the cartridge but close the valve when the solvent level is right above the glass frit. Do not allow the cartridges to go dry. If cartridges go dry, repeat the conditioning step.

10.8.1.6 Remove the manifold top and place one clean, labeled 16 x 125 mm disposable glass test tube in each position for each of the samples. Replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

10.8.1.7 Add exactly 2.0 mL of the concentrated extract to the appropriate Florisil cartridge. Turn the valve to the on position.

10.8.1.8 Allow the extract to gravity drip through the cartridge. The flow through the cartridges should be drop-wise, not streaming.

10.8.1.9 Just before the extract level drops below the glass frit, fill the cartridge with (90:10) Florisil solution. Allow this to pass through the cartridge, then just before it falls below the glass frit again, fill the cartridge again with (90:10) Florisil solution.

10.8.1.10 Allow all of the 90:10 solution to gravity drip through the cartridges.

10.8.1.11 After visible solvent has been allowed to gravity drip through the cartridge, apply the vacuum to pull remaining solvent through cartridge, typically no more than 5 seconds.

NOTE: Do not use the vacuum to recover solvent from the cartridge before gravity drip is complete. Doing so could result in the interfering compounds that should be retained in the packing to come through into the cleaned extract.

10.8.1.12 Remove the tubes from the vacuum manifold and concentrate them back down to just below 2.0 mL on the nitrogen evaporator. Quantitatively transfer the extract to a 4mL vial that has been calibrated to hold 2.0 mL and bring the extracts up to the 2.0 mL calibration mark with hexane.

10.8.1.13 Discard the used cartridges.

10.8.2 Sulfur Removal

NOTE: This step is typically performed by the instrument analyst, as it is performed after extracts are concentrated to final volume.

Sulfur can be removed by one of three methods: mercury, copper, or

tetrabutylammonium sulfite (TBA), according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

10.8.2.1 Sulfur Removal with Elemental Mercury

NOTE: Use Mercury in a hood and sparingly in order to minimize exposure and disposal costs.

10.8.2.1.1 Transfer approximately 2 mL of sample extract into a clean Teflon-sealed vial.

10.8.2.1.2 Add one to three drops of mercury to the extract vial and seal.

10.8.2.1.3 Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

10.8.2.1.4 Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

10.8.2.1.5 If the mercury turns black, sulfur was present. Decant or pipette off the extract to a clean vial and repeat the procedure by adding one to three drops of fresh mercury. Do this until the mercury does not turn black.

10.8.2.1.6 If the extract is cloudy, filter the extract through a 1um disposable syringe filter.

10.8.2.1.7 Properly dispose of the mercury waste.

10.8.2.2 Sulfur Removal with Copper Powder

NOTE: This technique requires the copper powder to be very reactive, as demonstrated by a bright and shiny appearance. A pre-cleaned, activated copper may be purchased from a valid vendor. If manual preparation of reactive copper is performed, take care to remove all traces of acid in order to prevent degradation of some analytes.

10.8.2.2.1 Weigh out copper into a 20 mL VOA VIAL assuming two grams of copper needed per sample.

10.8.2.2.2 Remove oxides by treating with 10% nitric acid.

10.8.2.2.3 Rinse the copper with DI organic-free water three times to remove all traces of acid.

10.8.2.2.4 Rinse the copper with acetone and dry under a stream of nitrogen.

10.8.2.2.5 Add approximately 2 grams of the copper powder to a 2 mL vial with approximately 1ml of sample extract and shake vigorously on a mechanical shaker for at least one minute.

10.8.2.2.6 After phase separate, draw off extract and transfer to a clean vial.

10.8.3 Sulfuric Acid Cleanup

NOTE: This step is typically performed by the instrument analyst, as it is performed after extracts are concentrated to final volume.

10.8.3.1 Add 1 mL of concentrated sulfuric acid to approximately 2 mL of sample extract in a Teflon capped vial.

CAUTION: There must be no water or acetone present in the extract or the reaction may shatter the sample container.

10.8.3.2 Vortex for about 5 seconds and allow to settle. (Centrifuge if necessary)

10.8.3.3 Remove the sample extract (top layer) from the acid using a Pasteur pipette and transfer to a clean vial.

CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transferring any amount of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column

10.8.3.4 If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

10.8.3.5 Properly dispose of the acid waste.

10.8.4 Silica Gel Clean-up for DRO extracts

10.8.4.1 Samples requiring silica gel clean-up for the Oklahoma DRO method should follow the procedure in section 10.8.4.1, which includes the

addition of a reverse surrogate. Samples requesting silica gel clean-up for other DRO methods should follow the procedure in section 10.8.4.1.2, which does not include an additional surrogate.

10.8.4.1.1 If the sample is logged for method 3630C_M, concentrate the extract and all associated QC to slightly below 1 mL on the N-Evap. Add 100uL of the "SilicaGelSurr" standard to each extract and then bring the extracts to a 1 mL final volume with methylene chloride. Transfer to the appropriate final extract vial per section 10.6. Proceed to section 10.8.4.2.

10.8.4.1.2 If the sample is not logged for method 3630C_M but silica gel clean-up is still requested, no further surrogate is added. Concentrate to 1 mL final volume normally per section 10.6. Proceed to section 10.8.4.2.

NOTE: Please note that some projects require analysis of extract that has been silica gel cleaned as well as analysis of extract that has not been cleaned. Due to the limited final volume of the extract, samples requiring analysis of both cleaned and un-cleaned extract must be extracted twice, and in separate batches with separate QC.

10.8.4.2 Add approximately 0.05 g of activated silica gel to the extract, cap, and vortex for approximately 15 seconds. Allow the silica gel to settle.

10.8.4.3 Transfer the extract to a new vial, leaving the silica gel behind. Submit for analysis.

10.9 Documentation

All observations are recorded either directly into LIMS or on the hard-copy benchsheets. Any hand-written data recorded on the hard-copy benchsheets are transferred into LIMS before extracts are delivered to the analytical group. The hard-copy benchsheets are then saved and scanned into pdf files and sent to QA for archiving.

10.10 Maintenance

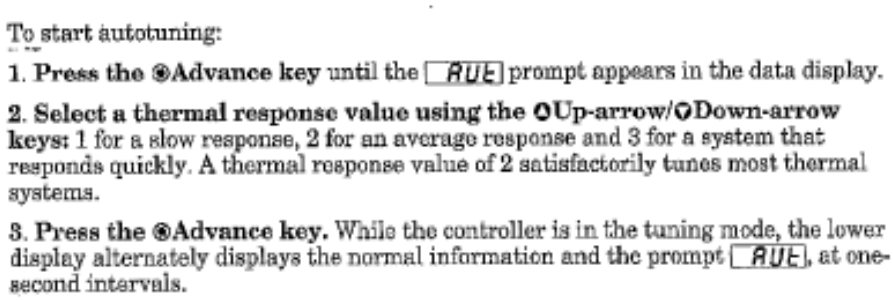
10.10.1 The chiller that operates the solvent recovery system should be checked periodically to ensure the water level is sufficient.

10.10.2 The SPE ports and valves used in the florisil are open and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to mitigate the risk of contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

10.10.3 The N-Evap needles are removed once a week and soaked overnight in a jar of methylene chloride. This is documented in the Organic Extraction Weekly

Cleaning Logbook.

10.10.4 The water bath used in the concentration of extracts has a thermostat that occasionally needs auto-tuned to keep the bath temperature within a narrow range. Record both the observed and the actual temperature on the benchsheet.



To start autotuning:

1. **Press the ◉Advance key** until the [AUE] prompt appears in the data display.

2. **Select a thermal response value using the ⏻Up-arrow/⏻Down-arrow keys:** 1 for a slow response, 2 for an average response and 3 for a system that responds quickly. A thermal response value of 2 satisfactorily tunes most thermal systems.

3. **Press the ◉Advance key.** While the controller is in the tuning mode, the lower display alternately displays the normal information and the prompt [AUE], at one-second intervals.

10.11 Troubleshooting

Unusual sample matrix may cause problems. If the extracts do not behave normally, contact a supervisor or senior analyst if you are unsure how to proceed. Document all observations and anomalies in a NCM.

11.0 Calibration

Not applicable to this procedure. See the determinative methods for calibration of the analytical instrumentation.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the

QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health & Safety Manual, and DV-HS-001P, "Waste Management Plan."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Flammable Solvents – Waste Stream C

14.2.3 1:1 MeCl2:Acetone – Waste Stream CA

14.2.4 Solid waste/sodium sulfate – Waste Stream D

14.3 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.2 Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.3 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

15.1.4 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

15.1.5 Method 3540C, Soxhlet Extraction, Revision 3, December 1996.

15.1.6 Method 3546, Microwave Extraction, Revision 0, February 2006.

15.1.7 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

15.1.8 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

15.1.9 Method 3660A, Sulfur Cleanup, Revision 1, July 1992.

15.1.10 Method 3665A, Sulfuric Acid/Permagante Cleanup, Revision 1, December 1996.

15.1.11 Method 3630C, Silica Gel Cleanup, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater

15.2.1 Method 608, Organochlorine Pesticides and PCBs.

15.2.2 Method 610, Polynuclear Aromatic Hydrocarbons.

15.2.3 Method 614, The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater

15.2.4 Method 625, Base/Neutrals and Acids.

15.3 ASTM D7065-11, Standard Test Method for Determination of Nonylphenols, Bisphenol A, p-tert-Octylphenol, Nonylphenol Monoethoxylate, and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass SpectrometryMethod Modifications:

16.0 Modifications

16.1 Method SW-846 3665A calls for the clean-up to be performed using 1:1 Sulfuric Acid:H_2O. This procedure calls for the clean-up to be performed using concentrated sulfuric acid.

16.2 ASTM D7065-11 calls for the samples to be concentrated to a 0.5 mL final volume. This procedure calls for a 1 mL final volume.

16.3 Method SW-846 3620C calls for the florisil lot check to be performed using a standard containing the some pesticides at various concentrations from 5 ug/L to 50 ug/L. Per the source method, 1 mL of the standard is diluted to 2 mL (for concentrations between 2.5 ug/L and 25 ug/L) and the cleanup is then carried out and the cleaned extract concentrated to 1 mL for a final concentration of 5 ug/L to 50 ug/L. This procedure calls for the lot check to be performed using a standard containing all the pesticides at the same concentration of 50 ug/L. 1 mL of this standard is cleaned up without prior dilution and then concentrated back down to 1 mL.

16.4 Method SW-846 3620C states that the florisil lot check passes if the pesticide recoveries are between 80% and 110% recovery. This procedure says the lot check passes if the pesticide recoveries are between 80% and 115%. This is done to match the CCV control limits.

16.5 Method SW-846 3620C states that the florisil lot check is to be performed using a standard containing the 2,4,5-Trichlorophenol at 0.1 ug/L. Per the source method, 0.5 mL of this standard is diluted to 2 mL (for a concentration of 0.025 ug/L) and the cleanup is then carried out and the cleaned extract concentrated to 1 mL for a concentration of 0.05 ug/L. This procedure calls for the lot check to be performed using a standard containing 2,4,5-trichlorophenol at 100 ug/L. 1 mL of this standard is cleaned up without prior dilution and then concentrated back down to 1 mL.

16.6 Method SW-846 3620C Section 11.1.3 states to condition the florisil cartridge with 4 mL of hexane. This procedure calls for 5 mL of hexane to be used. This is done for convenience.

16.7 Method SW-846 3630C calls for the silica gel clean-up to be performed with a column or SPE cartridge. This procedure calls for the silica gel to be added directly to the extract

and mixed. The reverse surrogate used indicates if the clean-up is effective.

17.0 Attachments

Attachment 1: Determinative and Extraction Methods Used in Conjunction with this SOP.
Attachment 2: Boiling Points of Solvents
Attachment 3: Kuderna-Danish Concentrator
Attachment 4: Florisil Check Solution

18.0 Revision History

- Revision 13, dated 30 November 2018
  - Annual Review
  - Addition note to section 1.3 indicating correct SOP reference for concentration of 8321 herbicides.
  - Updated condenser setting information in section 6.13 to reflect current practice.
  - Updated note in section 10.5.9 to reflect current practice.
  - Adjusted wording in section 10.8.1.2 to clarify “extracts” rather than “samples.”
  - Minor language adjustments insections 10.8.1.11 and 10.8.1.12 to clarify the application of the vacuum during recovery of florisil-cleaned extracts.
  - Added notes to sections 10.8.2 and 10.8.3 to specify that these steps are typically performed my instrument analysts.
  - Updated section 10.8.4 to provide clarification and reflect current practice.
  - Updated reference to Laboratory MDL Procedure CA-Q-S-006.
- Revision 12, dated 31 October 2017
  - Annual Review
- Revision 11, dated 31 October 2016
  - Added the paragraph referencing the QAM for general definitions in Section 3.0
  - Added the requirement to document the ID of pipettes used in Sections 6.1, 10.6.5.2 and 10.7.12.2.
  - Updated Section 10.1 to reflect current practices
  - Added the specification of using the S-evap for concentration in Sections 10.5, 10.5.8 and 10.5.12
  - Added the requirement to document both the observed and actual temperature in Sections 10.6.3 and 10.10.4
- Revision 10 dated 31 December 2015
  - Updated formatting and numbering throughout the document
  - Revised method code references to reflect current practice
  - Numbered NOTES where there were multiples (Sections 6.0, 10.4.5, 10.4.9, 10.5.5.2)
  - Updated drive reference in Section 6.1
  - Updated “Reagent Grade Chemicals” definition in Section 7.0 to be consistent with other SOPs
  - Added statement in Section 8 to specify that extracts are stored separately from standards
  - Updated Section 9.1 to be consistent with other SOPs
  - Added new section 10.2 for consistency with other SOPs
  - Added NOTE to Section 10.3
  - Added a requirement to Section 10.6.6 to rinse all concentrator tubes with methylene chloride before washing

  - Removed the reference to South Carolina in Section 10.8.2. The laboratory no longer holds certification for South Carolina by this method
  - Updated Section 12 to be consistent with other SOPs
  - Added NOTE to Section 14.3
  - Revised the concentration of 2,4,5-Trichlorophenol in the Florisil Check Solution described in Attachment 4.
    - The compound used to be at a concentration of 0.1 ug/mL in the standard
    - It is now at a concentration of 0.5 ug/mL
    - One mL of the standard is used in the Florisil check procedure, resulting in 0.5 µg of the compound loaded onto the 6 g of Florisil
  - Removed references to DV-MS-0005 in Section 1 and Attachment 1, the laboratory no longer performs this procedure
- Revision 9 dated 31 December 2014
  - Section 5.1.1.2 and Section 10.4.9 were revised to match current practice on the use of the solvent recovery system.
  - Section 6.1 Computer Software and Hardware was added.
  - Section 7.6 Baked Sodium Sulfate was revised to match current practice and the latest revision of CA-Q-S-001 DV-1.
  - Section 7.11 was revised to correct the TAL Reagent ID.
  - Section 9.1 was revised to include the statement “This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated”.
  - Section 9.4, 9.5, 9.6, and 9.7 were revised to remove information on Acceptance Criteria and Corrective Action. This information can be found in the analytical and QA SOPs.
  - Section 10.4.5 was revised to instruct the analyst to use approximately 1 teaspoon of sodium sulfate to dry extracts. This was done to limit the extract’s exposure to sodium sulfate which can cause low recoveries for some acid compounds. A note was also added to this section to instruct the analyst to use more sodium sulfate or a separatory funnel to remove water if a teaspoon of sodium sulfate is not sufficient.
  - The Note in Section 10.4.12 was revised to instruct the analyst to write an NCM and/or notify their supervisor if they have a concern that an extract concentrated too low.
  - Section 10.7.1 Florisil Clean-up was revised to give guidance on what to do if the florisil check fails.
  - Section 10.7.1 was revised to instruct the analyst to not use the vacuum to pull all of the solvent from the cartridge. This was done to prevent interfering compounds and 2,4,5-TCP from eluting off of the cartridge.
  - Section 10.7.1 and Attachment 4 Florisil Check Solution were revised to indicate which compounds are controlled and which compounds are monitored. In addition, surrogate compounds were added to the solution.
  - Section 10.7.1 and 10.9 were revised to instruct the analyst to soak the SPE ports in a jar with the valves open instead of disassembling the valves.
  - Section 10.7.3 was revised to instruct the analyst to perform the clean-up on approximately 2mL of extract. This was done to match current practice.
  - Section 10.7.4 Silica Gel Clean-up and Sections 15.1 and 16.0 were revised to match current practice.
  - Section 10.9 Maintenance was revised to include instructions on how to tune the water bath thermostat.
  - Attachment 3 – Concentration Summary was removed and replaced with WI-DV-0009. All other Attachments were re-numbered.
- Revision 8 dated 13 December 2013

- The procedure was revised to include ASTM D7065-11.
- The procedure was revised to include steps for silica gel clean-up for DRO extracts.
- Section 7 was revised to include details on the Florisil Solution and Florisil cartridges. These details were lacking in previous revisions.
- Section 10.4.2 was revised to give more detail on how to safely tighten the ground glass joint between the KD and concentrator tube.
- Section 10.6.3 was revised to give more detail about the required water level in the Turbo-Vap.
- Maintenance and Troubleshooting sections were added as Sections 10.8 and 10.9.
- Section 16 was revised to include method modifications from SW-846 3620C.
- Attachment 1 was updated to reflect the current SOPs in use in the laboratory.
- Attachment 3 was updated.

- Revision 7 dated 5 December 2012
  - Section 5 and Section 10.4.5 were revised to instruct the analysts to handle glass wool in a hood to avoid breathing in the dust.
  - Revised Section 10.4.8 to instruct the analysts to document both the observed and corrected temperatures.
  - Section 10.7.1.11 was revised to describe in more detail how the florisiled extracts are taken to the 2 mL final volume.
  - Section 14.2 was revised to include the waste stream for 1:1 MeCl2:Acetone – Waste Stream CA.
  - Attachment 1 was revised to include DV-OP-0015 as an acceptable extraction for Diesel Range Organics.
  - Attachment 3 was revised to include details on 8081/3510_LL concentration steps.
- Revision 6.0 dated 14 October 2011
  - The procedure was revised to remove instructions on how to concentrate and clean up extract for method 8070 and 607. TestAmerica Denver no longer supports these methods.
  - Section 1.3 was corrected to give the correct SOP number to Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS.
  - Section 7.5 was revised to state acetonitrile is tested before use. Previously this solvent was not tested before use.
  - The procedure was revised to include instructions that all extracts for analysis by method 8081, 8082, or 608 to be hexane exchanged only after concentration on the S-Evap. Previously the SOP instructed analysts to add the hexane exchange before the S-Evap for extracts that were concentrated by microwave extraction. This resulted in poor hexane exchanges, therefore the extracts are now concentrated before the exchange.
  - The procedure was revised to instruct analysts not to use the solvent recovery system when concentrating samples for analysis of low-level NDMA by GC/CI/MS/MS. This was done to eliminate a possible source of contamination in this ppt level analysis.
  - The procedure was revised to instruct analysts to use concentrated sulfuric acid in the acid clean up of PCB extracts.
  - The procedure was revised to clarify the exact steps used in the sulfur removal with mercury.

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Determinative and Extraction Methods Used in Conjunction with this SOP

Method Description	Determinative Method	Determinative Method SOP	Extraction Method	Extraction Method SOP
Diesel Range Organics & Jet Fuels	SW-846 8015B, 8015C, 8015D, California LUFT Method, & AK102 & AK103, NW-TPH, OK DRO	DV-GC-0027	WATER: SW-846 3510C, AK102 AK103 NW-TPH OK DRO SOIL: SW-846 3550B/C SW-846 3546 AK102, AK103 NW-TPH OK DRO	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Chlorinated Pesticides	SW-846 8081A, 8081B & EPA Method 608	DV-GC-0020 DV-GC-0016	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Polychlorinated Biphenyls	SW-846 8082, 8082A EPA Method 608	DV-GC-0021 DV-GC-0016	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Organo-phosphorus Pesticides	SW-846 8141A, 8141B, & EPA Method 614	DV-GC-0017	WATER: SW-846 3510C SOIL: SW-846 3540C	WATER: DV-OP-0006 SOIL: DV-OP-0010
Polynuclear Aromatic Hydrocarbons	SW-846 8310 & EPA Method 610	DV-LC-0009	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 SOIL: DV-OP-0016
Semi-volatiles by GC/MS	SW-846 8270C, 8270D & EPA 625	DV-MS-0011 DV-MS-0012	WATER: SW-846 3510C SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 or DV-OP-0008 SOIL: DV-OP-0016
Low-Level Semi-Volatiles by GC/MS	SW-846 8270C	DV-MS-0011	WATER: SW-846 3520C	WATER: DV-OP-0008
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0008 SOIL: DV-OP-0016 or DV-OP-0015
Isotope Dilution Analysis of n-Nitrosodimethylamine by GCMS SIM using LVI	SOP	DV-MS-0015	WATER: SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0021 SOIL: DV-OP-0016

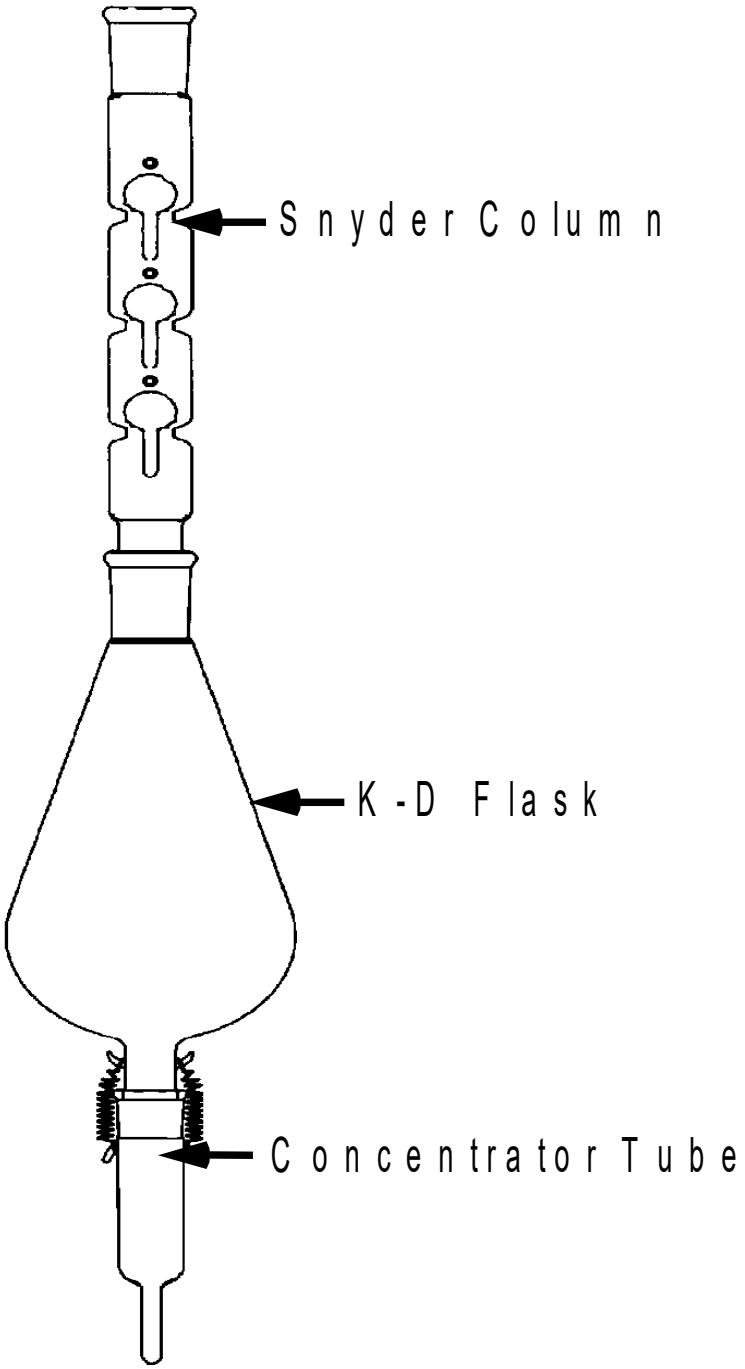
Attachment 2.

Boiling Points of Solvents

Solvent	Boiling Point (°C)
Methylene chloride	40
Acetone	56
Hexane	69
Methanol	65
Acetonitrile	82

Attachment 3.

Kuderna-Danish Concentrator



Attachment 4.

Florisil Check Solution
Prepared in Hexane

Compound	Concentration	Control
2,4,5-Trichlorophenol	0.05ug/mL	Y
Alpha-BHC	0.05ug/mL	Y
Alpha-Chlordane	0.05ug/mL	N
Aldrin	0.05ug/mL	N
Beta-BHC	0.05ug/mL	N
Dieldrin	0.05ug/mL	Y
Endosulfan I	0.05ug/mL	Y
Endosulfan II	0.05ug/mL	N
Endosulfan sulfate	0.05ug/mL	N
Endrin	0.05ug/mL	Y
Endrin Aldehyde	0.05ug/mL	N
Endrin Ketone	0.05ug/mL	N
Gamma-BHC	0.05ug/mL	Y
Gamma-Chlordane	0.05ug/mL	N
Heptachlor	0.05ug/mL	Y
Heptachlor expoxide	0.05ug/mL	N
Methoxychlor	0.05ug/mL	Y
4,4-DDD	0.05ug/mL	Y
4,4-DDE	0.05ug/mL	N
4,4-DDT	0.05ug/mL	Y
Tetrachloro-m-xylene	0.02ug/mL	Y
Decachlorobiphenyl	0.02ug/mL	Y

eurofins | Environment Testing TestAmerica

Eurofins TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

Eurofins TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: INCREMENTAL SAMPLING METHODOLOGY FOR SOILS AND SEDIMENTS [ASTM D 6323]

Approvals (Signature/Date):

Heather Fiedler, Technical Specialist — 7/6/20 Date	Reed Pottruff, Health & Safety Coordinator — 7/6/20 Date
Roxanne Sullivan, Quality Assurance Manager — 7/6/20 Date	Scott Hall, Laboratory Director — 7/6/20 Date

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 The purpose of this procedure is to obtain sub-samples from client provided samples which represent the concentration of the analytes of interest in the entire parent sample. This is based on the guidance in ASTM Standard D-6323 "Laboratory Subsampling of Media Related to Waste Management Activities," a DoD Quality Systems Manual (QSM) requirement.

1.2 This procedure applies to soils, sediments and other particulate matter. This method is highly dependent on client provided Data Quality Objectives. This procedure presents the laboratory's standard approach, but details at all stages of this procedure can vary from project to project. All project-specific variations must be documented and approved in writing. It is important that the analyst always check special project instructions in the TestAmerica LIMS (TALS) before proceeding.

1.3 TestAmerica has used incremental sampling methodology (ISM) for non-volatile organics (e.g., explosives residues by Method 8330), prior to acid digestion for metals analysis (e.g., Method 3050), and prior to analysis for volatile organics collected in multiple increments in the field and preserved in methanol (e.g., Method 5035). It can be used for a wide range of other analytical methods as well. However, this procedure is not applicable to soil samples to be analyzed for volatile organic compounds in which the entire sample provided by the lab's client is used for a single analysis (see the lab's volatile organics SOP for details).

1.4 This SOP addresses the pre-preparation of samples. The details of the twelve QC Elements, not otherwise addressed, are described in the associated preparation and/or analytical SOPs.

2.0 Summary of Method

2.1 For non-volatile analytes, the entire sample received from the client is air dried to a constant weight. Large non-representative pieces (rocks and twigs that will not pass through the sieve) may be removed manually. Other extraneous materials are removed by sieving. A mortar and pestle or sieve shaker or mechanical disaggregator may be used to break up soil agglomerates during the sieving process. Depending on the analytical method to be used after subsampling, and project objectives, the sample may be ground. The grinding option available at the laboratory is the ring-and-puck mill. A subsample is then taken using a multi-incremental approach.

2.2 ISM for Metals Analysis - the routine approach is to air dry, sieve to sub-10 mesh (2.1 mm), and collect 10 gram subsamples using 30 increments. The expectation is that the variability due to subsampling error will then be no more than 15% relative standard deviation (RSD) (see ASTM D-6323 for explanation and guidance for other acceptable variations). The Method 3050B digestion reagents are then increased proportionally to maintain the same chemistry as is used for 1 gram subsamples.

2.3 ISM for Explosives Analysis - the routine approach is to air dry, sieve to sub-10 mesh, grind (if logged for grinding), and collect 10 gram subsamples using 30 increments. If the samples are from firing points, then ring-and-puck grinding is required. The goal is to

achieve 10% or less RSD from subsampling variability. Further details for explosives are given in SOP DV-OP-0018 and are not discussed in this SOP.

2.4 ISM for Volatile Analysis – the multi-incremental sampling is done in the field, where the 30 increments of 5 grams each are added to a septum-cap bottle containing 200 mL methanol provided by the laboratory. From that point on, the lab's procedure for medium level soils (SOP DV-MS-0002) is followed.

2.5 The basic formula to use when working with clients to select the optimal approach for other methods or other precision objectives is given in Attachment 1 to this SOP. The Attachment defines the trade off between subsample size, particle size, and the desired level of precision.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

3.2 Sample or Client Sample – refers to the entire quantity of material delivered to the laboratory for testing.

3.3 Subsample – refers to the portion of sample taken in the laboratory for a given analysis. The objective of this procedure is to ensure that the subsample is a reasonably accurate representation of the entire sample.

4.0 Interferences

4.1 If multi-incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory effort entailed in this SOP may add little or no improvement in results.

4.2 Potential loss of lighter semi-volatile compounds (e.g., naphthalene) through the drying and grinding process has not been well studied. Before employing the procedure for such compounds, the possible loss of lighter compounds should be discussed with the client and if possible, investigated before the procedure is performed.

4.3 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or contamination causing misinterpretation of results. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running blanks.

4.4 Contamination by carryover can occur when a low concentration sample is processed immediately following a high concentration sample. For this reason, special care must be taken to follow the equipment cleaning steps.

4.5 As described in this SOP, the lab does not routinely grind samples for metals testing. It is expected that detection limits and reporting limits for some metals would have to be elevated based on long-term blank results if grinding is required.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, TestAmerica Denver Addendum to the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Anyone working in the grinding room needs to be enrolled in the Hearing Conservation Program. See DV-HS-0010 for details. Personnel operating grinding equipment are required to wear ear plugs when the equipment is turned on. When standing next to the Humbolt mechanical disaggregator described in Section 6.1.7 during operation, the decibel levels are above 80 decibels, therefore anyone operating the disaggregator must be enrolled in the Hearing Conservation Program and wear hearing protection. While the disaggregator is running, the decibel levels in the room are below 80 decibels, therefore personnel not enrolled in the Hearing Conservation Program can be in the room. Hearing protection is always available to every analyst and they are encouraged to use it.

5.3.2 Operations involving the handling of samples outside of sealed containers, e.g., sieving, are conducted in ventilation hoods to avoid exposure to dust. Dust masks are available for use in the grinding room, but are optional.

5.3.3 Operations involving the grinding of radioactive samples can be particularly hazardous due to the increased potential for exposure from airborne dust. If a sample is labeled as “CAT 1”, “CAT 2”, “CAT 3” or “CAT 4” and requires grinding through the ring and puck, contact the Radiation Safety Officer (RSO) immediately.

5.4 Primary Chemical and Material Hazards – cleaning solvents

MATERIAL	HAZARDS	EXPOSURE LIMIT (1)	SIGNS AND SYMPTOMS OF EXPOSURE
Acetonitrile	Flammable Poison	40 ppm – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Acetone	Flammable	1,000 ppm – TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment

6.1.1 Balance, capable of measuring ± 0.01 g. Calibration checked per SOP DV-QA-0014.

6.1.2 Sieve Shaker – used to facilitate the sieving of large sample volumes.

6.1.3 Ring and Puck - The grinding bowl and puck are cleaned after each use by washing with soap and water with a plastic brush, rinsing with hot tap water, rinsing with DI water, and then rinsing with a 10% acetonitrile solution in acetone. A final wipe down of the bowl and puck while still wet with solvent is done with a Kimwipe.

6.1.4 Trays – "baker's rack" type stack for air drying soils

6.1.5 Drying tower – custom built tower similar to "baker's rack" type stack for air drying soils, including air drying fans and air filters

6.1.6 Sieves - 10 and 30 mesh, brass for general use, stainless steel for metals testing. Sieves are cleaned after each use by washing with soap and water with a green plastic brillo pad (be careful not to damage the mesh), rinsing with hot tap water, rinsing with DI water. Prior to use, the sieves are rinsed with 10% acetonitrile in acetone and wiped with a Kimwipe. Sieves are allowed to dry in a hood prior to use.

6.1.7 Mortar and pestle – Porcelain, various sizes cleaned after each use by washing with soap and water, rinsing with hot tap water, and then rinsing with DI water. The mortars and pestles are rinsed with 10% acetonitrile in acetone, wiped with a Kimwipe, and allowed to dry in a hood prior to use.

6.1.8 Mechanical Disaggregator – Humbolt Manufacturing Part Number H-4199. Used in place of a mortar and pestle to quickly reduce cakes of dry soil. The stainless steel disaggregator reduces soil agglomerates and sieves the soil through a 10 mesh sieve. The mechanical disaggregator is used to break up soil agglomerates but it is not an alternative to the Ring and Puck. The mechanical disaggregator is cleaned after each sample by removing the hopper. The hopper is washed with soap and water, rinsed with tap water, rinsed with DI water, and then rinsed with 10% acetonitrile in acetone. The Hopper is then wiped dry with a laboratory tissue. The hammers and body of the disaggregator are cleaned after each sample by rinsing with DI water and wiping dry with a laboratory tissue.

6.2 Expendable Supplies

6.2.1 Subsampling tools:

6.2.1.1 Scored paper scoops (TAL-0150 and TAL-0150 LARGE from Commodity Management Services)

6.2.1.2 Plastic sample scoops – square-ended

6.2.2 Aluminum foil and aluminum dishes

6.2.3 Parchment paper to line trays for metals testing

6.2.4 Alconox detergent

6.2.5 Ottawa Sand – blank media for organics

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 10% Acetonitrile in Acetone – mix 100 mL of acetonitrile with 900 mL of acetone. This solution is used for rinsing purposes only so exact measurements are not required.

8.0 Sample Collection, Preservation, Shipment and Storage

Container Type	Preservative	Holding Time
Plastic or glass	By individual test *	By individual test *

After air drying, samples can be stored at room temperature.

* - Reference the analytical SOPs.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense

(DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 and 5.1 unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Grinding Blanks

9.2.1 Ring and Puck Grinding Blanks.

Before each sample is processed through the ring and puck mill, the ring and puck will be cleaned per Section 6.1. Then approximately 200 g of Ottawa sand will be ground. This ground sand will be saved and labeled with the sample ID of the next sample ground with the suffix “blank”. After a batch of samples has been processed through the ring and puck, a composite will be generated using sub-aliquots from all blanks ground before the samples. This is done by placing approximately 1 tablespoon of material from each of the individual sample blanks in a clean re-sealable plastic bag. The bag is then sealed and the material is mixed and homogenized by shaking and kneading the bag. A 10 g aliquot is then removed from the bag and labeled as the batch grinding blank. This composite is extracted and analyzed in the same manner as the field samples.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank with positive results for the same contaminant must be reprocessed and reanalyzed. If un-ground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative

9.3 Precision

9.3.1 On a project basis, the lab will discuss precision objectives with the client prior to initiating work. If evaluation of the RSD is needed, the laboratory will need to analyze at least one set of triplicate samples in every preparation batch. In other cases, the lab will employ duplicate matrix spikes and control limits will be

expressed as relative percent difference (RPD).

9.3.2 If the client supplies multiple field samples to use for replicate testing, then the laboratory will compare results to acceptance limits and qualify data if the precision limits are not met. If the replicates are prepared from the single field sample that is dried, ground, and sieved, then the acceptability of each grinding batch can be controlled based on the precision objectives established for the project.

9.4 Other QC samples (method blank, LCS, and MS/MSD) are created after subsampling, and vary depending on the analytical method. See DV-OP-0018 for special QC requirements for explosives, which usually require grinding a standard reference material.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Dry the Samples

10.3.1 The entire contents of the sample container must be processed. It is not acceptable to remove any aliquots until after the sample has been dried, sieved, and ISM performed. If the client requests aliquots to be taken before the sample is dried, sieved, and ISM performed, an NCM should be written to document this was done per client request.

10.3.2 Depending on the sample size, the samples are laid out in aluminum pans, or on large trays lined with aluminum foil to dry. Some clients may request metals analysis on the dried samples. In those cases, samples are laid out on parchment paper.

10.3.3 Spread the samples out in a thin layer to facilitate drying. Use a disposable wooden spatula to break up any clumps and agglomerates.

10.3.4 The tray or pan that the sample is laid out into is labeled with the sample ID. A second analyst checks to make sure that the labels on the tray or pan match the labels on the client sample container to ensure samples are not accidently mixed up. This check is documented in TALS.

10.3.5 Place the samples in a drying tower, hood or well ventilated area at room temperature. Document in TALS the date and time the samples were laid out to dry. An electronic temperature recording device records the temperature of the room and the data is downloaded weekly.

10.3.6 When the samples appear to be dry enough that they can be sieved without caking, subsample approximately 15 grams into an appropriate weighing vessel and record the exact weight, the date, and the time (see Attachment 3). Set this 15 gram aliquot (still in the weighing vessel) next to the rest of the drying sample. Take care to use an appropriate weighing vessel for the analytical methods requested, as the aliquot removed in this step will still be included in the volume used for ISM (i.e., Do not use an aluminum weigh boat for samples requiring metals analysis).

10.3.7 After 2 hours, reweigh the aliquot in the same weighing vessel and record the exact weight, the date, and the time. If the weight of the sample is within 10% of the previous weight, proceed to Section 10.4.

10.4 Sieve the Samples

10.4.1 Clean the sieves prior to use following the instructions in Section 6.

10.4.2 Some samples may require the use of a mortar and pestle or a mechanical disaggregator to break up dried clumps. Refer to Section 6.1 on how to clean and rinse the mortar and pestles and the mechanical disaggregator before use.

10.4.3 Record the weight of the entire dried sample in the Worksheet tab in TALS. This is a requirement for DoD QSM 5.0 and 5.1.

10.4.4 Sieve the entire dried sample through the appropriate sized sieve. Care must be exercised not to eliminate soil agglomerates during this step. The soil can be broken into small pieces with a gloved hand or another instrument (a wooden spatula for example). If a gloved hand is used, care should be taken to change out gloves in between samples to prevent cross-contamination.

10.4.5 Remove large rocks, vegetation, and twigs that do not pass through the sieve. Mosses and other types of fine vegetation should be physically shredded while sieving to release trapped soil and residues. The only materials that should be eliminated by sieving are rocks and vegetation. All soil must be broken up to pass through the sieve.

10.4.6 Place any soil that does not pass through the sieve into a clean mortar. Break up soil agglomerates using the pestle or, as an alternative, use the mechanical disaggregator. Be sure to break up all soil so that it can pass through the sieve. Only extraneous material such as rocks and vegetation should be removed with the sieve. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.4.7 Collect all of the material that passes through the sieve on a clean piece of foil or parchment paper.

10.4.8 An automatic sieve shaker can be used to help facilitate the sieving of samples. A receiver pan is placed under a sieve and the sample is added to the sieve. Then a lid or another receiver pan for a second sample is placed on top. The stack is then

clamped inside the sieve shaker for no more than 30 minutes. Inspect the samples to ensure that only extraneous material such as rocks and vegetation were removed with the sieve. If needed use a mortar and pestle to break up soil agglomerates. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.4.9 If metals or any other analyses are requested on the sample prior to grinding, perform ISM on the portion of the sample that passed through the sieve at this time before proceeding to any grinding steps in Section 10.6. Refer to Method Comments, Sample comments, and Login Comments for instructions if any other tests besides metals analyses are to be performed on an un-ground aliquot before proceeding to Section 10.6

10.5 Incremental Sampling Methodology for Metals and other methods requested on un-ground material.

10.5.1 Select an appropriate subsample container. For metals analyses, a 100 mL digestion cup is appropriate. For organic methods, an amber glass container is appropriate. Reach out to the departments performing the analysis for guidance on selecting the appropriate subsample container.

10.5.2 Remove the cap from the appropriate subsample container and place on a balance and tare. The entire sieved sample is spread out on a sheet of parchment paper to a 1 cm thickness.

10.5.3 Using a subsampling tool (described in section 6.2.1), take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of sieved material.

10.5.3.1 For methods 6010B, 6010C, 6020, and 6020A a 10 g - 11 g aliquot is required for each sample and each MS/MSD sample. Collect one extra 10 g - 11 g aliquot per sample in case re-digestion is needed.

10.5.3.2 For methods 7471A and 7471B, a 3 g - 3.3 g aliquot is required for each sample and each MS/MSD sample. Collect one extra 3g - 3.3g aliquot per sample in case re-digestion is needed.

10.5.3.3 For other methods, refer to method comments, sample comments, and login comments, or to the departments performing the analyses for instructions regarding aliquot size.

NOTE: Sub-out ISM samples will need to aliquoted into amber 40mL VOA vials and delivered to sample receiving with the appropriate paperwork. Aliquot size will vary and depends on the analysis needed.

10.5.4 Record the sample weight on the ISM Worksheet described in Attachment 2.

10.6 Grinding

The instructions in this section are to be used as a general procedure when grinding is requested prior to extraction and analysis for any method. Reference DV-OP-0018 for details on grinding samples for explosives analysis.

10.6.1 Ring and Puck Mill Grinding

10.6.1.1 See Section 6.1 on how to clean the ring and puck dish.

10.6.1.2 If the sample is logged for ring and puck grinding, a grinding blank per Section 9.3.1 consisting of baked Ottawa sand will be processed through the ring and puck dish before each sample. These individual blanks will be composited into one grinding blank for the associated samples and will be analyzed in addition to the normal extraction blank.

NOTE: When preparing the grinding blanks, it is not necessary to do five 60-second grinds. One 60-second grind of the Ottawa sand is sufficient.

10.6.1.3 After a grinding blank has been processed through a ring and puck dish, that blank is labeled as the blank associated to the next sample processed through that same dish. Do not clean the ring and puck dish after the blank.

10.6.1.4 In a hood, transfer the sample into a clean ring and puck dish. Do not overfill the dish (approximately 300 g of sample can fit in one dish). If needed, grind the sample in 300 g or smaller increments and recombine after the whole sample has been ground. The entire sample **must** be ground. Place the dish securely in the holder and close the door on the machine. Grind the sample for five 60-second periods with a one minute cooling time between grinds for a total of 5 minutes of grinding. Remove the dish and in a fume hood open the lid and inspect the sample. It should be the consistency of flour. The consistency of the material is checked by pinching some between two fingers of a gloved hand and feeling for grit and by looking for any un-ground fibers. If grit is detected or if fibers are observed, additional grinding is needed.

10.6.1.5 If the sample reaches a flour-like consistency before all 5 one-minute grinds have been completed, then it might be beneficial to not perform all 5 grinds in order to avoid excessive heat and to avoid packing the sample onto the side of the grinder. If the analyst inspects the sample and it has flour like consistency before all 5 grinds are completed, they can make the decision to stop after less than 5 grinds. A NCM should be written to document the deviation from the source method and the reasoning.

NOTE: During the one-minute cooling time, the dish should be placed in a shallow ice water bath to facilitate cooling. Be sure the

bath is shallow enough so that water does not get inside the dish.

NOTE: If multiple 300 g increments are used for grinding and the sample is recombined without mixing, it has been shown through Duplicate/Triplicate QC results that the sample is non-homogenous. To re-homogenize the sample, lay the sample out on foil/parchment paper and use provided scoop to mix and spread the sample into a 2-D slabcake.

10.7 Incremental Sampling after grinding.

10.7.1 Remove the cap from a 40 mL amber vial or other appropriate subsample container (refer to Section 10.5.1 for guidance) and place on a balance and tare. The entire ground sample is spread out on a sheet of parchment paper or aluminum foil to a 1 cm thickness.

10.7.2 Using a subsampling tool (described in Section 6.2.1), take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of ground material.

10.7.2.1 For explosives a 10 g to 11 g aliquot is required for each sample and each MS/MSD sample.

10.7.2.2 For other extractable methods a 30 - 33 g aliquot is common, but reference project instructions and method SOPs for more detail.

10.7.2.3 For other methods, refer to method comments, sample comments, and login comments, or to the departments performing the analyses for instructions regarding aliquot size.

10.7.3 Record the sample aliquot weight on the ISM Worksheet.

10.8 Maintenance

10.8.1 Approximately once a month, the cover on the Ring and Puck should be removed and any dirt should be cleaned up.

10.8.2 When excessive wear is noted, replace the hammers in the Mechanical Grinder.

10.8.3 Occasional lubrication of the Ring and Puck clamp is needed.

10.8.4 The o-rings in the Ring and Puck dishes should be replaced when worn.

10.9 Troubleshooting

Low recoveries for Tetryl in the explosives grinding LCS may be indicative of high temperatures during grinding. Review the cooling step noted in Section 10.6.1.5 in order to minimize the effect of the heat generated during the grinding process.

11.0 Calculations

Relative Standard Deviation

$$RSD = \frac{S}{\overline{X}}$$

Where: S = standard deviation

$\overline{X}$ = mean

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1 - 2 the laboratory reporting Limit (RL) and carried through the entire preparation and analytical procedure. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

The use of organic solvents to complete the equipment cleaning steps is minimized. Quantities are limited to residues on equipment that quickly evaporate in a hood.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual, *Waste Management and Pollution Prevention*.

14.2 The following waste streams are produced when this method is carried out:

- Solid Waste – Waste Stream S
- Flammable Solvent Waste – Waste Stream C

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 "Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples," USEPA, November 2003.

15.2 "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities" ASTM D 6323-98 (Reapproved 2003)

15.3 DoD Quality Systems Manual Version 5.0, 2009. Appendix B, Table 3.

15.4 DoD Quality Systems Manual Version 5.1, 2017. Table B-23.

16.0 Method Modifications

Item	Method	Modification
N/A	N/A	*No method modifications specified.*

17.0 Attachments

Attachment 1: Consideration of Fundamental Error in Selecting MIS Options
Attachment 2: ISM Worksheet
Attachment 3: ISM Constant Weight Worksheet

18.0 Revision History

Revision 13, dated 6 July 2020

- Removed sections that prompted analyst to place samples in bags to re-homogenize after puck milling.

Revision 12, dated 3 March 2020

- Annual Reveiw
- Updated copyright information

Revision 11, dated 11 December 2018

- Minor formatting and language corrections throughout
- Replaced all references to “mechanical grinder” to read “mechanical disaggregator” to distinguish more clearly between grinding/milling and soil disaggregation.
- Added new part numbers for preferred subsampling tool to section 6.2.1.
- Added DoD 5.1 reference information to section 9.1.2.
- Added new drying tower to equipment section 6 and to section 10.3.5.
- Removed note in section 10.3.7 which allowed for skipping constant weight analysis for non-DOD samples.
- Added section 10.4.3 to meet DoD 5.1 requirement that dried sample is weighed prior to sieving.
- Removed notes regarding DoD 5.1 specific requirements from section 10.4.6 and 10.4.8, and incorporated these specific requirements into standard procedure for all samples. These requirements include weighing and retaining material that does not pass through the sieve.
- Added wording to 10.4.9 to allow for non-metals tests that are not ground.
- Added sections 10.5.1 and 10.7.1 regarding selection of appropriate subsample container.
- Changed “100 mL digestion cup” in section 10.5.2 to “the appropriate subsample container.”

- Corrected wording in 10.5.3 to read “the layer of sieved material” rather than “the layer of ground material.”
- Added sections 10.5.3.3 and 10.7.2.3 regarding aliquot sizes for methods other than metals.
- Changed language in section 10.7.2 from “a square ended spatula” to “a subsampling tool described in section 6.2.1.”
- Removed section 10.9.1 comment regarding securing ring and puck as it no longer requires securing. Added note about troubleshooting low tetryl recoveries.
- Revised MDL policy reference in section 12.1.

Revision 10, dated 3 October 2017

- Updated section 2.1 to clarify air drying to a constant weight.
- Updated section 8.0 to reflect the secondary storage option for air dried samples.
- Updated section 10.3.6 and 10.3.7 to demonstrate drying to a constant weight.
- Updated section 10.5.2 to reflect ISM Sub-out practices.
- Added attachment 3.

Revision 9, dated 28 February 2017

- Removed all references of the Ball Mill from the body of the instructions.
- Updated Section 3.1 to reference DV-QA-003P and QAM for general terms
- Updated Section 9.1 to be consistent with other SOPs
- Added current Sections 10.1 and 10.2 – NCM reference and instructions
- Updated Section 12 to include current MDL, LOQV, DOC and Training information
- Updated solid waste stream from D to S in Section 14.2

Revision 8, dated 29 February 2016

- Added aluminum dishes to Section 6.2
- Added Section 7.1 and 7.2
- Added what corrective action is used for Ring and Puck Grinding blanks that have hits in Section 9.4.1, as this differs from the corrective action for Ball Mill.
- Changed the acceptable weight range in Section 10.3.2.2 from 3-3.05 g to 3-3.3 g to be consistent with the 10% provided for other methods.
- Added more description to Section 10.4.2.2 to instruct the analyst not to wash the Ring and Puck dish at this step.
- Removed Section 10.6.5 that provided instruction about maintenance of the centrifuge as this equipment is not used in this SOP.
- Removed all revision histories 2010 and earlier (available upon request)

Revision 7, dated 28 February 2015

- Annual Technical Review
- Reformatted the SOP
- Added detail to Section 5.3.1 about the Humbolt mechanical grinder.
- Added information to Section 5 about the hazards of grinding radioactive samples.

- Revised Section 9.4.1 to give more detail on how the Ring and Puck composite grinding blanks are created.
- Revised Section 10.6 to include maintenance on the centrifuge.
- Added Attachment 2: ISM Worksheet

Revision 6, dated 05 February 2014

- Annual Technical Review
- Edited Section 6.1, subsection “Ball Mill” to allow for un-baked sand to be used in the cleaning of the ball mill stones and to allow the use of 1 pint cans.
- Edited Section 6.1, subsection “Sieves” to state a brillo pad can be used on the sieves so long as the mesh is not damaged.
- Added a comment to Section 9 stating that this procedure meets DoD QSM 5.0 criteria unless otherwise stated.
- Removed Acceptance Criteria information to Section 9. This information can be found in the analytical SOPs.
- Added a NOTE in Sections 10.4.1.1 and 10.4.2.4 giving instructions on how to ensure the sample is homogenous after it has been split into separate grinding containers and then later re-combined.
- Added Section 10.6 Maintenance and Section 10.7 Troubleshooting per DoD QSM 5.0.

Revision 5, dated 31 January 2013

- Annual Technical Review
- Added Section 1.4 to address the 12 QC Elements.
- Updated Section 2.3 and 10.4.1 to allow the use of Ball Mill grinding. The laboratory successfully completed a Method Validation for Ball Mill grinding and therefore is able to offer this to all clients.
- Section 6 was updated to include cleaning procedures for sieves, Ring and Puck dishes, Ball Mill stones, Mortar & Pestles, and Paint Cans.
- Section 2.1, Section 6, and Section 10.2.2 were updated to include the mechanical grinder that can be used in place of mortar and pestle for samples that do not require metal testing.
- Section 9.3.3 was updated to include acceptance criteria for Grinding Blanks for DoD samples.
- Section 10.3.1 was updated to instruct the analysts to aliquot the samples directly into 100mL digestion cups for metals analysis.
- Removed Attachment 2: How to Batch Samples in LIMS.

Revision 4.2, dated 31 January 2012

- Removed all references to Multi-Incremental Subsampling which is now trade-marked.
- Updated Section 6.1 to reflect the correct number of small and large grinding stones used in the Ball Mill grinding of samples.
- Updated Attachment 2.

Revision 4.1, dated 20 January 2011

- Added detail about the electronic temperature recording device that records the temperature of the room.
- Revised procedure to state that during the one-minute cooling time, the dish will be placed in a shallow ice water bath to facilitate cooling.
- Revised Attachment 2 to include the method Dry_Grind and more details on how to batch samples that are logged for both MULTI_INC and grinding methods.

Earlier revision histories have been archived and are available upon request.

Attachment 1

Consideration of Fundamental Error in Selecting MIS Options

The following formula given in ASTM D-6323 was used to produce the table that follows.

$$S^2 = 18 * f * e * d^3 / M_s$$

where,

S^2 = the relative variance of the contaminant concentration due to the fundamental error

f = shape factor, a dimensionless number, a value of 0.5 can be taken as typical (Pierre Gy, 1982)

e = the population's average density (g/cm^3). For this table a typical soil density of 2.5 g/cm^3 was used.

d = the diameter of the largest particle in centimeters, and

M_s = the mass of the sample in grams

Sample Mass and Maximum Particle Size to Achieve a Desired RSD

Subsample Mass (g)	Sieve Size (US Standard Mesh)	At 5% RSD Max Size (cm)	At 10% RSD Max Size (cm)	At 15% RSD Max Size (cm)
0.1	35	0.02	0.04	0.05
1	18	0.05	0.08	0.10
2	13	0.06	0.10	0.13
5	12	0.08	0.13	0.17
10	10	0.10	0.16	0.22
30	7	0.15	0.24	0.31
50	6	0.18	0.28	0.37
100	5	0.22	0.35	0.46

Attachment 2

ISM Worksheet

G:/QA/Edit/FORMS/Organic Prep Forms/MASTER ISM Spreadsheet_Rev1

ISM BATCH:

Use this spreadsheet to document alquot weights when aliquotting into digestion or extraction vessels. If aliquoting into a temporary vessel, no need to document the exact weight because the sample aliquot will be transferred and weighed at the time of analysis.

Login	Sample	Method -->											
			(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											

Attachment 3

ISM Constant Weight Worksheet

Located: \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Organic Prep Forms

DV-F-0070-10-03-2017-Rev 0

ISM Batch Number:

Use this spreadsheet to document drying samples to a constant weight.

			Analyst Initial Date/time 1	Analyst Initial Date/time 2	Dry?		Analyst Initial Date/time 3	Dry?	
Login	Sample	Tare weight (weight of weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 1, 2	Proceed with ISM?	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 2, 3	Proceed with ISM?
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Microwave Extraction of Solid Samples by Method [SW-846 3546]

Approvals (Signature/Date):

Andrew Pepping — 4/22/19 — Date
Technical Specialist

Doug Gomer — 4/22/19 — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan — 4/22/19 — Date
Quality Assurance Manager

for Charles Newton — 4/22/19 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing the sample and organic solvent. This procedure achieves analyte recoveries equivalent to those from soxhlet or sonications methods, but uses less solvent. This SOP is based on SW-846 Method 3546.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate solvents and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

2.0 Summary of Method

A measured weight of sample, typically 15 g, is solvent extracted using a microwave extractor.

3.0 Definitions

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as

a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Sodium sulfate is not used in the extraction vessel. This is because salts are known to super heat when exposed to microwave energy. Samples are extracted without the addition of sodium sulfate, but the extracts are dried with sodium sulfate after the extraction, before concentration of the extracts. If the sample is excessively wet the aliquot can be divided among two or three extraction vessels and the extracts combined prior to concentration.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6 Paint chips are an especially difficult matrix to extract. Oftentimes the paint chips dissolve or partially dissolve in solvents and therefore can ruin glassware and extraction vessels. It is the laboratory's experience that paint chips are best extracted by method SW-846 3580 instead of 3550C or 3546.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 A post-run cool down must be used after each extraction to prevent the possibility of operator burns. Pressure builds up in the closed vessel at high temperatures. Care should be taken when opening the vessel when it is above room temperature.

5.1.2 Samples that contain metal fragments or metal components of any kind should not be extracted by this procedure. These samples should be extracted by method SW-846 3550C instead. Care should be taken to inspect samples carefully as they are aliquotted.

5.1.3 Eye protection that satisfies ANSI Z87.1 (as described in the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	Hazards	Exposure Limit [(2)]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous. It is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Equipment

6.1.1 Microwave extractor. Mars5: MarsExpress™CEM MARS® and Microwave extractor. Mars6: MarsExpress Plus™ CEM MARS®

At least once a year, power measurement calibration should be performed at 400 W, 800 W, and 1600 W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.

6.1.2 Microwave extraction vessels. 75 mL Teflon™ Express vessels with stopper and cap (CEM Corp.) in addition to 110 mL borosilicate glass tubes accompanying 110 mL Teflon™ Express Plus vessels with stopper and cap (CEM Corp).

6.1.3 Hand wrench to tighten the caps on the extraction vessels.

6.1.4 MARS 40 position carousel (CEM Corp) and 20 position carousel (CEM Corp)

6.1.5 Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.

6.2 Supplies

6.2.1 Media bottles, 100 mL or 250 mL capped with aluminum foil.

6.2.2 Stainless steel conical funnels

6.2.3 Ashless cellulose filter paper

6.2.4 Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.

6.2.5 Metal spatulas or tongue depressors.

6.2.6 Solvent dispenser pump.

6.2.7 Filter flask.

6.2.8 Vacuum pump.

6.2.9 Washing tool for Teflon™ extractor vessels. This tool is a long thin sponge-like brush.

6.2.10 40 mL VOA vials and caps

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Methylene chloride – Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.2 Acetone - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Hexane - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1

before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Baked Sodium Sulfate, 12-60 mesh - Heat sodium sulfate in a 400°C oven for at least four hours. QA personnel post the list of approved lots at solvent storage areas.

7.5 Baked Ottawa Sand – Heat Ottawa sand in a 400°C oven for at least four hours.

7.6 35% Nitric Acid – Dilute concentrated (70%) Nitric Acid 1:1 in water.

7.7 Standards - Please reference SOP DV-OP-0020 and WI-DV-0009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils for Method 8082A[2]	Glass with Teflon-lined lids	15 grams	Cool, ≤ 6°C	None	SW-846
Wipes for Method 8082A[2]	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	15 grams	Cool, ≤ 6°C	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	14 days	SW-846

[1] Exclusive of analysis.

[2] Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require the 14 day holding time for Method 8082. The states of Alabama, California, Colorado, Connecticut, Nevada, New Jersey, Pennsylvania, and Rhode Island require the 14 day holding time for Method 8082.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

9.4.1 A method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

9.4.2 The method blank consists of 15 g of baked Ottawa sand free of any of the analyte(s) of interest.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

9.5.1 At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

9.5.2 The LCS consists of 15 g of baked Ottawa sand to which the analyte(s) of interest are added at known concentration.

9.5.3 Method AK102 requires LCS and a LCSD for every batch for every spike compound.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.6.2 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

9.6.3 DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must also be documented as a nonconformance, with a cause and corrective action described.

10.3 Critical Procedural Considerations

10.3.1 As stated throughout this SOP, analysts must review the LIMS Method

Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.3.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample.

NOTE: Rotate glassware; do **not** use specific glassware, equipment or positions for the MB and LCS/LCSD.

10.4 Periodic cleaning.

10.4.1 Mars5 Express Microwave Extractor. CEM Mars At least once every four weeks, the extraction vessels must be cleaned using a "Clean Method" on the microwave. The method is under the User Directory with the settings that follow:

- Sample Type: Inorganic
- Control Type: Ramp to Temperature
- Power: 100%
- Ramp: 5 minutes to 180°C
- Hold: 10 minutes

10.4.2 Mars6 Express Plus Microwave Extractor. CEM Mars At least once every four weeks, the extraction vessels must be cleaned using a "Clean Method" on the microwave. The method is under the Classic Method, "Cleanup2" with the settings that follow:

- Sample Type: Inorganic
- Control Type: Ramp to Temperature
- Stage 1
- Power: 1600%
- Ramp: 15 minutes to 180°C
- Hold: 10 minutes
- Temperature guard : 200°C

10.4.3 Fill each tube with 30 mL of the nitric acid solution described in Section 7 and cap tightly. Place the tubes in the carousel, then run the "Clean Method"

10.4.4 Allow the vessels to cool, and then dispose of the nitric acid in waste stream J. Rinse the vessel with DI water three times.

10.4.5 Fill each tube with 30 mL of 1:1 Methylene Chloride: Acetone solution and

cap tightly. Place the tubes in the carousel, then run the "Clean Method" again.

10.4.6 Allow the vessels to cool, and then dispose of the solvent in waste stream CA. Allow the vessels to air dry.

10.5 Assemble and Clean the Extraction Tubes Immediately Before Use.

10.5.1 If the microwave tube, cap, or plugs are wet, pre-rinse with acetone.

10.5.2 Rinse the microwave tube, cap and plug twice with methylene chloride. The plugs can be placed in a large glass jar to help facilitate the rinse.

10.5.3 Discard the solvent in the correct waste stream.

10.6 Aliquot Samples

10.6.1 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. If the sample is a wipe, transfer the wipe to the extraction vessel.

10.6.2 Label microwave vessel with the sample ID, method, and batch number. The label needs to be flat.

NOTE: For method 8270 borosilicate glass tubes are to be used in housing the sample in addition to required QC; respectively. The Glass tubes will be inserted into the retaining vessel and capped for extraction.

This method is performed using the Mars6 Microwave only

10.6.3 Do not use specific vessels or carousel positions for the MB and LCS.

10.6.4 For each MB and LCS sample, weigh 15 g to 17 g of baked Ottawa sand into labeled VOA vials or similarly clean glass intermediate containers with a lid. Record a nominal weight of 15 g in the initial volume field, but record the actual weight to the nearest 0.1 g in the notes column.

10.6.5 For each sample and MS/MSD, weigh 15 g to 17 g of sample into labeled VOA vials or similarly clean glass intermediary containers with a lid. Record the weight to the nearest 0.1 g directly into LIMS or hand record the weight on the benchsheet.

NOTE: For wipe samples, the original sample containers that the wipes are received in should be used in place of the intermediary sample containers described above.

10.6.6 Cap the intermediary sample containers either with the appropriate lid or aluminum foil.

10.6.7 Place the labeled intermediary sample containers on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Checklists (See WI-DV-0009).

10.7 Prepare a bottle with a bottle-top dispenser with the appropriate solvent(s).

10.7.1 Methylene Chloride is used for soil and wipe samples for the following methods:

- SW-846 8015B
- SW-846 8015C
- SW-846 8015D
- Low-Level NDMA (8270D_SIM_LL)

10.7.2 For soil extraction by all other methods, the solvents used are acetone and methylene chloride. These are added separately.

10.7.3 For wipe samples by method 8081 and 8082, the solvent used is hexane.

10.7.4 For wipe samples by method 8270 SIM, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.8 Add Surrogate and Spike Solutions

NOTE: The standards should be allowed to come to room temperature before spiking the samples.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009.

10.8.1 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples. Document the standards and pipette(s) used on the benchsheet.

10.8.2 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard (see WI-DV-0009) to the intermediary container for each field sample and QC sample. Verify the ID of the standard used on the benchsheet.

10.8.3 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard (see DV-OP-0009) to the intermediary container for each field sample and QC sample. Verify the ID of the standard used on the benchsheet.

10.9 Making sure not to overflow the intermediary sample container, remove the cap, and slowly add approximately 15 mL of the appropriate solvent to the container. See below for the appropriate solvent:

NOTE: The solvent should be added as soon as possible after the addition of the surrogate and spiking standards to prevent loss of the more volatile compounds.

10.9.1 15 mL of methylene chloride is added to the container for soil and wipe

samples for the following methods:

- SW-846 8015B, 8015C, and 8015D
- Low-Level NDMA (8270D_SIM_LL)

10.9.2 15 mL of acetone is added to the intermediary container for all other soil samples.

10.9.3 15 mL of hexane is added to the container for wipe samples by method 8081 and 8082, the solvent used is hexane.

10.9.4 15 mL of 1:1 methylene chloride and acetone is added to container for wipe samples by method 8270 SIM.

10.10 Mix the contents of the intermediary sample container using a spatula or wooden tongue depressor for 30 seconds. Then, cap the intermediary sample container and use a vortex mixer to mix the contents of the intermediary sample container for an additional 30 seconds. This mixing must generate a thoroughly wetted and disaggregated sample slurry. If clay clumps or other sample aggregation is evident after 1 minute of combined manual and vortex mixing, sand may be added to facilitate disaggregation, followed by an additional minute of mixing (manual + vortex as described above). If sand is added during this step, document this in an NCM.

10.11 Transfer the mixed/wetted sample to a microwave extraction vessel using three 5 mL methylene chloride rinses. This will bring the total combined solvent volume to 30 mL.

NOTE: The solvent should completely cover and saturate the sample. Additional solvent may be needed depending on the matrix of the individual sample. The sample and solvent must not fill more than 2/3 of the vessel.

NOTE: If the sample matrix appears to be unusual, or especially wet, the combined sample/solvent mixture can be equally divided between two or three separate microwave extraction vessels. The vessels will be extracted independently, but the extracts will be re-combined before concentration. This will prevent the extraction vessels from over-heating and venting if the sample is unusually wet, oily, or bulky (if a 15 g aliquot would fill the tube more than ¾ full). If the sample is split into two or three separate vessels, document this in an NCM.

NOTE: For method 8270, the mixed/wetted sample or QC will be transferred into borosilicate glass tubes. The glass tubes are then inserted into the retaining vessel and capped for extraction.

10.12 Seal the vessels by placing the plug on top of the vessel, small side down, and hand tighten the cap over the plug.

NOTE: Care should be taken to ensure that the plug, the cap, and the threads of the vessel are clean of any material or debris.

10.13 After being sealed, the vessels must be inverted several times to ensure that the

material is well mixed and saturated. It is recommended that when extracting with 100% methylene chloride to vent and re-cap the vessels before continuing to relieve excess pressure and thereby preventing the vessels from venting during the extraction.

NOTE: 8270 Samples extracted using the borosilicate glass tubes will not be inverted, as this will result in the sample + solvent spilling out of the glass tube into the retaining vessel.

10.14 Load vessels into the carousel.

10.14.1 There must be at least 8 vessels in the carousel. Adding blank vessels with sand and solvent may be necessary.

10.14.2 Balance the tubes around the carousel to ensure that all samples are exposed to an equal amount of energy during the extraction. See Attachment 1 for details. Only samples using the same extraction solvent should be placed in the same carrousel and run at the same time.

10.14.3 For the vessels to be correctly loaded in the carousel the cap should completely touch the top of the carousel with no other part of the extraction vessel visible.

10.15 Place the carousel into the microwave, making sure that it sits on the turning apparatus correctly. The carousel should be able to rotate. Close the door.

10.16 Mars5 Express: The Method Menu screen should indicate “Start Current Method” as being 3546 Full Xpress. Press the green “Start/Pause” button to begin the extraction.

NOTE: If a different method is shown, go to the “Load Method” on the menu screen. Choose “User directory” and place the cursor on the desired method. Press the “Home” button to return to the main menu, where the test highlighted will appear under the “Start Current Method”.

10.16.1 The method is under the User Directory with the settings that follow:

- Sample Type: Organic
- Control Type: Ramp to Temperature
- Power: 100% (1600 W)
- Ramp: 20 minutes to 115°C
- Hold: 10 minutes

10.17 Mars6 ExpressPlus: The “One Touch Method” menu should be selected. Next, the “CEM 3546 glass 110C” method should be selected. At the bottom right hand corner of the screen should be a green “start” selection. Press the green “Start” button to begin the extraction.

NOTE: If a different method is shown, press the back arrow, found on the bottom left hand of the screen” until you reach the appropriate menu.

10.17.1 The method is under the CEM 3546 glass 110C" program with the settings that follow:

- Sample Type: Organic
- Control Type: Ramp to Temperature
- Stage 1
- Power: 500-1500W
- Ramp: 15 minutes to 110°C
- Hold: 15 minutes

10.17.2 When the extraction is complete, the vessels will need to return to room temperature prior to opening the vessels. The microwave will indicate the approximate temperature of the vessels.

CAUTION: If the carousel is removed from the microwave before the vessels are at room temperature, do NOT open the vessels. The vessels may be placed in a rack outside of the microwave to cool down.

10.17.3 The microwave contains a solvent sensor that will indicate the presence of solvent in the microwave and will stop the extraction. To minimize this, care needs to be taken not to overfill the vessel and to properly cap and tighten the vessel prior to extraction. If the solvent sensor indicates the presence of solvent, open the door and inspect the tops of the tubes for evidence of a solvent leak. If solvent has vented or leaked out of an extraction vessel, the sample must be re-aliquotted and the extraction started over. It is best to re-aliquot the sample into two or three separate extraction vessels to prevent over-heating again. Document this in an NCM.

10.18 Assemble and Clean Filter Funnels and Media Jars.

10.18.1 Without gloves on, fold a 18 cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 100 mL or 250 mL media bottle.

NOTE: For low-level NDMA samples by method 8270D_SIM_LL, use designated glass funnels instead of the stainless steel funnels and instead of re-usable media jars, use disposable amber bottles. This is done to prevent contamination.

10.18.2 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with the extraction solvent (see Section 10.7), so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the

rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.18.3 Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.18.4 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.18.5 Discard the solvent in the correct waste stream.

10.19 Filter the Extracts

10.19.1 After the extraction method is complete and the vessels reach room temperature, quantitatively transfer the entire sample through solvent rinsed sodium sulfate funnels and into the media jar. The quantitative transfer is performed by rinsing the microwave extraction vessel at least three times with solvent.

NOTE: The quantitative rinse is vital in order to achieve good recoveries. The rinses should be significant enough that when done, the extract volume is between 75 mL and 100 mL.

NOTE: If the sample aliquot was split between two or three tubes, the extracts from all the tubes shall be combined at this time. Filter all of the extracts through the same sodium sulfate funnel and collect in the same media jar.

NOTE: During the 8270 extraction, it has been noted that solvent may be found in the retaining vessel after extractions. This contains analytes of interest and should be filtered into the funnel with the rest of the sample. Quantitatively rinse the glass tube; DO NOT rinse the retaining vessel.

10.19.2 Once the solvent has completely drained into the collection apparatus, rinse the funnel contents with 10 to 20 mL of additional solvent. Dispose of the solid sample and sodium sulfate into Waste Stream D and cap the media jar with aluminum foil.

NOTE: For 8270 extractions, dispose of the glass tube

10.20 If the extract contains visible solids, it will be necessary to filter the extract again prior to concentration.

10.21 Store the extract refrigerated at $\leq$ 6°C until concentration. Ensure that the extracts in 1:1 Methylene chloride:acetone are placed in a flammable rated refrigerator.

10.22 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

10.23 All glassware and microwave tubes, plugs, and caps are washed according to DV-OP-0004.

10.24 Maintenance

10.24.1 As needed, wipe out the inside and outside of the microwave with a damp cloth.

10.24.2 See Section 10.4 for vessel cleaning.

10.24.3 At least once a year, power measurement calibration should be performed at 400 W, 800 W, and 1600 W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.

10.25 Troubleshooting

10.25.1 If it appears that the solvent sensor is malfunctioning, ensure that the sensor is aligned at a 45 degree upward angle on the back of the unit.

10.25.2 The snorkel vent should be set inside of a hood, but care should be taken so that the opening is not blocked. Make sure the snorkel does not press against the back of the hood.

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health

and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 1:1 MeCl2:Acetone – Waste Stream CA

15.2.3 Flammable solvent – Waste Stream C

15.2.4 Solid waste/sodium sulfate – Waste Stream D

15.2.5 Nitric Acid Waste – Waste Stream J

15.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3456 Microwave Extraction, Revision 0, February 2007.

16.2 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water", Manchester Environmental Laboratory, Dept of Ecology, State of Washington.

17.0 Method Modifications:

17.1 SW-846 Method 3546 calls for samples to be either air-dried and ground or mixed with sodium sulfate prior to extraction. This procedure does not call of the air-drying of samples unless requested by the client as this may lead to loss of the more volatile compounds. Sodium sulfate is not used in the extraction vessel, rather the extracts are dried with sodium sulfate after extraction and prior to concentration. Salts are known to superheat when exposed to microwave energy.

17.2 SW-846 Method 3546 calls for samples to be aliquoted on a balance capable to weighing to 0.01 g. This SOP calls for a balance capable to weighing to 0.1 g as this is sufficient to report data to 3 significant figures.

17.3 SW-846 Method 3546 Section 1.4 states "2-20 g of material is usually necessary and can be accommodated by this extraction procedure." This SOP calls for 30-33 g of material.

17.4 SW-846 Method 3546 Section 11.7 states "Add approximately 25 mL of the appropriate solvent system to the vessel." This SOP calls for the addition of 25-30 mL of solvent.

17.5 Method NWTPH-Dx calls for samples to be extracted by method SW-846 3550C. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method NWTPH-Dx is a possible determinative method by this procedure.

18.0 Attachments

Table 1: Determinative Methods Using Microwave Extraction

Attachment 1: Proper Carousel Loading

19.0 Revision History

- Revision 9, April 25, 2019
  - Removed all references to Alaska methods AK 102 and AK 103 as these will now be conducted with Sonication in DV-OP-0016 exclusively.
  - Annual Technical Review
- Revision 8, March 6, 2018
  - Changed nominal sample weight from 30 grams to 15 grams in accordance with the microwave extraction best practices and standardization procedure provided by corporate QA. This change is reflected in sections 2.0, 8.0, 9.4, 9.5, and 10.6.
  - Changed all references to WI-DV-009 to correct document ID: WI-DV-0009.
  - Modified sections 10.6-10.11 to specify the use of the intermediary sample container required in order to implement the microwave extraction best practices and standardization procedure provided by corporate QA.
  - Modified section 10.7.2 to clarify that acetone and methylene chloride are added separately in accordance with the microwave extraction best practices and standardization procedure provided by corporate QA.
  - Modified, added, and/or rearranged sections 10.9-10.11 in accordance with the microwave extraction best practices and standardization procedure provided by corporate QA. This change involves modifying the addition of solvents, mixing of the sample/solvent mixture, and transferring from an intermediary container into the microwave extraction vessel.
  - Updated sections 6.1.1, 6.1.2, 6.1.4, 10.4.1, 10.4.2, 10.6.2, 10.6.5, 10.11, 10.14, 10.15, 10.15.1, 10.17.1, 10.17.2 with notes to reflect the usage of the new microwave for 8270 FS, HSL list analytes only.
- Revision 7, January 31, 2017
  - Annual Technical Review
  - Added paragraph to Section 3.0 referencing the QAM for general definitions
  - Added paragraph to Section 6.0 to record IDs of pipettes and equipment
  - Updated language in Section 9.6.3 requiring LCSDs when no MS/MSD
  - Added note to Section 10.3.2 on rotating glassware/equipment/positions
  - Added current Section 13.2 defining LOQV
- Revision 6, January 31, 2016
  - Annual Technical Review
  - Updated Section 9.1 to contain verbiage consistent with other SOPs
  - Added Section 9.6.3 regarding DoD MS/MSD requirements

- Changed the "Clean Method" frequency from two to four weeks in Section 10.4.1
- Changed the waste stream from C to CA in Section 10.4.5
- Section 10.5.2 changed the rinse requirement to be performed twice.
- Added Section 10.6.3 instructing not to use specific vessels or positions for the MB and LCS.
- Modified Section 10.6.4 weight recording requirements
- Added Section 10.6.6 – cap with aluminum foil
- Added the documentation of the standards and pipette used in Section 10.8.1
- Clarified the need to punch a hole in foil when spiking to Section 10.8.2 & 10.8.3
- Clarified the process for adding solvent to vessels in Section 10.9
- Added the requirement to place 1:1 Methylene chloride:acetone extracts in a flammable rated refrigerator to Section 10.18
- Revised Section 13.1 – Method Detection Limit Study (MDL)
- Revised Section 13.2 – Demonstration of Capabilities
- Revised Section 13.3 - Training Requirements
- Updated Section 17.4 to reflect the addition of 25-30 mL of solvent
- Archived all revision histories 2010 and earlier

- Revision 5, January 31, 2015
  - Annual Technical Review
  - Reformatted SOP
  - Revised Section 7.4 to remove the requirement to test the sodium sulfate before use. This was done to reflect current practice in CA-Q-S-001-DV-1.
  - Added "NWTPH DRO" to the procedure
  - Revised Section 10.5.2 to state that the plugs and caps can be rinsed in a large glass jar.
  - Added a note in Section 10.15.1 to state that for method 8270D_SIM_LL, designated glass funnels and disposable amber bottles will be used to filter the extracts.
  - Added Sections 16.2-16.5 to list AK102, AK103, and NWTPH methods as references.
  - Removed Section 17.8, redundant with 17.5.
  - Updated Table 1 to reference the correct methods and SOPs.
- Revision 4, January 31, 2014
  - Annual Technical Review
  - Revised Section 1.2 to state that the procedure may be used for additional methods when appropriate solvents are used instead of pH as there are no pH adjustments made in the procedure.
  - Removed TeflonTM lined caps from the Equipment and Supplies list in Section 6 as the lab now uses aluminum foil.
  - Added footnote to the table in Section 10 stating some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require 14 day hold time for method 8082A.
  - Revised Section 9.1.2 to state that this procedure meets all criteria of DoD QSM 5.0.
  - Revised Section 9.4 to clarify that one method blank is processed with each batch.
  - Removed "Acceptance Criteria" and "Corrective Action" information from Sections 9.4, 9.5, 9.6, and 9.7. This information can be found in the analytical SOPs.

- Added a bullet point in Section 10 to clarify that any deviations discovered after the procedure is performed are to be documented in an NCM.
- Revised Section 10 to remove the instruction to place the label towards the bottom of the vessel. This is not necessary. Also removed the requirement that the label must include the date. The label includes the batch number, which is unique and the date of extraction is recorded in the batch.
- Revised the procedure to state the periodic acid cleaning of the tubes should be done at least once every two weeks instead of weekly.
- Removed methods "NWTPH DRO" and "Okla_DRO" from the procedure. The lab does not perform microwave extraction for these methods at this time.
- Added sub-sections for Maintenance and Troubleshooting to Section 10 per DoD QSM 5.0.
- Added low-level NDMA and 8015D as a possible analytical method to Section 10 and to Table 1
- Removed 8310 as a possible analytical method in Table 1.
- Added Attachment 1 to give instructions on how to properly load the vessels in the carousel.

- Revision 3, January 31, 2013
  - Annual Technical Review
  - Sections 4.2 and 10.5.4 were revised to remove the optional addition of sodium sulfate to the samples before extraction. It was determined that the better option when dealing with wet samples is to split the sample into two or three tubes and re-combine the extracts before concentration.
  - Section 4 was revised to add instructions on how to deal with paint chip samples.
  - Section 5 was revised to add comments about the dangers of metal fragments in samples.
  - Section 6 was revised to include the requirement that the Power Measurement Calibration procedure be performed on the unit every year.
  - Section 8 was revised to update the hold times for Method SW-846 8082A.
  - Section 10.8 was revised to give more detail on how full the extraction vessel should be once solvent has been added.
  - Section 10.13.1 was revised to allow the carousel to be removed from the microwave unit before the vessels are cool so long as the vessels are not opened.
  - Section 10.15.1 was revised to add a note about the importance of quantitative transfers and rinses while filtering the extracts.
  - Section 10.15.1 was revised to add instructions to combine all extracts from samples that were originally split across two or three tubes.
  - Section 15 was revised to include the waste stream CA.
  - Added the Note to Table 1

- Revision 2.0, January 31, 2012
  - Annual Technical Review
  - Updated Section 4.2 and Section 10.5.4 to describe when sodium sulfate should be used in the extraction vessel.
  - Updated Section 6.0 to allow the use of aluminum foil to cap 100mL and 250mL media jars.
  - Updated Section 6.1 to include details on computer software and hardware.
  - Updated Section 7.0 to include details on the purity of reagents and standards.

- Updated Section 9.1.4 and Section 10.1 to more accurately reflect the NCM process.
- Corrected grammatical and formatting errors
- Updated Section 10.3 to include a solvent cleaning after the weekly acid cleaning.
- Updated Section 10.5.4, Section 10.7.2, and Section 10.7.3 to include an option to split the sample aliquot into two separate microwave vessels.
- Updated Section 10.10 and 10.13.2 to give details on how to prevent vessels from over-heating and venting and steps to be taken if venting does occur.
- Updated Section 10.16 to accurately reflect how the laboratory handles extracts with suspended sediment.
- Updated Section 10.19 to reference SOP DV-OP-0004 on how to clean the microwave vessels.

- Revision 1 dated 01 Jan 2011
  - Added 8270C SIM as a valid determinative method by microwave extraction.
  - Changed the procedure to call for the extract to be filtered thru a conical steel funnel lined with cellulose filter paper instead of a glass funnel with glass wool. This was done to help remove sediment from the extracts.
  - Removed details about the surrogate and spike standards used in the extraction. This information can now be found in DV-OP-0020.
  - Added instructions to Section 7 on how to prepare the nitric acid solution used in the weekly cleaning of the tubes.
  - Changed the solvent used in the extraction of samples for method 8081 and 8082. The samples are now extracted in a 1:1 Mixture of MeCl2:Acetone instead of a 1:1 Mixture of MeCl2:Hexane.
  - Revised the procedure in Section 10.5 for aliquotting samples to state that 30 to 33g of sample should be used instead of 30±2g and that the weight should be recorded to the nearest 0.1g instead of the nearest mg.

Earlier revision histories have been archived and are available upon request.

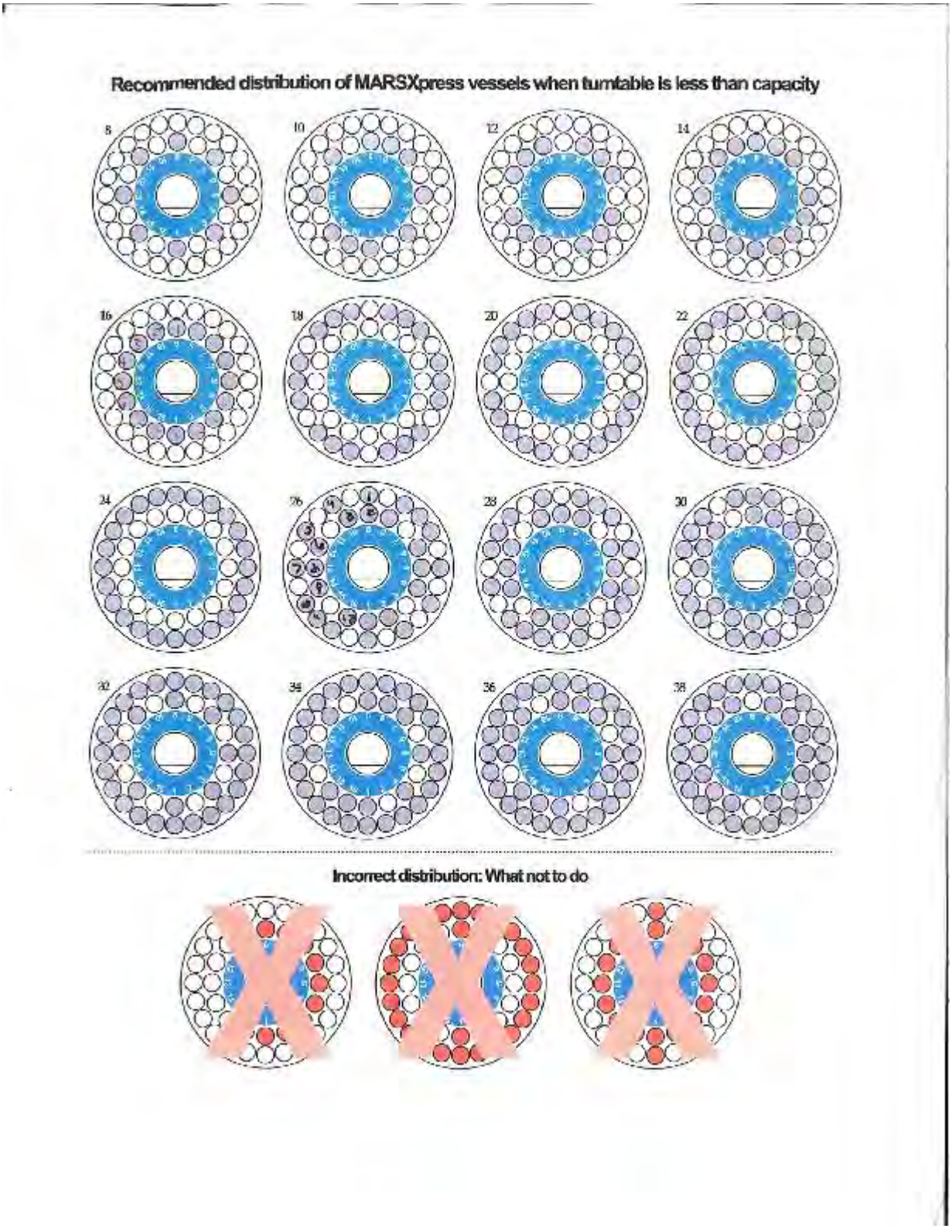
TABLE 1.

Determinative Methods Using Microwave Extraction

Method Description	Determinative Method	SOP
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls (PCBs)	SW-846 8082 SW-846 8082A	DV-GC-0021
Diesel and Residual Range Organics	SW-846 8015B SW-846 8015C SW-846 8015D NWTPH-Dx AK102 AK103	DV-GC-0027
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM SW-846 8270D SIM	DV-MS-0002
Low-Level NDMA by Isotope Dilution, GC/MS SIM, Large Volume Injection	SW-846 8270C/D SIM	DV-MS-0015

ATTACHMENT 1.

Proper Carousel Loading



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Solid Phase Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Water Samples [SW-846 3535A]

Approvals (Signature/Date):

Andrew Pepping — 12/10/18
Technical Specialist — Date

Doug Gomer — 12/10/18
Health & Safety Manager / Coordinator — Date

Roxanne Sullivan — 12/11/18
Quality Assurance Manager — Date
Thomas Lill, signing for Roxanne Sullivan.

Richard Clinkscales — 12/10/18
Laboratory Director — Date

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the extraction of nitroaromatic and nitroamine explosive residues by solid phase extraction (SPE) from aqueous samples. This procedure is based on SW-846 method 3535A.

1.2 This procedure does not describe the analysis of the extracts. For those details, see the following SOPs:

1.2.1 DV-LC-0002, *Analysis of Nitroaromatic and Nitroamine Explosive Compounds by HPLC*

1.2.2 DV-LC-0010, *Analysis of Nitroaromatic and Nitroamine Explosives Compounds by APCI/LC/MS*

2.0 Summary of Method

2.1 Aqueous samples undergo solid phase extraction (SPE). For samples that are to be analyzed by method 8330A or 8330B, 25 g of NaCl is added to a 500 mL sample aliquot and extracted. For samples that are to be analyzed by method 8321A or 8321B LC/MS, or by 8321B LCMSMS, a 1,000 mL aliquot is extracted. The analytes are absorbed onto the sorbent material in the SPE cartridge and then eluted with 2.5 mL of 0.1% acetic acid in acetonitrile. The concentrated extract is diluted 1:1 with an aqueous solution of calcium chloride prior to analysis by method 8330A or 8330B or with water prior to analysis by method 8321A or 8321B.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

3.2 Explosives: As used in this SOP, the term “explosives” refers specifically to the analytes listed in the Tables of EPA Method 8330A, 8330B, and the DoD Quality Systems Manual (QSM). These include compounds that can be readily detonated with heat, shock, or ignition, such as nitroglycerin, RDX, and TNT. It also includes production by-products and degradation products of true explosives.

3.3 SPE: Solid Phase Extraction

3.4 LIMS: Laboratory Information Management System

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2 Contamination by carryover can occur when a low-concentration sample is analyzed immediately following a high-concentration sample.

4.3 The extraction of high-level samples can cause contamination in the extractions lab, especially on the solid-phase manifold. If water samples appear to have a red tint or if the extracts appear to be multi-phasic, the project manager and client should be contacted and care should be taken to minimize cross-contamination.

4.4 It has been determined that tetryl can adhere to the cartridge in such a manner that pure acetonitrile will not elute the compound off of the cartridge packing. It is surmised that tetryl may become ionized in the extraction procedure and adhere more tightly to the cartridge packing than the other explosives. Therefore the elution is performed with 0.1% acetic acid in acetonitrile. The lab has demonstrated increased recoveries for tetryl when this slightly acidic elution solvent is used.

4.5 Samples with suspended solids or sediment can cause the extraction cartridge to clog. It may be necessary to filter the samples before extraction to prevent this. If a sample is filtered prior to extraction, an NCM should be written.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3 Primary Materials Used

5.3.1 The following is a list of materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not contain all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

5.3.2 A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

MATERIAL (1)	HAZARDS	EXPOSURE LIMIT (2)	SIGNS AND SYMPTOMS OF EXPOSURE
ACETONITRILE	FLAMMABLE POISON	40 PPM – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
METHANOL	FLAMMABLE POISON IRRITANT	200 PPM - TWA	A slight irritant to the mucous membranes. Toxic effects are exerted upon the nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause the skin to be become dry and cracked. Skin absorption can occur, symptoms may parallel inhalation exposure. Irritant to the eyes.
PHOSPHORIC ACID	CORROSIVE	1 PPM - TWA	Ingestion can cause severe burns to the throat, mouth, and stomach, abdominal pain and nausea. Severe exposures by ingestion can lead to shock, circulatory collapse, and death. Inhalation is not an expected hazard unless misted. Corrosive, contact with skin or eyes can cause redness, pain, severe burns, blurred vision, and permanent eye damage.
ACETIC ACID, GLACIAL	CORROSIVE POISON FLAMMABLE	10 PPM – TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to the skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
METHYLENE CHLORIDE	CARCINOGEN IRRITANT	25 PPM (TWA) 125 PPM (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

(1) ALWAYS ADD ACID TO WATER TO PREVENT VIOLENT REACTIONS.

(2) EXPOSURE LIMIT REFERS TO THE OSHA REGULATORY EXPOSURE LIMIT.

6.0 Equipment and Supplies

6.1 Equipment

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1.1 Vacuum manifold for SPE cartridges. Capable of maintaining approximately 66 cm (26") of Hg. After each use, the valves and tube caps are removed from the manifold, set to the open position and placed in a jar with acetonitrile. The jar is placed into a sonication bath for a minimum of 30 minutes. The jar used to sonicate the valves should be replaced at least weekly to avoid contamination.

6.1.2 Nitrogen evaporation apparatus (N-EVAP) for the concentration of some water extracts.

6.1.3 Balance capable of measuring ± 0.1 g. Calibration checked per SOP DV-QA-0014. Used to measure the initial sample mass and volume.

6.1.4 Pipettor with disposable 1.0 mL tips. Calibration checked per SOP DV-QA-0008. Used to add surrogate and spike standards to samples.

6.1.5 Pipettor with disposable 0.1 mL tips. Calibration checked per SOP DV-QA-0008. Used to add surrogate and spike solution to samples.

6.1.6 Pipettor capable of dispensing 5 to 50 mL. Calibration checked per SOP DV-QA-0008. Used to calibrate vials to hold 5 mL for the final volume determination for water extracts.

6.2 Supplies

6.2.1 pH paper, wide range.

6.2.2 Volumetric Flasks and Graduated Cylinders, glass, Class A, various sizes

6.2.3 Amber Glass Vials, 8.0 mL, with Teflon-lined screw caps. For the storage of final extracts. Vials used to store the final extracts are calibrated to hold a volume of 5 mL by using a calibrated pipette to deliver 5 mL of acetonitrile into the vial and marking the meniscus with a fine-tipped permanent marker. This volume is accurate to ± 2%.

6.2.4 Disposable pipettes, used for non-quantitative transfers only.

6.2.5 SPE Cartridges for Methods 8330A and 8330B (PoraPak RDX 6 cc tubes, Waters part no. WAT047220).

6.2.6 SPE Cartridges for method 8321A and 8321B (Strata SDB-L 500 mg packed into 6 mL tubes, Phenomenex part no. 8B-S014-HCH).

6.2.7 SPE tubing, non-PTFE in composition, with weights attached to one end. Tubes are cleaned before and after each use with acetonitrile followed by reagent water.

6.2.8 Miscellaneous laboratory apparatus (beakers, filter flasks, Büchner funnels, volumetric flasks, pipettes etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.2.9 Glass fiber filter paper, Ahlstrom, catalog number 1510-0900 or equivalent.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent Water – TestAmerica Denver has three ELGA Analytical water purification systems equipped with UV lamps. The water coming from the ELGA system should have a resistivity of 18 - 18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.2 Acetonitrile, CH_3CN - HPLC grade (ACN) – Each lot of solvent is tested following CA-Q-S-001. QA personnel post the list of approved lots at solvent storage areas.

7.3 Phosphoric acid, 85% H_3PO_4 (Phosphor Acid) – Purchased ready to use. Used to make up the $CaCl_2$ Solution in Section 7.4**.**

7.4 Calcium Chloride Solution, 5 g/L (CaCl2_Sol) - Used to bring the 8330A and 8330B extracts up to volume.

Place 5 ± 0.05 g of reagent grade $CaCl_2$ into a one-liter volumetric flask containing approximately 500 mL of reagent water. Swirl the solution until the $CaCl_2$ is dissolved. Add approximately 1 mL of 85% H_3PO_4 to acidify the solution and make up to volume with reagent water.

7.5 Approximately 0.1% Acetic Acid in Acetonitrile (0.1%AAinACN) – Open a new 4-liter bottle of acetonitrile and add 4 mL of acetic acid. Cap and mix. This reagent is given a 1 year expiration date.

7.6 Baked Sodium Chloride – Added to 8330A or 8330B samples to facilitate the extraction of picric acid. Bake in 400 °C oven for at least 4 hours.

7.7 Methylene Chloride – Used to pre-condition the SPE cartridges.

7.8 Standards

7.8.1 Please reference SOP DV-OP-0020 and WI-DV-0009 for information regarding the surrogate and spike standards used in this procedure.

7.8.2 The LCS standards should remain in the freezer for storage. The standard is to be brought to room temperature before use.

8.0 Sample Collection, Preservation, Shipment and Storage

Method	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
8321A or 8321B	Amber glass; Teflon caps	1 Liter	Cool. ≤ 6 °C, not frozen	7 Days	SW-846 8330B
8330A or 8330b	Amber glass; Teflon caps	500 mL	Cool. ≤ 6 °C, not frozen	7 Days	SW-846 8330B

[1] Exclusive of Analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 and 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank consists of reagent water, which is free of any of the analyte(s) of interest. The method blank is processed and analyzed just as if it were a field sample.

9.5 Laboratory Control Sample (LCS)

One LCS must be processed with each preparation batch (see Section 9.6.2). The LCS consists of reagent water to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

NOTE: If insufficient sample volume is available for an MS/MSD, an NCM must be written and a LCSD must be prepared.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.6.2 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Clean the SPE manifold and tubing - Pull acetonitrile, followed by water from the ELGA system, through each tube and each port on the vacuum manifold.

NOTE: For method 8321A and 8321B be sure to use the Strata SDB-L cartridges. For method 8330A and 8330B be sure to use the PoraPak RDX cartridges.

10.4 Precondition the SPE cartridge

10.4.1 For method 8330: Fill each cartridge two times with methylene chloride (12 mL total) and draw it through the column by gravity. Then fill each cartridge two times with acetonitrile (12 mL total) and draw it through the column by gravity.

10.4.2 For method 8321: Fill each cartridge two times with acetonitrile (12 mL total) and draw it through the column by gravity or using a vacuum manifold. Empty the in-process tank into waste stream C.

10.5 Condition the SPE cartridge - Fill the cartridge four times with reagent water and draw the water through the cartridge. Fill the cartridge a fifth time and close the valve to prevent the water from dripping through. Cap each cartridge using clean and dry caps.

10.6 Prepare MBs and LCSs – For every MB and LCS sample by method 8330A and 8330B, place 500 mL of reagent water in a disposable 500 mL Boston round bottom bottle. For every MB and LCS sample by method 8321A and 8321B, place 1,000 mL of reagent water in a disposable 1,000 mL Boston round bottle.

NOTE: Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

10.7 Inspect the water sample for the presence of sediment. The sample must be free of particulate matter before it is introduced into the SPE extraction cartridge. Particulate matter will obstruct the media and cause the analysis to fail. If the samples have sediment or suspended solids, proceed to Section 10.8. If the samples are particulate free, proceed to Section 10.9.

10.8 Filter Samples with Solids. If the sample contains suspended solids or settled solids that will likely clog the solid phase cartridge, then the sample can be vacuum filtered though glass fiber filter paper to separate the solids from the water. Rinse a filter flask and a Büchner funnel lined with filter paper once with acetonitrile and twice with water. Under vacuum, filter the sample. Do not rinse the original sample container as this will incorrectly raise the initial volume of the sample. If there is no sediment remaining in the original sample container, return the sample to the original sample container. Whenever possible, the sample should be extracted directly from the original sample container. This will allow the sample container to be rinsed. If the original sample container contains sediment, the sample can be extracted in the filter flask. Write an NCM stating that the sample had to be filtered to remove suspended solids.

10.9 Aliquot 8330 Samples received in 1 L ambers - Whenever possible, the sample should be extracted directly from the original sample container. This will allow the sample container to be rinsed at the end of the procedure. If the original sample container is a 1,000 mL container and the requested method is 8330A or 8330B, then a 500 mL aliquot should be transferred to a new disposable 500 mL amber bottle. Write an NCM to document that the rinse of the original sample container could not be performed. Follow the procedures in Section 10.10 to aliquot either gravimetrically or volumetrically.

10.10 Aliquot Samples - It is the laboratory's standard procedure to aliquot samples gravimetrically. Check the Method Comments to see if volumetric aliquotting is required.

10.10.1 Aliquot Gravimetrically - Weigh the full sample bottle (either the original container or the filter flask) to the nearest gram using a top loading balance, and record the weight on the benchsheet. After the extraction, weigh the empty sample bottle, and record the weight. Subtract the empty bottle weight from the full bottle weight and record the difference as the sample volume in mL. If the initial volume is less than the nominal volume by 20% or more, prepare an NCM. If there is any indication that the density of the sample is not 1 g/mL, measure the density of the sample using a calibrated pipette and refer to Section 11.2. Proceed to Section 10.11.

10.10.2 Aliquot Volumetrically - For each sample, rinse a Class A graduated cylinder (500 mL for 8330, 1,000 mL for 8321) once with acetonitrile and twice with reagent water. Carefully pour the sample from the original container into the graduated cylinder, making sure that if any sediment is present, it is not transferred to the graduated cylinder. Record the volume to the nearest 10 mL. If the initial volume is less than the nominal volume by 20% or more, prepare an NCM. Transfer the sample back to the original sample container. Rinse the graduated cylinders with reagent water and add the rinse to the sample. If sediment was present in the original sample container the sample can be transferred from the graduated cylinder into a new amber glass bottle. Write an NCM to document that the rinse of the original sample container could not be performed. Place the original sample bottle beside the new sample bottle so a

second analyst can check that the correct sample was aliquotted. Proceed to Section 10.11.

10.11 Salt the Samples for method 8330A and 8330B -- For methods 8330A and 8330B add 25 g of baked Sodium Chloride to every field sample and QC samples. This is done to facilitate the extraction of picric acid.

10.12 Add Surrogate Standards to Sample Containers - Add surrogate standard to each field sample and QC sample using a calibrated pipette. Reference WI-DV-0009 to determine the correct surrogate standard and the correct volume to be used. The surrogate standard should be added to the sample in the original sample container unless the sample had significant sediment, was received in the improper container, or aliquoted volumetrically. Record the ID of the standard and the pipette used on the bench sheet.

10.13 Add Spike Standards to Sample Containers - Add spike standard to each LCS, MS, and MSD sample using a calibrated pipette. Reference WI-DV-0009 to determine the correct spike standard and the correct volume of standard to be used. The spike standard should be added to the MS and MSD samples in the original sample containers unless the sample had significant sediment or was received in the improper container. Record the ID of the standard and the pipette used on the bench sheet.

NOTE: The addition of spikes and surrogates to samples must be done only after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009. Also at this time the witness checks the sample labels to ensure samples are correctly identified.

10.14 Cap the samples and mix to ensure the salt, the surrogate, and the spike standards are mixed into the sample completely.

10.15 Connect the Tubing - Using the tubing that has been rinsed with acetonitrile and water, connect the cartridge to the sample container. If the extraction is being performed directly from the sample container care should be taken if there are solids that have settled to the bottom of the bottle. Clip the tubing so that the end is not resting on the bottom, but suspended above the solids.

10.16 Load the Sample onto the Cartridge

10.16.1 Begin drawing the sample through the cartridge at a rate of approximately 10 mL/minute. The solution should come out of the cartridge as individual drops. If the sample comes out of the cartridge in a stream instead of drops, the elution rate is too fast. 10 mL/minute is approximately 45 drops every 15 seconds.

10.16.2 Do not let the extraction tube go completely dry.

10.16.3 A cartridge is considered clogged if a flow rate of 4 mL/min. cannot be achieved. This is approximately 1 drop every second. If the cartridge clogs during sample loading, a second cartridge can be used for the sample and then extracts are combined. Alternatively, measure the amount of sample successfully extracted, and use that volume for the extraction constant. If this approach is used, then

the surrogate and spike volumes must be corrected for the new initial volume. Narrate with an NCM. See Section 11 on how to calculate the actual surrogate and spike volumes.

10.16.4 Once the sample has been drawn into the tubing, rinse the walls of the sample container with at least 15 mL of reagent water. This will serve as the cartridge post-rinse. After all of the sample and the water used to rinse the container has gone through the line, close the valve and remove the line and the cap from the cartridge. It is important not to let the cartridge go dry, but to leave water in the cartridge.

10.17 Elute the Cartridge

10.17.1 Turn off the vacuum and remove the manifold lid. Wipe the needles dry with a laboratory tissue being careful not to spread contamination from needle to needle. The tissue used can be wetted with acetonitrile. Place the lid on a clean lab tissue. Place the vial holder inside the manifold and place 8 mL amber vials that have been calibrated to 5 mL inside the manifold on top of the vial holder. Replace the manifold lid and be sure that each valve needle is positioned inside a vial.

10.17.2 Using a serological pipette or a bottle-top re-pipettor, add 2.5 mL of 0.1% acetic acid in acetonitrile to each cartridge. Turn on the vacuum pump while the valves on the manifold are still closed, then quickly open and close each valve to create a vacuum in the cartridges. Turn off the vacuum pump and break the vacuum in the manifold. Open the valves to allow the 0.1% acetic acid in acetonitrile to slowly drip gravimetrically (approximately one drop in 5 seconds).

10.17.3 After the solution has stopped dripping, reapply the vacuum to ensure that the last portion of solvent is collected. <u>This is a very important step</u>.

10.17.4 If two cartridges were used, transfer all extracts into one collection vial and evaporate down to approximately 2.5 mL using a N-Evap. Continue to the next step.

10.18 Bring the extract up to the 5 mL final volume

10.18.1 For method 8330A and 8330B, add the calcium chloride solution to adjust the volume of the collected extract to the mark on the calibrated vial.

10.18.2 For method 8321A, add reagent water to adjust the volume of the collected extract to the mark on the calibrated vial.

10.19 Maintenance

10.19.1 As needed, the inside of the manifold block should be cleaned by washing with soap and water, rinsing with acetonitrile, and wiping with a laboratory tissue.

NOTE: The gasket covers should be checked weekly to ensure there are no signs of contamination showing such as discoloration etc, and to ensure that the gasket cover is not losing its seal.

10.19.2 After each use, the valves and tube caps are removed from the manifold, set to the open position and placed in a jar with acetonitrile and placed in a sonication bath for at least 30 minutes. If samples are suspected to be highly contaminated, a 1:1 mixture of acetonitrile and methylene chloride can be used.

10.19.3 Before and after each use, lines are rinsed with acetonitrile, followed by a water rinse.

10.19.4 Visually inspect lines after use and replace if there is any sign of contamination.

10.20 Troubleshooting

10.20.1 If the vacuum in not strong enough, change the seal on the manifold lid. Also check the pressure relief ball and replace it if cracked.

10.20.2 If a sample clogs the cartridge before a significant volume has been extracted, re-aliquot and re-prep the sample at a dilution. This can be done in the same batch, but the re-prep counts as an additional sample towards the 20 sample batch limit.

10.20.3 Consult a supervisor and/or the QA department with unusual sample matrices.

11.0 Calculations and Data Reduction

11.1 Volume of Surrogate or Spike Extracted = (V_{SA}) x (V_E) ÷ (V_I)

Where:

V_{SA} = Volume of Spike or Surrogate originally added.
V_E = Volume of Sample that was extracted through the cartridge
V_I = Volume of Sample that was originally spiked

Example: 0.1 mL of surrogate standard was added to a 253 mL sample.

During the extraction, the cartridge clogged and only 233 mL of sample was actually extracted.

Vol of Surrogate Extracted = 0.1 mL x 233 mL ÷ 253 mL = 0.092 mL

Therefore the initial volume on the benchsheet should be entered as 233 mL and the volume of surrogate should be entered as 0.092 mL.

11.2 Initial Volume

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g/mL)}$$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid/liquid extraction. Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.1 The following waste streams are produced when this method is carried out:

14.1.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.1.2 Flammable solvent waste – Waste Stream C

14.1.3 Aqueous sample waste - Waste Stream X

14.1.4 Methylene chloride – Waste Stream B

14.1.5 Non-hazardous solid waste such as used cartridges can be disposed of in the regular trash.

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3535A, Solid-Phase Extraction (SPE), Revision 1, February 2007.

15.1.2 Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 0, September 1994.

15.1.3 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.4 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

15.1.5 Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 1, January 1998.

15.1.6 Method 8330B, Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography, Revision 2, October 2006.

16.0 Method Modifications:

16.1 Modifications from SW-846 8330

Method 8330 prescribes the shelf life for standards as follows:

Standards	Concentration	Shelf Life
Stock standards	1,000,000 µg/L (1,000 ppm)	One year
Intermediate standards	2.5 to 1,000 µg/L	Thirty days
Working standards	1 to 500 µg/L	Daily

This SOP describes the use of 1,000 µg/L and 500 µg/L standards, which are assigned a six-month shelf life based on TestAmerica's experience with these materials.

16.2 Modifications from SW-846 3535A

16.2.1 Method 3535A prescribes a 1 liter sample volume. This SOP describes a 500 mL sample volume.

16.2.2 Method 3535A prescribes a 10 mL acetonitrile wash followed by a 30 mL water wash. This SOP describes a 12 mL methylene chloride wash followed by a 12 mL acetonitrile wash, followed by a 24 mL water wash. It is the lab's experience that the methylene chloride wash is helpful in removing interferences from the cartridge packing.

16.2.3 Method 3535A prescribes a 5mL acetonitrile elution. This SOP describes a 2.5 mL elution with approximately 0.1% acetic acid in acetonitrile. The lab has demonstrated increased recoveries for tetryl when this slightly acidic elution solvent is used. The extract is then brought to a 5 mL final volume with calcium chloride solution.

17.0 Attachments

None

18.0 Revision History

Revision 9 dated 11 December 2018

- Updated section 2.1 to reference 8321B by LCMSMS.
- Updated the definition of explosive in Section 3.2 with method references
- Corrected SOP reference in Section 7.2
- Removed vacuum draw from section 10.4.1 (preconditioning step for method 8330). Preconditioning should be done entirely by gravity draw for method 8330
- Rearranged sections 10.11 through 10.13 so that addition of salt occurs prior to addition of surrogate and spike standards. This allows salt to dissolve prior to loading onto the cartridge.
- Added note to 10.16.3 to clarify that an NCM should be generated.
- Added clarification to the container rinse in section 10.16.4 and absorbed “Wash the cartridge” section into 10.16.4. The post rinse is now included in the container rinse step.
- Updated MDL policy reference in section 12.1.
- Updated all references to WI-DV-009 to reference correct work instruction name, WI-DV-0009.
- Minor formatting and language corrections throughout

Revision 8 dated 31 October 2017

- Annual Review

Revision 7 dated 31 October 2016

- Updated section 3.1 to reflect consistent definition verbiage and reference to the QAM
- Added the paragraph associated to section 6.0 to document supporting equipment IDs
- Revised section 6.2.7 to indicate that non-PFTE tubing is used in lieu of Teflon lines and other verbiage.
- Updated the verbiage in section 6.3 to reflect current Software & Hardware information
- Revised section 7.8.2 removing requirement of sub-aliquoting the standard to match what the standards SOP outlines.
- Updated section 9.1 and subsection to reflect current and consistent verbiage regarding laboratory QA/QC requirements
- Removed reference to AFCEE in section 9.1.2
- Added LCSD required when no MS/MSD to sections 9.5.1 Note and 9.6.2
- Added the note to section 10.6 regarding the need to rotate glassware and extraction positions
- Added the note to section 10.20.1 to reflect the need of gasket cover maintenance
- Added section 10.20.4 to visually inspect lines after use and replace if contaminated
- Updated section 12.1, 12.2 and 12.3 to reflect current and consistent verbiage regarding laboratory method performance requirements.

Revision 6 dated 12 October 2015

- Section 16 was revised to describe method modifications in more detail.

Revision 5 dated 14 April 2015

- Annual Technical Review
- Removed reference to DV-LC-0025 “Analysis of Picric Acid by LC/MS/MS” from Section 1. The laboratory no longer maintains this method and SOP. Therefore the procedure was revised to remove the requirement to add 6M HCl to the samples for method 8321A.
- The procedure was revised to change the elution solvent from acetonitrile to 0.1% acetic acid in acetonitrile. This was done to improve the recoveries of tetryl. Section 4.4 was added to document the interference that was observed in tetryl which caused it not to fully elute when acetonitrile was used as the elution solvent. Added Section 16.3 to state this method modification.
- Section 4.5 was added to discuss how samples with sediment can interfere with the procedure.
- Section 10.16.1 was revised to better describe the proper rate of sample loading on the SPE cartridge.
- Added Section 15.5, reference for Method 8000C (required in Arizona)

Revision 4 dated May 30, 2014

- Annual Technical Review.
- Removed reference to DV-LC-0028 “Analysis of Nitroaromatic and Nitroamine Explosive Compounds by APCI/LC/MS/MS” from Section 1. The laboratory no longer maintains this method and SOP.
- Section 6.1 was revised to remove the requirement to disassemble the valves before soaking them in solvent.
- Section 7.2 was revised to require the testing of acetonitrile on a lot basis.
- Section 7.3 was revised to correct how the 6M HCl is prepared. The reagent is prepared using reagent water, HPLC grade water is not necessary.
- Section 7.9 was revised to instruct the analyst to only remove a portion of the LCS standard from the storage freezer each day.
- Updated table in section 8.0 and removed sections 8.1-8.3 as they were redundant with all info not in table.
- Revised Section 9.1 to state prep SOPs do not include acceptance criteria for QC samples – reference analytical SOPs.
- Section 9.1.2 was revised to state that this procedure meets all criteria of DoD QSM 5.0.
- Removed “Acceptance Criteria” and “Corrective Action” information from Section 9. This information can be found in the analytical SOPs.
- Revised Section 10.3 to include a methylene chloride rinse of the cartridge for method 8330. This was done to remove interferences. Methylene chloride was added to Section 5 Safety, Section 7 Reagents, and Section 14 Waste Management.
- Revised Section 10 to instruct the analyst to filter all samples with visible sediment, removing the instructions to decant samples that have settled solids.
- Added instructions to cap and mix the samples after the addition of the surrogate, spike, and acid or salt.
- Revised the instruction in Section 10.17 on how to prevent cross-contamination from the needles after the sample has been loaded onto the cartridge and before the cartridges have been eluted.
- Added sub-sections for Maintenance and Troubleshooting to Section 10 per DoD QSM 5.0.
- Updated section references to reflect revisions.
- Formatting changes throughout.

Revision 3 dated May 30, 2013

- Annual Technical Review
- Corrected formatting and grammatical errors.
- Section 6.1 was revised to give more detail on the cleaning of the valves and tube caps. The valves and tube caps should be sonicated in a jar of acetonitrile for at least 30 minutes before use. The jar uses should be replaced at least weekly.
- Section 6.2 was revised to remove aluminum foil and dishes as part of the supply list. These items are not used in the procedure.
- The procedure was revised to define reagent water as water coming from the ELGA purification system. The option for bottled HPLC water was removed. This was done to ensure consistency in the procedure and to reduce the cost and environmental impact of bottled water (shipping, empty bottle waste).
- Sections 10.6, 10.8, 10.9, and 10.10 were revised to instruct analysts to use disposable amber bottles instead of solvent-rinsed beakers. This was done to reduce the chance of cross-contamination and to reduce solvent usage and waste.

Revision 2.1 dated May 25, 2012

- Annual Technical Review
- Corrected formatting and grammatical errors.
- Updated Section 6.1 to incorporate the addition of 0.1 mL tips used to add surrogate and spike standards to samples.
- Revised Section 7.3 to state that 6M HCl Solution is added prior to extraction, when extracting for method 8321A or 8321B.
- Revised Section 7.7 to state that baked Sodium Chloride is used when extracting for method 8330A or 8330B.
- Updated Section 10.3 to more accurately describe the rinsing of all tubes and ports on the vacuum manifold.
- The instructions for aliquotting samples was moved to be before the instructions on surrogating and spiking the samples to match actual lab practice
- Updated Section 10.16 to include a reference to the Calculation Data Reduction Equation found in Section 11.
- Revised Section 11 to include a detailed equation, with example, on how to calculate the actual surrogate and spike volumes of samples when the extraction cannot be completed after use of a second cartridge.

Earlier revision histories have been archived and are available upon request.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Soil Samples [SW-846 8330A & 8330B]

Approvals (Signature/Date):

Andrew Pepping — 12/12/18	Doug Gomer — 12/12/18
Technical Specialist	Health & Safety Manager / Coordinator
Roxanne Sullivan — 12/12/18	Richard Clinkscales — 12/12/18
Quality Assurance Manager	Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the extraction of nitroaromatic and nitroamine explosive residues from soil samples. This procedure is based on SW-846 8330A and 8330B, but can also be performed on samples for analysis by method SW-846 8321A.

1.2 This procedure does not describe the analysis of the extracts. For those details, see the following SOPs:

1.2.1 DV-LC-0002, *Analysis of Nitroaromatic and Nitroamine Explosive Compounds by HPLC.*

1.2.2 DV-LC-0010, *Analysis of Nitroaromatic and Nitroamine Explosives Compounds by APCI/LC/MS.*

1.2.3 DV-LC-0025, *Analysis of Picric Acid by LC/MS/MS.*

1.3 Application of 8330A versus 8330B

1.3.1 This procedure is for extraction by either Method 8330A or 8330B. The most important differences in the two source methods are the more rigorous sample collection and preparation measures in 8330B, which are designed to produce more representative results. The more rigorous 8330B process is specifically intended to complement the incremental field sampling process described in Appendix A of method 8330B. If incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory homogenization and subsampling effort 8330B requires may add little or no improvement in the overall precision of results.

1.3.2 A larger sample size is used for 8330B (10 g) than is used for 8330A (2 g). A larger sieve size is used for 8330B (10 mesh) than is used for 8330A (30 mesh).

2.0 Summary of Method

Solid samples are air dried to a constant weight and sieved. Soil agglomerates are broken with a mortar and pestle, sieve shaker, or mechanical disaggregator. For samples requiring the more rigorous homogenization techniques found in method 8330B, the analyst employs a ring and puck grinder. The samples are extracted with a 0.1% acetic acid in acetonitrile mixture on a shaker table.

3.0 Definitions

3.1 Definition of terms used in this SOP may be found in the Glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) or SOP DV-QA-003P, *Quality Control Program.*

3.2 **Explosives:** As used in this SOP, the term "explosives" refers specifically to the analytes listed in Table 1. These include compounds that can be readily detonated with heat, shock, or ignition, such as nitroglycerin, RDX, and TNT. It also includes production by-products and degradation products of true explosives.

3.3 **TALS:** TestAmerica Laboratory Information Management System

3.4 **ISM:** Incremental Sampling Methodology - This is a requirement of method 8330B and describes the technique used to take a 10 g aliquot from a sample in at least 30 increments.

3.5 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in TALS, and is the primary basis of prioritizing work.

3.6 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.7 **Grinding Batch:** A grinding batch is up to 20 samples processed through the same grinding procedure. When using the ring and puck mill, the grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner, due to the expiration of the grinding LCS.

3.8 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client.

3.9 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in TALS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.10 **Aliquot**: A part that is a definite fraction of a whole; as in "take an aliquot of a sample for testing or analysis." In the context of this SOP, "aliquot" is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2 Contamination by carryover can occur when a low-concentration sample is extracted immediately following a high-concentration sample.

4.3 Samples from an ammunition plant or depot usually contain analytes that were deposited via water and leaching and therefore are more uniformly dispersed. Therefore, as per SW-846 8330B Section 11.1.4.2, ring and puck is not necessary.

4.4 Samples from firing ranges and impact zones can contain particles of explosives at a variety of sizes, shapes, and compositions. Therefore the entire sample must be processed through a ring and puck prior to removal of the subsample for analysis. Samples collected at the firing point can contain nitrocellulose fibers. These fibers present a special problem in the grinding step. In order to get the fibers to release the target analytes they must be very finely ground. For these samples only the ring and puck should be used. The client needs to be consulted when selecting a grinding mechanism.

4.5 Tetryl decomposes rapidly with exposure to heat as well as methanol/water solution. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately. When tightening caps on 40 mL glass vials, cut resistant gloves must be worn.

WARNING: Soil samples with explosive concentrations greater than 2% cannot be accepted by the laboratory unless they have moisture content of 25% or greater. Under no circumstances shall a soil sample with an explosive concentration greater than 10% be accepted by the laboratory.

5.2.1.1 If a sample is expected to have an explosive concentration $\geq$ 2% (but less than 10%), the EH&S Coordinator and Group Leader shall be notified before any work is performed. Additional safety precautions may be implemented as required due to high concentrations of explosives.

5.2.1.2 Soil samples with high concentrations (between 2 and 10%) of explosives should not be ground using a mortar and pestle. Visual observation of a soil samples is important prior to grinding samples. Any samples containing metal fragments, powders, waxy appearing pieces, or other suspicious material should be brought to the attention of the Group Leader and the EH&S Coordinator before proceeding with the procedure. Bypassing the grinding step and proceeding to solvent dilution is an alternative for samples that are determined to be unsafe to grind.

5.2.2 Anyone working in the grinding room needs to be enrolled in the Hearing Conservation Program. See SOP DV-HS-0010 for details. Personnel operating the grinding equipment are required to wear ear plugs when the equipment is turned on. When standing next to the Humbolt mechanical grinder described in Section 6.1.11 during operation, the decibel levels are above 80 decibels, therefore anyone operating the grinder must be enrolled in the Hearing Conservation Program and wear hearing protection. While the grinder is running, the decibel levels in the room are below 80 decibels, therefore personnel not enrolled in the Hearing Conservation Program can be in the room. Hearing protection is always available to every analyst and they are encouraged to use it.

5.2.3 Operations involving handling samples outside of sealed containers are conducted in ventilation hoods to avoid exposure to dust. Dust masks are available for use, but are optional.

5.2.4 Operations involving the grinding of radioactive samples can be particularly hazardous due to the increased potential for exposure from airborne dust. If a sample is labeled as a “CAT 1”, “CAT 2”, “CAT 3” or “CAT 4” sample, and requires grinding thru the ring and puck, contact the RSO immediately.

5.3 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating. ***This list does not contain all materials used in the method.*** The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
ACETONITRILE	Flammable Poison	40 PPM – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
METHANOL	Flammable Poison	200 PPM - TWA	A slight irritant to the mucous membranes. Toxic effects are exerted upon the nervous system, particularly the optic nerve. Symptoms of overexposure may include

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
	Irritant		headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause the skin to be become dry and cracked. Skin absorption can occur, symptoms may parallel inhalation exposure. Irritant to the eyes.
ACETIC ACID, GLACIAL	Corrosive Poison Flammable Liquid and Vapor	10 PPM - TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
(1) ALWAYS ADD ACID TO WATER TO PREVENT VIOLENT REACTIONS. (2) EXPOSURE LIMIT REFERS TO THE OSHA REGULATORY EXPOSURE LIMIT.			

6.0 Equipment and Supplies

6.1 Equipment

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1.1 Balance, capable of measuring ± 0.01 g. Calibration checked per SOP DV-QA-0014.

6.1.2 Orbital shaker table, capable of maintaining 150 rpm for 18 hours.

6.1.3 Pipettor with disposable 1.0 mL tips, accurate to ± 2%, calibration checked daily in accordance with SOP DV-QA-0008.

6.1.4 Bottle-top pipettor, able to dispense 8 to 20 mL, accurate to ± 2%, calibration checked daily in accordance with SOP DV-QA-0008. If the pipettor does not have a digital display, then the calibration check should be performed whenever the pipette is adjusted.

6.1.5 Ring and Puck - for the grinding of soils per method 8330B

The grinding bowl and puck are cleaned after each use by washing with soap and water with a plastic brush, rinsing with hot tap water, rinsing with DI water, and then rinsing with a 10% acetonitrile solution in acetone. A final wipe down of the bowl and puck while still wet with solvent is done with a Kimwipe (TNT in particular is reported to be prone to adhering to steel surface). In addition, sand blanks are used to monitor potential carry-over for each batch of samples (see Section 9.10.1 for details).

6.1.6 Sample drying systems

6.1.6.1 Trays – “baker’s rack” type of stack for the air drying of soils per method 8330B Trays – “baker’s rack” type of stack for the air drying of soils per method 8330B

6.1.6.2 Drying tower – custom built tower similar to "baker’s rack” type stack for air drying soils, including drying fans and air filters.

6.1.7 Sieves, 10 and 30 mesh – Sieves are cleaned after each use by washing with soap and water and a green plastic brillo pad, (be careful not to damage the mesh), rinsing with hot tap water, rinsing with DI water. Prior to use, the sieves are rinsed with 10% acetonitrile in acetone and wiped with a Kim Wipe. Sieves are allowed to dry in a hood prior to use.

6.1.8 Receiver pans and lids – Receiver pans are cleaned after each use by washing with soap and water, rinsing with hot tap water, rinsing with DI water. Prior to use, the receiver pans are rinsed with a 10% acetonitrile in acetone and wiped dry with a Kim Wipe.

6.1.9 Sieve shaker – used to facilitate the sieving of large sample volumes.

6.1.10 Mortar and pestle – cleaned after each use by washing with soap and water, rinsing with hot tap water, and then rinsing with DI water. Prior to use, the mortars and pestles are rinsed with 10% acetonitrile in acetone and wiped with a Kim Wipe and allowed to dry in a hood prior to use.

6.1.11 Mechanical Disaggregator – Humbolt Manufacturing Part Number H-4199. Used in place of a mortar and pestle to quickly reduce cakes of dry soil. The disaggregator reduces soil agglomerates and sieves the soil through a 10 mesh sieve. The mechanical disaggregator is used to break up soil agglomerates, but it is not an alternative to Ring and Puck. The mechanical disaggregator is cleaned after each sample by removing the hopper. The hopper is washed with soap and water, rinsed with tap water, rinsed with DI water, and then rinsed with 90:10 Acetone:Acetonitrile. The Hopper is then wiped dried with a laboratory tissue. The hammers and body of the disaggregator are cleaned after each sample by rinsing with DI water and wiping dry with a laboratory tissue.

6.2 Supplies

6.2.1 Glass vials, various sizes.

6.2.1.1 Amber glass, 40 mL, with Teflon-lined screw caps for the sonication of soil samples.

6.2.1.2 Amber glass, 8.0 mL, with Teflon-lined screw caps, for the storage of final extracts.

6.2.2 Aluminum foil and aluminum dishes.

6.2.3 Parchment paper

6.2.4 0.2-µm PTFE syringe filters and disposable syringes.

6.2.5 Wooden spatulas – used to lay samples out to dry.

6.2.6 Subsampling tools:

6.2.6.1 Scored paper scoops (TAL-0150 and TAL-0150 LARGE from Commodity Management Services)

6.2.6.2 Plastic sample scoops – square-ended

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Ottawa Sand – baked at 400 °C for at least 4 hours.

7.2 Acetonitrile, CH_3CN - HPLC grade (ACN). Each lot is tested per CA-Q-S-001 DV-1.

7.3 Soil Extraction Solvent – approximately 0.1% acetic acid in acetonitrile – Open a new 4-liter bottle of acetonitrile and add 4 mL of acetic acid, then cap and mix. This reagent is given a 1 year expiration date.

7.4 Standards

Please reference SOP DV-OP-0020 for information regarding the surrogate and spike standards used in this procedure.

7.5 Grinding LCS Bulk Material

A standard is purchased in a matrix of -20/+70 Sieved Soil that contains the compounds at the concentrations listed in Table 2. This standard comes packaged in 500 g containers. This standard is stored in a refrigerator at 0 °C to 6 °C and is given a 1 year expiration date. After grinding the ground LCS is stored refrigerated and has a three day expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Soil samples to be extracted by method 8330A for analysis by method 8330A should be collected in eight-ounce wide mouth jars with Teflon-lined caps. When sampling for DoD projects that must comply with DoD QSM requirements for drying and sieving the entire contents of a soil sample container, a separate container should be used to collect a soil sample for this analysis.

8.2 For soil samples to be extracted by method 8330B for analysis by either method 8330B or method 8321A, it is not uncommon to receive samples of 1 kg or more. Samples may be shipped in wide mouth jars or clean plastic bags.

8.3 Sample extracts must be stored refrigerated in amber glass containers at ≤ 6 °C and not frozen.

8.4 Soil and sediment samples should be air dried at ambient temperature until dry enough to sieve. See Section 10.3 for details. Once the sample is air dried, the sample can be stored at room temperature.

8.5 All soil and sediment samples must be extracted within 14 days of collection and analyzed within 40 days after extraction begins.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass/ plastic	4 grams (8330A)/up to 1 kg (8330B)	Cool ≤ 6 °C	14 Days	SW846 8330A/B

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the TestAmerica LIMS (TALS) and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument. See QC Policy DV-QA-003P for further details.

Grinding Batches – A grinding batch is up to 20 samples processed through the same grinding procedure. When using the ring and puck mill, the grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner, due to the expiration of the grinding LCS.

9.4 Method Blank (MB)

A method blank (MB) must be prepared and analyzed with each batch of samples. The MB consists of Ottawa sand with surrogates added. The MB is created at the time of extraction after the samples have been dried, sieved, and ground and is then carried through all extraction and analysis steps. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

One LCS must be analyzed with each batch of samples. The LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. The LCS is prepared by spiking the analytes of interest into Ottawa sand. The LCS is created at the time of sample extraction after the samples have been dried, sieved, and ground. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

NOTE: DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD is not required unless requested by the client.

9.6 Matrix Spike Sample (MS) and Matrix Spike Duplicate (MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The soil matrix spikes are created at the time of extraction. Spikes and surrogate compounds are added after the sample has been dried, sieved, and ground. One MS/MSD pair must be processed for each preparation batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

If insufficient sample volume is available for MS/MSD, a LCSD must be performed and an NCM must be written.

NOTE: DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD is not required unless requested by the client.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, DU, TRL, MS, and MSD) is spiked with surrogate compounds.

9.8 Sample Duplicate (DU)

A duplicate sample is required after ring and puck grinding is performed. A duplicate sample is also required for method 8330B, even if grinding is not performed. A sample duplicate is a second aliquot of one of the samples in the batch. Field blanks cannot be used for duplicate testing. The results for duplicates are reported separately, and cannot be averaged when reporting results. Sample duplicate results are used to evaluate the precision of the method. As such, results should be greater than or equal to the RL for a valid statistical comparison.

9.9 Sample Triplicates (TRL)

A triplicate sample is required after ring and puck is performed. A triplicate sample is also required for method 8330B, even if grinding is not performed. The lab will determine the %RSD as defined below. Results for the %RSD as well as the individual replicate results will be reported to the client. The method suggests that the %RSD for the subsampling error is acceptable if it is < 10%. For DoD QSM 5.1, the %RSD is acceptable if it is < 20% for results above the LOQ.

The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{s}{\bar{C}} \times 100\%$$

Where s is the standard deviation of the average concentration and is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (C_i - \bar{C})^2}{n-1}}$$

In the event that the laboratory is requested to perform the evaluation of field replicate precision, three field replicates designated by the client will be processed through the entire homogenization and extraction steps. The %RSD for these replicates will be calculated as indicated above and reported to the client.

9.10 Grinding Blank (GB)

9.10.1 Ring and Puck Grinding Blanks

Before each sample is processed through the ring and puck mill, the ring and puck will be cleaned per Section 6.1.5. Then approximately 200 g of Ottawa Sand will be ground. This ground sand will be saved and labeled with the sample ID of the next sample ground with the suffix “blank”. After a batch of samples has been processed through the ring and puck, a composite will be generated using sub-aliquots from all blanks ground

before the samples. This is done by placing approximately 1 tablespoon of material from each of the individual sample blanks in a clean re-sealable plastic bag. The bag is then sealed and the material is mixed and homogenized by shaking and kneading the bag. A 10 g aliquot is then removed from the bag and labeled as the batch grinding blank. This composite is extracted and analyzed in the same manner as the field samples.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank producing positive results for the same contaminant, must be reprocessed and reanalyzed. If un-ground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative.

9.11 Grinding LCS (LCSSRM)

One Grinding LCS must be ground and analyzed with each batch of samples that are processed through the ring and puck. The Grinding LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. The Grinding LCS is prepared by grinding a 500 g aliquot of the Grinding LCS Bulk Material described in Section 7.5 without having air-dried the material before hand. The Grinding LCS must be ground using the same grinding apparatus (ring and puck) as the samples were ground. The Grinding LCS is used to monitor the effects of the grinding process on the analytes of interest. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Corrective Action: If the Grinding LCS fails the acceptance criteria, samples associated with the Grinding LCS must be reprocessed and reanalyzed. If un-ground sample is not available, then the results of the grinding LCS must be described in a non-conformance memo and discussed in the final report case narrative.

10.0 Procedure

NOTE: Rotate sieves and any applicable equipment; do **not** use specific sieves or equipment for the MB and LCS/LCSD.

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 **Dry the Samples –** Refer to the Flowchart in Appendix 1 and the batching instructions in Appendix 3.

10.3.1 Check the Method Comments to see if the samples are for a project with the Department of Defense (DoD), if yes, then the entire contents of the sample container must be dried. Check TALS to make sure the client sent more than one container if additional tests are being requested. If additional tests are logged and the client only sent one container, the Project Manager should be notified.

10.3.2 If the sample is logged for method 8330B, or for an ISM method, then the entire contents of the sample container must be dried. Check each sample to make sure the client sent more than one container if additional tests are being requested. If additional tests are logged and the client only sent one container, the Project Manager should be notified.

10.3.3 If the samples do not fall under the descriptions given in Section 10.3.1 or Section 10.3.2 then only a portion of the sample container needs to be dried. In these cases, lay out at least 20 g to dry.

10.3.4 Depending on the sample size, the samples are laid out in aluminum pans, or on large trays lined with aluminum foil to dry. Some clients may request metals analysis on the dried samples. In those cases, samples are laid out on parchment paper.

10.3.5 Spread the samples out in a thin layer to facilitate drying. Use a disposable wooden spatula to break up any clumps and agglomerates.

10.3.6 The tray or pan that the sample is laid out into is labeled with the sample ID. A second analyst checks to make sure that the labels on the tray or pan match the labels on the client sample container to ensure samples are not accidently mixed up. This check is documented in TALS.

10.3.7 Place the samples in a hood or well ventilated area at room temperature. Document in TALS the date and time the samples were laid out to dry. If the samples are very wet, a fan can be used to help facilitate the drying process, but care should be taken so that the air flow is not strong enough to cause cross-contamination between samples. An electronic temperature recording device records the temperature of the room and the data is downloaded weekly.

10.3.8 When the samples appear to be dry enough that they can be sieved without caking, subsample approximately 15 grams into an appropriate weighing vessel and record the exact weight, the date, and the time (see Appendix 5). Set this 15 gram aliquot (still in the weighing vessel) next to the rest of the drying sample. Take care to use an appropriate weighing vessel for the

analytical methods requested, as the aliquot removed in this step will still be included in the volume used for ISM (i.e. Do not use an aluminum weigh boat for samples requiring metals analysis).

10.3.9 After 2 hours, reweigh the aliquot in the same weighing vessel and record the exact weight, the date, and the time. If the weight of the sample is within 10% of the previous weight, proceed to Section 10.4.

10.4 Sieve the Samples - Refer to the Flowchart in Appendix 2.

10.4.1 If the client requirements specify a particular sieve size, those instructions take precedence.

10.4.2 If the sample is logged for prep method “8330_P_2g” then a 30 mesh sieve should be used.

10.4.3 If the sample is logged for prep method “8330_Sonc_10g” then a 10 mesh sieve should be used.

10.4.4 Some clients will request metals analyses to be performed on the sieved sample. In those cases, a stainless steel sieve should be used. Brass sieves should be avoided.

10.4.5 Clean the sieves prior to use following the instructions in Section 6.1.7.

10.4.6 Some samples may require the use of a mortar and pestle or a mechanical disaggregator to break up dried clumps. Refer to Sections 6.1.10 and 6.1.11 on how to clean and rinse the mortar and pestles and the mechanical disaggregator before use.

10.4.7 Record the weight of the entire dried sample in the Worksheet tab in TALS. This is a requirement for DoD QSM 5.0 and 5.1.

10.4.8 Sieve the entire dried sample through the appropriate sized sieve. Care must be exercised not to eliminate soil agglomerates during this step. The soil can be broken into small pieces with a gloved hand or another instrument (a wooden spatula for example). If a gloved hand is used, care should be taken to change out gloves in between samples so not to cross-contaminate samples.

10.4.9 Remove large rocks, vegetation, and twigs that do not pass through the sieve. Mosses and other types of fine vegetation should be physically shredded while sieving to release trapped soil and residues. The only materials that should be eliminated by sieving are rocks and vegetation. All soil must be broken up to pass through the sieve.

10.4.10 Place any soil that does not pass through the sieve into a clean mortar. Break up soil agglomerates using the pestle. Or as an alternative use the mechanical disaggregator. Be sure to break up all soil so that it can pass

through the sieve. Only extraneous material such as rocks and vegetation should be removed with the sieve. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.4.11 Collect all of the material that passes through the sieve on a clean piece of foil or parchment paper.

10.4.12 An automatic sieve shaker can be used to help facilitate the sieving of samples. A receiver pan is placed under a sieve and the sample is added to the sieve. Then a lid or another receiver pan for a second sample is placed on top. The stack is then clamped inside the sieve shaker for no more than 30 minutes. Inspect the samples to ensure that only extraneous material such as rocks and vegetation should be removed with the sieve. If needed use a mortar and pestle to break up soil agglomerates. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.5 Grind the Samples - Refer to the Flowchart in Appendix 2.

10.5.1 If the samples are <u>not</u> logged with a pre-prep method of "ISM_DD_SI_PM_SS," skip this section and proceed to Section 10.6.

10.5.2 <u>Ring and Puck Grinding</u> *Samples logged for "ISM_DD_SI_PM_SS"*

10.5.2.1 See Section 6.1.5 on how to clean the ring and puck dish.

10.5.2.2 If the sample is logged for ring and puck grinding, a grinding blank per Section 9.10.1 consisting of baked Ottawa sand will be processed through the ring and puck dish <u>before</u> each sample. These individual blanks will be composited into one grinding blank for the associated samples and will be analyzed in addition to the normal extraction blank.

NOTE: When preparing the grinding blanks, it is not necessary to do five 60-second grinds. One 60-second grind of the Ottawa sand is sufficient.

10.5.2.3 After a grinding blank has been processed through a ring and puck dish, that blank is labeled as the blank associated to the next sample processed through that same dish. Do not clean the ring and puck dish after the blank.

10.5.2.4 Prepare a grinding LCS per Section 9.11 with every batch. The grinding LCS will be analyzed in addition to the normal extraction LCS.

NOTE: A grinding batch will consist of no more than 20 samples that have been ground within three days of each other. The grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner**.** A grinding batch must have one Grinding LCS, and at least one Grinding Blank. If more than one Grinding Blank is prepared, it must be very clear on the benchsheet which individual sample blanks were used to build each Grinding Blank.

10.5.2.5 In a hood, transfer the sample into a clean ring and puck dish. Do not overfill the dish (approximately 300 g of sample can fit in one dish). If needed, grind the sample in 300 g or smaller increments and recombine after all sample has been ground. The entire sample must be ground. Place the dish securely in the holder and close the door on the machine. Grind the sample in five 60-second periods with a one minute cooling time between grinds for a total of 5 minutes of grinding. Remove the dish and in a fume hood, open the lid and inspect the sample. It should be the consistency of flour. The consistency of the material is checked by pinching some between two fingers of a gloved hand and feeling for grit and by looking for any un-ground fibers. If grit is detected or if fibers are observed, additional grinding is needed.

NOTE: During the one-minute cooling time, the dish should be placed in a shallow ice water bath to facilitate cooling. Be sure the bath is shallow enough so that water does not get inside the dish.

10.5.2.6 If the sample reaches a flour-like consistency before all 5 one-minute grinds have been completed, then it might be beneficial to <u>not</u> perform all 5 grinds in order to avoid excessive heat and to avoid packing the sample onto the side of the grinder. If the analyst inspects the sample and it has a flour-like consistency before all 5 grinds are completed, they can make the decision to stop after less than 5 grinds. An NCM should be written to document the deviation from the source method and the reasoning.

NOTE: If multiple 300 g increments are used for grinding and the sample is recombined, it has been shown through Duplicate/Triplicate QC results that the sample is non-homogenous. To re-homogenize the sample, place all volume in to a clean plastic bag, seal, and carefully shake the bag for 1-2 minutes until the sample is thoroughly mixed. Lay out the sample back on the

foil/parchment paper. This must be done on all samples regardless if this sample will be used for DU/TRL QC.

10.6 Aliquot the Samples

10.6.1 All aliquots should be taken using a subsampling tool described in section 6.2.6. This is done to ensure that finer sample material does not fall off of the sampling tool, as can happen if a spatula was used instead. This is particularly necessary when samples are not ground to a consistent grain size using the ring and puck.

10.6.2 **2 Gram Aliquot – Extraction Method "8330_P_2g"** – Remove the cap from a labeled 40 mL amber vial and place the vial on a balance and tare. Spread the entire sample out to a thickness no greater than 1 cm. Use a disposable subsampling tool to build a 2.0 g to 2.2 g aliquot by taking at least five small portions from random locations through the entire thickness of the sample. Record the exact sample weights on the benchsheet and cap the vial with a Teflon™ lined lid. Save the remaining soil for possible re-extraction. Create an LCS and a method blank by placing 2.0 g to 2.2 g of baked Ottawa sand in labeled vials. Record a nominal weight of 2 g in the initial volume field, then record the actual weight to the nearest 0.1 g in the notes column.

10.6.3 **10 Gram Aliquot – Extraction Method "8330_Sonc_10g"** – Remove the cap from a labeled 40 mL amber vial and place the vial on a balance and tare. Spread the entire sample out to a thickness no greater than 1 cm. Use a disposable subsampling tool to build a 10 g to 11 g aliquot by taking at least thirty small portions from random locations through the entire thickness of the sample. Record the exact sample weights on the benchsheet and cap the vial with a Teflon™ lined lid. Save the remaining soil for possible re-extraction. Create a LCS and a method blank by placing 10 g to 11 g of baked Ottowa sand in labeled vials. Record a nominal weight of 2 g in the initial volume field, then record the actual weight to the nearest 0.1 g in the notes column. If the samples were ground create a grinding blank per Section 9.10, and take an aliquot from this composite. Aliquot the grinding LCS as you would a sample.

10.7 Add Surrogate, Spikes, and Solvent to the Samples

10.7.1 Refer to WI-DV-0009 for the correct surrogate and spike standards to use and the correct volume.

10.7.2 The surrogate and spikes standards are kept in a freezer, but should be allowed to come to room temperature before use. Record the ID of the standard and pipette(s) used on the benchsheet.

10.7.3 The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009.

10.7.4 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.7.5 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard to each sample and each QC sample.

10.7.6 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard to each LCS and MS/MSD.

NOTE: Do not add the spike standard to the grinding LCS. The grinding LCS is created using the material described in Section 7.5 and already contains the analytes of interest.

10.8 Add Extraction Solvent

10.8.1 2 Gram Extraction – Extraction Method "8330_P_2g"

10.8.1.1 Taking into account the volume of surrogate and spike standard added to each sample, bring the extract volume up to 10 mL with the soil extraction solvent described in Section 7.3. Use either a 10 mL Class A graduated cylinder or a bottle top pump that has been calibration checked.

Example: If 0.5 mL of surrogate standard was added to a sample, add exactly 9.5 mL of the soil extraction solvent.

Example: If 0.5 mL of surrogate standard and 0.5 mL of spike standard was added to a LCS, add exactly 9 mL of the soil extraction solvent.

10.8.1.2 Proceed to Section 10.9.

10.8.2 10 Gram Extraction – Extraction Method "8330_Sonc_10g"

10.8.2.1 Taking into account the volume of surrogate and spike standard added to each sample, bring the extract volume up to 20 mL with the soil extraction solvent described in Section 7.3. Use either a 25 mL Class A graduated cylinder or a bottle top pump that has been calibration checked.

Example: If 1 mL of surrogate standard was added to a sample, add exactly 19 mL of the soil extraction solvent.

Example: If 1 mL of surrogate standard and 1 mL of spike standard was added to an LCS, add exactly 18 mL of the soil extraction solvent.

10.9 Extract the Samples

10.9.1 Cap vial with a Teflon-lined cap, vigorously hand shake the vial for one minute, or until all material is well mixed, and place it in a box. Place the box on the platform shaker so that the vials are lying on their side. Set the platform shaker at 150 rpm and allow the samples to be shaken for at least 18 hours. Record the start time on the benchsheet.

10.9.2 After the 18 hour extraction, remove the vials from the shaker table and record the stop time on the benchsheet.

10.9.3 If needed, centrifuge the vial at no more than 2,200 rpm to help separate the solids from the extract. Remove approximately 10 mL of the supernatant solution. Filter the supernatant solution using a 0.2-µm PTFE syringe discarding the first mL into the waste. Filter the remaining supernatant into a labeled 8-mL amber vial.

10.9.4 Submit the extract for analysis to the appropriate analytical lab.

10.10 Maintenance

10.10.1 Approximately once a month, the cover on the Ring and Puck should be removed and any dirt should be cleaned up.

10.10.2 When excessive wear is noted, replace the hammers in the Mechanical Disaggregator.

10.10.3 Occasional lubrication of the Ring and Puck clamp is needed.

10.10.4 The o-rings in the Ring and Puck dishes should be replaced when worn.

10.10.5 Every 6 months the centrifuge should be lubricated and tightened.

10.11 Troubleshooting

Low recoveries for Tetryl in the explosives grinding LCS may be indicative of high temperatures during grinding. Review the cooling step noted in Section 10.5.2.5 in order to minimize the effect of the heat generated during the grinding process.

11.0 Method Performance

11.1 Method Detection Limit (MDL)

A valid method detection limit (MDL) study for each analyte of interest must be performed prior to analyzing samples for the first time and verified annually thereafter. Separate soil MDL studies are performed for 8330A using 2 g and 8330B using 10 g of Ottawa sand. Separate soil MDL studies are performed for explosive

method 8321A using 2 g of Ottowa sand and 8321A using 10 g of Ottowa sand. An MDL study for picric acid by method 8321A is performed using 10 g of Ottowa sand. An MDL study for explosives by 8321 LC/MS/MS is performed using 10 g of Ottowa sand. The procedure for determining detection limits is defined in Policy CA-Q-S-006. Quarterly MDLV and LOQV studies are performed for the DoD program (QSM 4.2 and 5.0).

11.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs that specify the requirement. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

11.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

11.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

11.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

11.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

11.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

11.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

11.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC

has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

13.0 Waste Management

13.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

13.2 The following waste streams are produced when this method is carried out:

13.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

13.2.2 Flammable solvent waste – Waste Stream C

13.2.3 Solid sample waste - Waste Stream D

13.2.4 Waste soil sample vials - Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

14.0 References / Cross-References

14.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

14.1.1 Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 0, September 1994.

14.1.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

14.1.3 Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 1, January 1998.

14.1.4 Method 8330B, Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography, Revision 2, October 2006.

14.2 DoD Environmental Data Quality Workgroup, Frequently Asked Questions (FAQs) Concerning the Implementation of EPA SW-846 Method 8330B, November, 2014.

15.0 Method Modifications:

15.1 Method 8330 prescribes the shelf life for standards as follows:

Standards	Concentration	Shelf Life
Stock standards	1,000,000 µg/L (1,000 ppm)	One year
Intermediate standards	2.5 to 1,000 µg/L	Thirty days
Working standards	1 to 500 µg/L	Daily

This SOP assigns a six-month shelf life to the working level standard based on TestAmerica's experience with these materials. The standards are stored in a freezer.

16.0 Attachments

Table 1. Analyte List

Table 2. Grinding LCS Bulk Material

Appendix 1 Flowchart for Drying Explosive Soils

Appendix 2 Flowchart for Grinding and Sieving Soils

Appendix 3 Instructions for Batching in TALS

Appendix 4 ISM Worksheet

Appendix 5 ISM Constant Weight Worksheet

17.0 Revision History

- Revision 11, dated 12/12/2018
  - Changed references to WI-DV-009 to correct document ID, WI-DV-0009 throughout SOP.
  - Minor formatting and language corrections throughout.
  - Changed references to "mechanical grinder" to "mechanical disaggregator" throughout SOP, including sections 2.1, 6.1.11, 10.4.6, 10.4.9, 10.10.2. This was done to distinguish more clearly between grinding and soil disaggregation.
  - Added 6.1.6.2 to equipment section to include drying tower.
  - Expanded section 6.2.6 to include new subsampling tool.
  - Updated sections 9.1.2 and 9.9 to reference DoD 5.1 rather than DoD 5.0.
  - Removed note after 10.3.9 which allowed for skipping constant weight analysis for non-DoD samples.
  - Added section 10.4.7 to meet DoD requirement that entire dried sample is weighed prior to sieving.
  - Removed use of grinding stones from section 10.4.11.
  - Removed notes regarding client-specific requirements from sections 10.4.10 and 10.4.12, and incorporated these specific requirements into standard procedures for all samples. These requirements include weighing and retaining material that does not pass through the sieve.
  - Updated sections 10.6.1, 10.6.2, 10.6.3 to reference "subsampling tool" rather than

"square-ended scoop."
  - Removed section 10.11 comment regarding securing ring and puck as it no longer requires securing. Added note about troubleshooting low tetryl recoveries.
- Revision 10, dated 31 October 2018
  - Annual Review
- Added 10.3.8 Revision 9, dated 5 October 2017
  - Added wording to 2.1 to clarify that samples are dried to a constant weight.
  - Added 10.3.8 and 10.3.9 and removed 15.2 to include procedure for drying samples to a constant weight.
  - Added Appendix 5
- Revision 8, dated 2 December 2016
  - Added the comment requiring the documentation of equipment IDs to Section 6.1
  - Added Section 6.3 Computer Software and Hardware
  - Added Note to Section 9.5 requiring a LCSD when there is no volume for MS/MSD
  - Added Note to Section 10.0 regarding the rotation of sieves and equipment
  - Updated Section 10.5.2.4 to include the samples should be ground the same length of time as the LCSSRM, renumbered the notes and included a sample grind time exception.
  - Updated Section 10.5.3.5 to include Note 1 from Section 10.5.3.6.
  - Renumbered Note in Section 10.5.3.6
  - Updated Section 10.7.2 to reflect standard SOP
  - Removed the Ball Mill Grinder reference from the entire SOP.
- Revision 7, dated 31 January 2016
  - Annual Technical Review
  - Deleted previous Section 4.3 no longer applied – vegetation and rocks are removed
  - Added Section 4.6 regarding tetryl decomposing with exposure to heat.
  - Added paragraph to Section 7 to contain reagent grade verbiage consistent with other SOPs
  - Section 8.1 – clarified the paragraph to be specifically about method 8330A
  - Added "and not frozen" to section 8.3
  - Revised Section 9.1 to have consistent verbiage and instructions as other SOPs
  - Added Note to Section 9.6 regarding DoD MS/MSD requirements
  - Changed duplicate to triplicate in Section 9.9
  - Clarified instruction to not clean the ring and puck dish after the blank in Section 10.5.3.3
  - Added the weight recording requirements to Sections 10.6.2 & 10.6.3
  - Added "and pipette(s)" to Section 10.7.2
  - Changed the centrifuge speed from 2500 rpm to 2200 rpm and 5mL to 10mL volume of supernatant solution to remove in Section 10.9.3
  - Modified/Rearranged Section 11 to be consistent with other SOPs
  - Removed previous Section 11.1 "Initial Demonstration of Capability"
  - Added current Sections 11.2 "Demonstrations of Capabilities" & 11.3 "Training Requirements"
  - Reformatted Section 14 and added Section 14.2 – reference to DoD Frequently Asked Questions for 8330B
  - Removed references to AFCEE and USACOE throughout document as these programs were incorporated into the DoD program.
  - Removed all 2010 and earlier revision histories
- Revision 6, dated 31 January 2015

  - Annual Technical Review
  - Reformatted SOP.
  - Revised Section 3.7 and Section 9.2 to state that a Ball Mill grinding batch is opened and closed the same day, while a Ring and Puck grinding batch can be open for up to 3 days.
  - Revised Section 5.2.2 to give information on the hazards of the Humbolt grinder.
  - Revised Section 9.9.1 to give more detail on how the Ring and Puck composite grinding blanks are created.
  - Revised Section 10.3.2 and Appendix 1 to state that any sample logged with an ISM method must have the entire sample container dried.
  - Revised Section 10.10 to include maintenance on the centrifuge.
- Revision 5, dated 27 January 2014
  - Annual Technical Review
  - Removed Section 1.2.3, DV-LC-0028 no longer performed.
  - Added detail about sieve size to Section 1.3.2.
  - Edited Section 6.1, subsection “Ball Mill” to allow for un-baked sand to be used in the cleaning of the ball mill stones and to allow the use of 1 pint cans. The section was also revised to change the minimum time the stones have to be tumbled during the cleaning process from 3 hours to 2 hours. This was done based on analyst’s experience.
  - Edited Section 6.1, subsection “Sieves” to state a brillo pad can be used on the sieves so long as the mesh is not damaged.
  - Updated Section 9.1 to reflect current practice, added a comment stating that this procedure meets DoD QSM 5.0 criteria unless otherwise stated.
  - Removed Acceptance Criteria and Corrective Action information to Section 9. This information can be found in the analytical SOPs.
  - Revised Section 9.6 to state that if there is no volume for a MS/MSD, a LCSD must be performed.
  - Added information to Section 9.9 for DoD acceptance criteria for triplicates.
  - Updated sections 10.1, 10.2 and 11.2 to reflect current practice
  - Added a NOTE in Sections 10.5.2.1 and 10.5.3.6 giving instructions on how to ensure the sample is homogenous after it has been split into separate grinding containers and then later re-combined.
  - Added Section 10.5.3.6 giving guidance on what to do if the sample reaches a flour-like consistency before all 5 grinds have been completed. This was done to avoid over-heating samples and packing the sample against the grinding dish wall.
  - Added Section 10.10 Maintenance and Section 10.11 Troubleshooting per DoD QSM 5.0.
  - Updated Appendix 2 and Appendix 3 to reflect the current method names used in LIMS.
  - Formatting changes throughout
- Revision 4, dated 30 October 2012
  - Annual Technical Review
  - Section 4.6 was added to document the adverse affect headspace in the ball mill can has on the grinding LCS.
  - Section 6.1 and Section 10 were revised to include the description of the Spacer Can in the Ball Mill apparatus.
  - Section 6.1 and Section 10 were revised to include the Mechanical Grinder used as an alternative to mortar and pestle.
  - Section 9.6 was revised to state that LCSDs are not required for DoD work.

  - Section 10 was revised to reference the Explosive Review Checklist in WI-DV-0009.
  - Section 10.3.2 was revised to instruct the analyst to eliminate as much headspace as possible during the Ball Mill grinding step.
  - Appendix 3 was revised to give more detail on the steps taken to ensure all pre-ground ISM aliquots are taken before the sample is ground. It was also revised to include the use of the Explosive Extraction Checklist in WI-DV-0009.
- Revision 3, dated 10 October 2011
  - The procedure was revised to have the extraction performed by shaker table instead of cooled sonication bath. This was done to increase lab capacity and to create a more rugged extraction.
  - Section 5 was revised to include the requirement that analysts wear cut-resistant gloves when tightening vial caps.
  - Section 7.2 was revised to include the lot approval process for acetonitrile.
  - Sections 7.5 and 9.3 were revised to mandate a 3 day expiration date on the Grinding LCS after it has been ground.
  - Section 9.8 and 9.9 were revised to require a duplicate and triplicate whenever method 8330B is performed, not just when samples are ground.
  - Section 10.2.2 and 10.2.3 were revised to have the analyst use the prep method instead of the pre-prep method to determine sieve size. This is a simpler determination and matches the flow chart in Appendix 2.
  - Section 10.7.3 was revised to change the speed of the centrifuge to prevent the breakage of the extract vials.
- Revision 2, dated 11 January 2011
  - Details about the surrogate and spike standards used in this procedure have been moved to SOP DV-OP-0020.
  - Revised Section 9 to state that duplicates and triplicates are required when ring and puck or ball mill grinding is performed.
  - Revised the procedure to include instructions and details for the laboratory's new LIMS.
  - Revised Section 4 to give more details on the grinding of samples.
  - The procedure was revised to state that samples should be ground on the ball mill for only 8 hours. At that time, the samples should be inspected and only ground longer if required.
  - Added detail in Section 10.1 about the electronic temperature monitoring device that records the temperature of the drying room.
  - Revised the flowcharts to be flowcharts only and not worksheets. All data is now recorded in TALs benchsheets.
  - Added instructions in Appendix 3 on how to batch samples in TALSs
  - Added the option to use an automatic sieve shaker.

Earlier revision histories have been archived and are available upon request.

Table 1. Analyte List

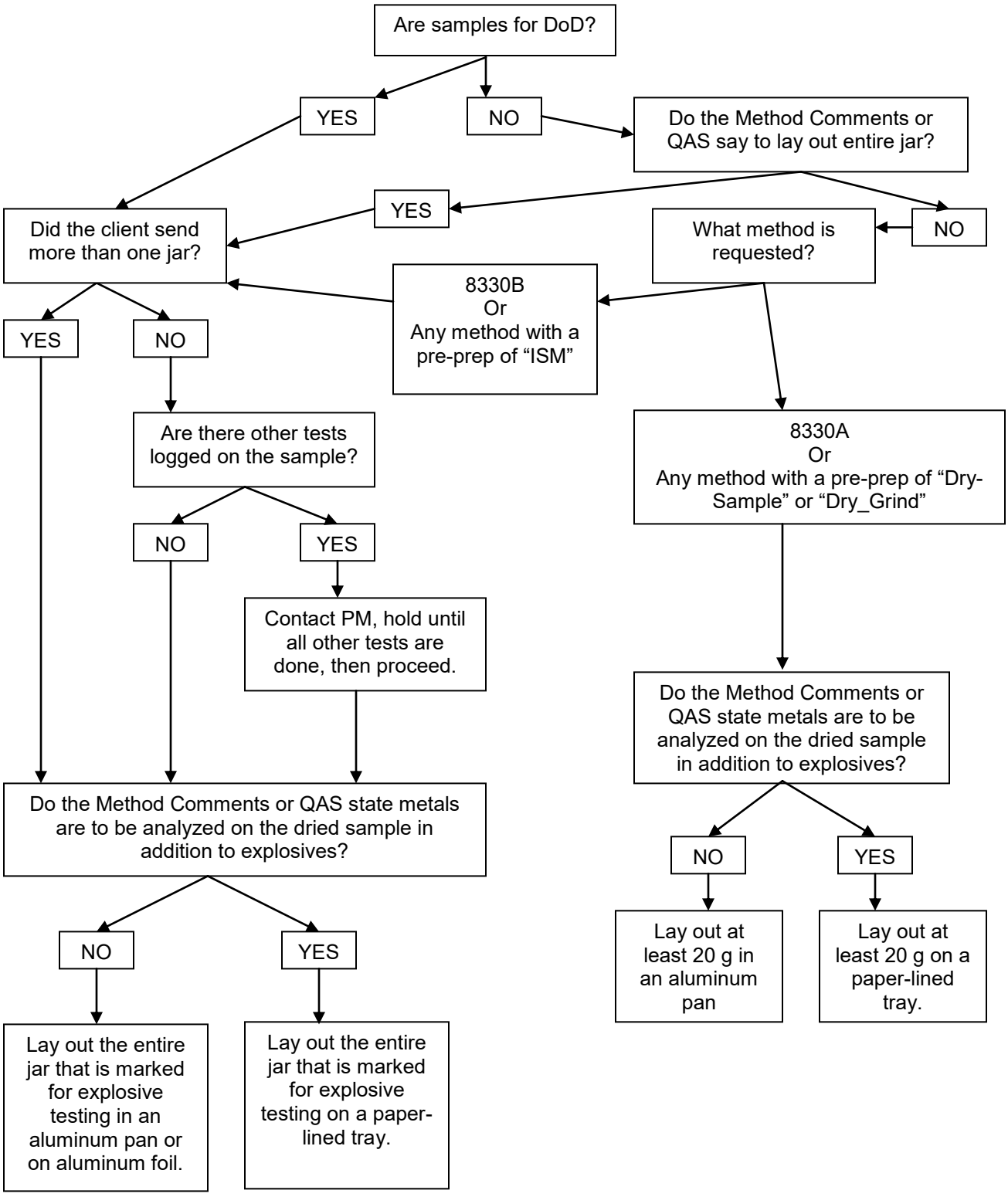
Compound	CAS #	Symbol
Octahydro-1,3,5,7-tetranitro-1,3,5,7,-tetrazocine	2691-41-0	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	RDX
1,3,5-Trinitrobenzene	99-35-4	1,3,5-TNB
1,3-Dinitrobenzene	99-65-0	1,3-DNB
Methyl-2,4,6-trinitrophenyl nitramine	479-45-8	Tetryl
Nitrobenzene	98-95-3	NB
2,4,6-Trinitrobenzene	118-96-7	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	19406-51-0	4-Am-DNT
2-Amino-4,6-dinitrotoluene	35572-78-2	2-Am-DNT
2,6-Dinitrotoluene	606-20-2	2,6-DNT
2,4-Dinitrotoluene	121-14-2	2,4-DNT
2-Nitrotoluene	88-72-2	2-NT
4-Nitrotoluene	99-99-0	4-NT
3-Nitrotoluene	99-08-1	3-NT
Nitroglycerin	55-63-0	NG
PETN	78-11-5	PETN
2,4-Diamino-6-nitrotoluene**	6629-29-4	--
2,6-Diamino-4-nitrotoluene**	59229-75-3	--
Picric Acid	88-89-1	PA
1-Nitroso-3,5-dinitro-hexahydro-1,3,5-triazine**	5755-27-1	MNX
3,5-Dinitroaniline**	618-87-1	3,5-DNA
1,2-Dinitrobenzene (8330 surrogate)	528-29-0	1,2-DNB
Nitrobenzene-d5 (8321 surrogate)	--	NB-d5

** Compounds are only analyzed and spiked upon request.

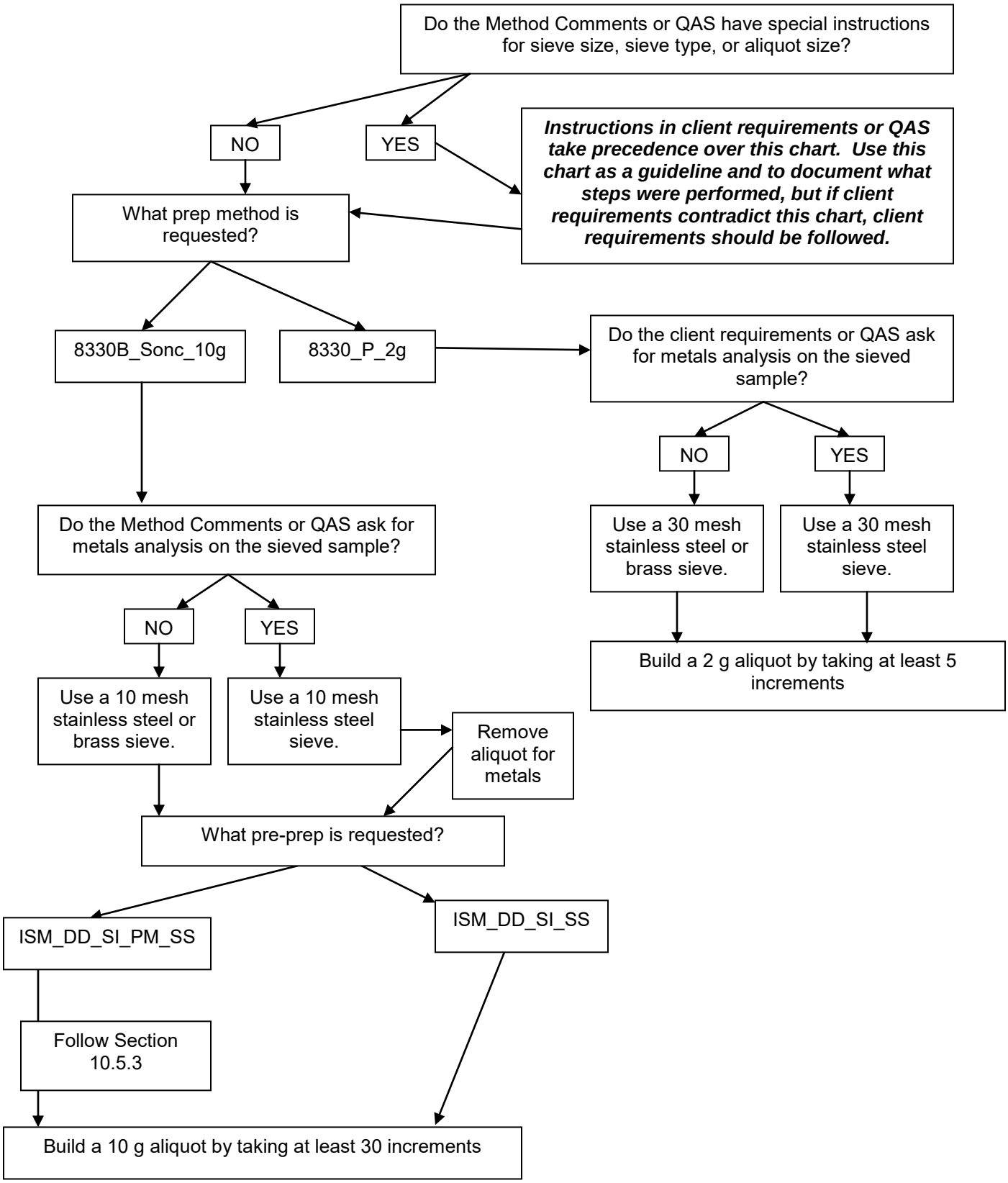
Table 2. Grinding LCS Bulk Material

Compound	Concentration (µg/Kg)
4-Amino-2,6-dinitrotoluene	600
2-Amino-4,6-dinitrotoluene	600
1,3-Dinitrobenzene	600
2,4-Dinitrotoluene	600
2,6-Dinitrotoluene	600
HMX (Octahydro-1,3,5,7-TNTC)	600
1,3,5-Trinitrobenzene	600
Nitrobenzene	600
2-Nitrotoluene	600
3-Nitrotoluene	600
4-Nitrotoluene	600
RDX (Hexahydro-1,3,5-TNTriaz)	600
Tetryl (Methyl-2,4,6-TNPN)	600
Nitroglycerin (Trinitroglycerin)	600
Pentaerythritol tetranitrate (PETN)	600
2,4,6-Trinitrotoluene	600

Appendix 1 – Flowchart and Worksheet for Drying Explosive Soils



Appendix 2 – Flowchart and Worksheet for Grinding and Sieving Explosive Soils



Appendix 3

How to Batch:

ISM_DD_SI_PM_SS (Dry, Disaggregate, Sieve, Ring & Puck, Subsample)
ISM_DD_SI_SS (Dry, Disaggreage, Sieve, Subsample)
Dry_Sample (Dry, Sieve, 2g prep)
Dry_Grind (Dry, Sieve, 2g Prep)

Overview
These five pre-prep methods can be logged in for not just for samples for explosives by 8330A or 8330B and 8321A or 8321B, but also for samples for metals analysis, or perchlorate, or any other method where the client is asking the lab to dry, sieve, and possibly grind the sample before extraction or digestion.

If one sample is logged in for 8330B and 6010B and 6020B and 7471A and all of these methods have the pre-prep of ISM_DD_SI_SS, the sample will show up on the backlog 4 times, (once for each analytical method). This would happen if the client wants us to dry, sieve, and perform ISM for each of these methods.

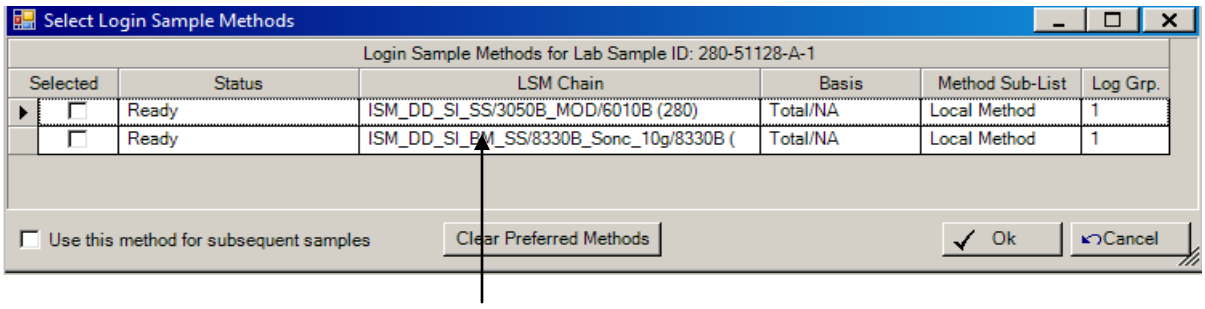
If one sample is logged in for 8330B with a pre-prep of ISM_DD_SI_PM_SS and the same sample is logged in for method 6010B with a pre-prep of ISM_DD_SI_SS, that means that the client wants us to dry, sieve the sample, perform ISM for method 6010B, then ring and puck and perform ISM for 8330B.

We will use a different status to indicate where the samples are.

- A status of “Batched” means the samples have been laid out to dry.
- A status of “Scheduled” on ISM_DD_SI_PM_SS means the samples have been laid out to dry, but possibly need ISM performed before grinding.
- A status of “Partial” means the samples have been sieved.
- A status of “2nd Level Review” on methods ISM_DD_SI_SS, Dry_Sample, or Dry_Grind means that the aliquots have been taken and someone has checked your work.
- A status of “2nd Level Review” on ISM_DD_SI_PM_SS means samples have completed the grinding.

Steps for Samples logged for <u>both</u> ISM_DD_SI_SS <u>and</u> Ring & Puck

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Batch the samples under the ISM_DD_SI_SS method.
   NOTE: Do not put samples in the same batch that require different sieve sizes.
3. Scan your samples into the batch. A window will appear called “Select Login Sample Methods”.



4. Select only the methods that have ISM_DD_SI_SS as the pre-prep.
    a. You can only batch ISM samples under a ISM_DD_SI_SS batch.
    b. You can only batch ring and puck samples under a ISM_DD_SI_PM_SS batch.
    c. You can batch “Dry_Grind” and “Dry Sample” samples under the same batch.
5. Save the batch and print the benchsheet.
6. Print labels. Print one label for each method logged on each sample. Write on the label the analytical method.
7. Lay out the samples to dry. Place all of the labels on the sample tray for the sample. Also label the tray “Grinding Needed” if the sample is logged for Ring and Puck.
8. Set the Ring and Puck Methods to “Scheduled” in the backlog. Do not batch them at this time.
9. Sieve the samples and take the required ISM aliquots. Whenever possible, aliquot the samples directly in the digestion cup for metals, or microwave tube or beaker for organics and record the aliquot masses in the ISM worksheet in Appendix 4. Place the ISM aliquots on the tray with the sample so a 2nd analyst can perform a label check. Document these steps on the TALS batch sheet
10. The Notes field in the Worksheets tab can be used to document if there was rocks or vegetation that did not go thru the sieve. Write NCMs for any samples that contained rocks or vegetation that was removed from the sample.
11. In the Worksheet tab, you can record the weight of the sample before and after drying and the weight of the sample that went through the sieve and the weight of the sample that did not go through the sieve. These measurements are not normally required, so they only need to be performed if client requested.
12. Have a 2nd analyst review the ISM_DD_SI_SS batch to ensure all required ISM aliquots have been performed. Take the ISM_DD_SI_SS batch to 2nd level review.
13. Now the samples are ready to be ground by Ring and Puck.
14. As the samples are ground, add them to the ISM_DD_SI_PM_SS batch.
15. As you add samples to the grinding batch, watch the LSM window to ensure that all ISM_DD_SI_SS methods are at 2nd level review. If there are ISM_DD_SI_SS methods that are not at 2nd level review, perform ISM on the sample for the requested methods before grinding the sample.
16. Take the samples to 2nd level review.
17. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Steps for Samples logged for Ring & Puck ONLY. No ISM_DD_SI_SS methods logged.

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Pull the samples from the walk-in cooler and take custody of the samples. Take note of what shelf the sample came from.
3. Batch the samples and print out labels. Then remove the samples from the batch to place them back on the backlog.
4. Lay the samples out on parchment or foil. Label each tray with the sample ID and the grinding method (Ring & Puck)
5. Document the date and time the samples were laid out to dry. Document if the samples were laid out on parchment or foil. Document that a label check was performed.
6. Set the samples to Scheduled in the backlog to show that they are laid out to dry.

7. Once the samples are dry enough to sieve, sieve the samples and document what sieve size on the Explosive Review Checklist.
8. Open the batch with a grinding LCS. As the samples are ground, add them back to the original ISM_DD_SI_PM_SS batch. There can only be 20 field samples in each batch,. Ring and puck batches can be open for up to 3 days.
9. As you add samples to the grinding batch, watch the LSM window to ensure that the samples do NOT require any non-ground aliquots.
10. Take the samples to 2nd level review.
11. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Steps for Samples logged for only ISM_DD_SI_SS, Dry_Grind, or Dry_Sample. No grinding methods logged.

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Batch the samples under the pre-prep method logged. Do not put samples in the same batch that are logged for different pre-prep methods. Do not put samples in the same batch that require different sieve sizes.
3. Scan your samples into the batch. If your samples are logged in for more than one of these three methods, a window will appear called “Select Login Sample Methods”.
4. Select only the methods that have ISM_DD_SI_SS as the pre-prep.
    a. You can only batch ISM samples under a ISM_DD_SI_SS batch.
    b. You can batch “Dry_Grind” and “Dry Sample” samples under the same batch.

    If the LSM window shows methods with pre-preps of ISM_DD_SI_PM_SS or ISM_DD_SI_BM_SS, then stop and follow the instructions above under the header “Steps for Samples logged for both ISM_DD_SI_PM_SS and Ring & Puck.
5. Save the batch and print the benchsheet.
6. Print labels. Print one label for each method logged on each sample. Write on the label the analytical method.
7. Lay out the samples to dry. Place all of the labels on the sample tray for the sample.
8. Sieve the samples and take the required aliquots. Whenever possible, aliquot the samples directly in the digestion cup for metals, or microwave tube or beaker for organics and record the aliquot masses in the ISM worksheet in Appendix 4. Place the aliquots on the tray with the sample so a 2nd analyst can perform a label check.
9. The Notes field in the Worksheets tab can be used to document if there was rocks or vegetation that did not go thru the sieve. Write NCMs for any samples that contained rocks or vegetation that was removed from the sample.
10. In the Worksheet tab, you can record the weight of the sample before and after drying and the weight of the sample that went through the sieve and the weight of the sample that did not go through the sieve. These measurements are not normally required, so they only need to be performed if client requested.
11. Have a 2nd analyst review the batch to ensure all required aliquots have been performed. Take the batch to 2nd level review.
12. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Appendix 4

ISM Worksheet

G:/QA/Edit/FORMS/Organic Prep Forms/MASTER ISM Spreadsheet_Rev1

ISM BATCH:

Use this spreadsheet to document alquot weights when aliquotting into digestion or extraction vessels. If aliquoting into a temporary vessel, no need to document the exact weight because the sample aliquot will be transferred and weighed at the time of analysis.

Login	**Sample**	Method --->											
			(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											

Appendix 5

ISM Constant Weight Worksheet

Located: \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Organic Prep Forms

DV-F-0070-10-03-2017-Rev 0

ISM Batch Number:

Use this spreadsheet to document drying samples to a constant weight.

			Analyst Initial Date/time 1	Analyst Initial Date/time 2	Dry?		Analyst Initial Date/time 3	Dry?	
Login	Sample	Tare weight (weight of weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 1, 2	Proceed with ISM?	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 2, 3	Proceed with ISM?
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!

10/3/2017 1 of 1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Sample Management and Chain of Custody

Approvals (Signature/Date):

Alyssa Gomez 11.2.18
Alyssa Gomez Date
Sample Receiving Supervisor

Doug Gomer 11/2/18
Doug Gomer Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 11/2/18
Roxanne Sullivan Date
Quality Assurance Manager

Richard Clinkscales 11/2/18
Richard Clinkscales Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 PURPOSE

This procedure describes the management of samples throughout the laboratory, from receipt of samples through archiving of old samples. Included are verification of sample identity, chain-of-custody documentation, verification of sample condition and preservation, notification to the lab of short-holding-time and rush samples, resolution of sample receipt discrepancies, requirements for sample delivery acceptance, and sample archiving.

2.0 SCOPE

2.1 TestAmerica Denver has one sample receiving area. Both radiological and non-radiological samples are received in this area. Personnel working in the Sample Control area are required to complete radiation safety training, as described in the Radiation Safety Manual (RSM), before handling radioactive samples. See SOP DV-RS-0001, *Purchase, Receipt, Handling and Identification of Radioactive Material*, for the procedures specific to incoming radioactive samples.

2.2 This procedure describes the Internal Chain-of-Custody requirements from receipt of samples via a Chain of Custody form until samples are removed from the coolers and archived by Waste Management. After samples are archived, custody of the samples is transferred to Waste Management as described in SOP DV-HS-0005, *Excess Sample Material Management*. For cradle to grave tracking of any sample, both this procedure and SOP DV-HS-0005 must be used.

2.3 Any soils received with a USDA soil import permit must be handled according to the requirements in SOP DV-QA-0019, *Quarantine Soil Procedures*.

3.0 SAFETY

3.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual and the Radiation Safety Manual.

3.2 Safety concerns specific to this procedure:

3.2.1 Potentially Hazardous and Biologically Active Samples

3.2.1.1 Do not open any shipping containers marked “Biohazard”, “Blood”, “Tissue”, “High Hazard”, “Extremely Hazardous”, or other phrases that indicate that special safety problems may exist. Set them aside, and contact the Environmental Health & Safety Coordinator (EHSC) to determine how to proceed.

3.2.1.2 Also contact the EHSC if blood, tissue, or other biological fluids are observed.

3.2.1.3 Samples with visible amounts of non-aqueous liquid (e.g., solvents, petroleum products, etc.) must be stored separately from other samples to avoid cross-contamination, and the EHSC should be called in to evaluate the samples and the need

for additional safety precautions.

3.2.1.4 If the COC or other paperwork accompanying samples from DOE sites indicates that the samples may contain beryllium, beryllium oxide, and/or asbestos, do not open the sample container itself. These samples must be handled under appropriate ventilation to ensure contamination control and minimize possible worker exposure.

3.2.2 Broken or Leaking Samples

3.2.2.1 If a shipping container is opened and found to contain a broken sample container, the material from the broken container is presumed to be potentially hazardous.

3.2.2.2 Ensure that proper PPE is worn. Blue Grip gloves (cut resistant) or equivalent should be worn if handling broken glass and cleaning out coolers.

3.2.2.3 The entire shipping container must be kept in a hood if it is emitting fumes or dust or may contain high levels of Beryllium, Beryllium Oxide or asbestos. Contact the EHSC. The container shall remain in the hood until the reaction is under control as determined by the EHSC or until the high hazard material is contained.

3.2.2.4 The Waste Coordinator (or designee) is contacted so that proper management of the broken hazardous sample material can be disposed of as described in Section 8.0 of this SOP. Communication with the waste management group should include the number and type of containers that require disposal.

3.2.2.5 If other intact sample containers in the same shipping container are visibly dirty or have come into direct contact with spilled sample material, then the unbroken sample containers will need to be decontaminated, as discussed in Section 8.0.

3.3 Primary Materials Used

There are no reagents, standards, or other chemical materials used in this procedure. Therefore there are no materials that have a serious or significant hazard rating.

4.0 DEFINITIONS

4.1 Chain-of-Custody Form: A critical legal record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses.

4.2 **Class 7 Radioactive Material:** Department of Transportation (DOT) terminology used to describe radioactive material having a specific activity and quantity. This term should not be confused with the definition of a radioactive material. It is used strictly for circumstances involving the shipment of radioactive items and does not constitute a limit below which a material is not considered radioactive.

4.3 **Sample Delivery Acceptance:** The point in time at which TestAmerica is first obligated to initiate preparation and/or analysis of samples.

4.4 **Internal Chain-of-Custody Report (ICOC):** The report used to document the storage, handling and archival of client samples while in possession of the TestAmerica Denver laboratory. The Laboratory Information Management System (LIMS) has the capability to track sample custody, transfers, and disposal.

5.0 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (TAL Denver Quality Assurance Manual) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include, but are not limited to:

- A COC filled out completely;
- Samples must be properly labeled;
- Proper sample containers with adequate volume for the analysis and necessary QC;
- Samples must be preserved according to the requirements of the requested analytical method;
- Sample holding times must be adhered to;
- All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time;
- The project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

The PM needs to be notified if the client needs to drop off samples during non-routine working hours so they can schedule a time with the Sample Receiving Department so a DOT trained person is here to receive the samples.

NOTE: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

.

6.0 PROCEDURE

6.1 Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described (see SOP DV-QA-0031).

6.2 Sample shipments arrive at the central shipping and receiving area. The delivery

person rings the exterior bell to have one of the Sample Receiving staff unlock the door.

6.3 Sample Receiving personnel electronically sign the shipping receipt to indicate receipt of the coolers for FedEx and UPS shipments. For coolers dropped off by clients or private courier, the Sample Receiving personnel sign the CoC.

6.4 All coolers are scanned on the outside for radioactivity. See SOP DV-RS-0001 for procedure.

6.5 If the samples being received are marked as being radioactive or the samples are from a company on the list of clients who handle known radioactive samples (see WI-DV-044), follow the procedures in SOP DV-RS-0001.

6.6 The Sample Receiving Checklist (Attachment 1a) is used to document the receiving and login process. All samples received from sites in Alaska use the Alaska Sample Receiving Checklist (Attachment 1b). Some clients have a client-specific checklist. The PM is responsible for identifying these client specific forms

6.7 The CUR (Condition Upon Receipt) form (Attachment 3) will be use to document any issues with the condition of the cooler upon receipt. The checklist/CUR forms will remain with the coolers throughout the sample receiving process.

6.8 For the protection of the people working in the area, it is important to first open the coolers under the hood. Examine the contents and open any bags containing samples under the hood to ensure no unwarranted exposures occur.

6.9 Once it has been determined that there are no broken samples and no fumes coming from the cooler, the temperature inside the cooler is determined and recorded on the Sample Receiving Checklist. Some clients have a client-specific checklist. (See Section 6.9.5 on how to address broken containers in a cooler.)

6.9.1 The infrared (IR) temperature gun is calibrated daily (see calibration procedures in SOP DV-QA-0001). Record in Sample Receiving Temperature Logbook.

6.9.2 Take a reading from a temperature blank, if present. The IR reading should be taken 1-3” from the sample and the observed temperature is recorded on the Sample Receiving Checklist.

NOTE: West Virginia requires that the temperature is to be checked on <u>each sample bottle</u>. Any deviations shall be noted on the CUR (by sample ID unless all samples exceed temperature limits).

6.9.2.1 Record the ID of the temperature gun used on the checklist.

6.9.2.2 The correction factor determined during the calibration of the IR gun is recorded on the checklist.

6.9.2.3 The correction factor is applied to the observed temperature and the actual temperature is recorded when the data are entered

into TALS (e.g., if the correction factor is -0.2 ºC, and the reading is 2.6ºC, the recorded temperature is 2.4ºC).

6.9.3 If multiple coolers are received for one project record as many temperatures as feasible on the first checklist. Use another checklist if more cooler temperatures need to be documented. Only one checklist is needed for the rest of the questions. Place a diagonal line through the bottom portion the checklists that are only needed for temperature documentation. If the temperature in the cooler is greater than 6ºC or less than 0 ºC, record this observation as a deviation on a CUR form (Attachment 3). If the temperature is below zero, check to determine if any samples are frozen and note on the CUR. Place a green temperature sticker on the cooler which means to notify the PM/PMA to contact the client on how to proceed. After all coolers are triaged PMs for the coolers that are out of temperature (marked with green sticker) are notified by phone for instructions on how to proceed.

6.9.4 If a chain of custody has multiple coolers and only some of them are outside temperature it may be necessary for the Login staff to un-pack the cooler to identify the samples. A detailed inventory of the samples and containers in the out-of-temperature cooler must be made in the comments section of the CUR. If this is not necessary for the client at the time of notification it will be done as part of the normal cooler un-packaging.

NOTE: In addition to notification of the client of temperature excursions, for any samples submitted for drinking water (potable water) analyses the laboratory must recommend resampling to the client. If resampling is not feasible and the client requests the analysis proceed, this must be noted in the case narrative.

6.9.5 If there is a broken bottle also note this on the CUR form. If the cooler contains broken sample containers, refer to SOP DV-HS-0005. Record the identification of all broken sample containers received on the CUR and contact the PM. Record observation on Sample Receiving Checklist. Include a copy of the list of broken samples when transferring the cooler to the Waste Management Group.

6.10 Record the following initial checks on the Sample Receiving Checklist:

6.10.1 Company name and sampling site.

6.10.2 Condition of cooler custody seals, if present.

6.10.3 Presence or absence of custody form(s).

The chain of custody has necessary dates and signatures from earlier transfers. Some chain of custody forms may have a brightly colored sticker which identifies they contain short hold parameters.

NOTE: The Alaska and client-specific Sample Receiving Checklists have additional items that are documented.

6.11 Identify any short holding time parameters. Place a Priority Form (Attachment 4) on the cooler along with a Priority I, II or III sign. Priorities are assigned to samples according to the following:

6.11.1 Priority I – Hexavalent Chromium, Volatile Soils, BOD, CBOD, Hydrazine (waters and solids), VOA soil samples preserved with DI water that must be frozen within 48 hours of sampling, one-day Turnaround Time (TAT)

6.11.2 Priority II – All other 48 hour hold time tests including Cyanide preservation, color, nitrite by Spec, orthophosphate by spec, nitrate by IC, nitrite by IC, Orthophosphate by IC, Settleable Solids, Turbidity, samples for 2-3 day TAT.

6.11.3 Priority III – Any rush analyses >3 day TAT, projects received but not logged on previous day, any sub-in work from other TestAmerica Labs, Dissolved Oxygen, Free Carbon Dioxide, Sulfite, pH, or Ferrous Iron.

6.12 Rapidly expiring holding times are assigned priority based on the amount of holding time remaining.

6.13 The PM (or designee) will come to Sample Receiving and perform the following:

6.13.1 Identify the project(s) not specified on the COC and supply the sample receiving staff with the appropriate TALS project number.

6.13.2 Provide analytical and report due date, if different from what is on the COC.

6.13.3 Provide any information on sub-outs and rushes as necessary.

6.13.4 Look for any expiring Holding Times that sample receiving staff may have missed.

6.13.5 Look for any hand written instructions on preprinted COCs or any extra information provided by the client not on the COC that needs to be clarified before the job is logged.

6.14 Coolers with Unidentified Projects

For coolers where local PMs cannot identify the projects or insufficient information is provided on the COC to login the samples, follow the guidance in WI-DV-0105, *Processes for Missing Info on COCs.* This provides a roadmap by which the samples can be logged in correctly. Typically this occurs for coolers that are sent directly to the lab by the client but are logged and managed by sister labs.

6.15 Before the cooler(s) are unpacked, verify that the cooler temperature(s) have been documented on the Sample Receiving Checklist(s). If there are no temperatures recorded or the number of coolers does not match the number of temperatures recorded, measure and record the temperature of all coolers associated with that COC.

6.16 Unpacking the Cooler

6.16.1 Unpack the cooler, and line up the bottles in the numerical order given on the COC. Check for indications that the cooler contains Radioactive Materials:

6.16.1.1 Blue CAT I stickers

6.16.1.2 Radioactive Package Monitoring and Receipt Form

6.16.1.3 If neither are present refer to WI-DV-0044, *TA Denver Known Rad Clients List* posted on the fume hood.

6.16.2 Check inside of lid for a Revenue Center sticker

6.16.2.1 This is a small, square white sticker with a barcode, city name, and code on it

6.16.2.2 Adhere this sticker to the checklist somewhere to the right of the questions without obscuring any information on the checklist

6.16.3 Air bills can be adhered to the back of the checklist, if there are more than can fit on the checklist an additional piece of blank paper can be used

6.16.4 Remove containers from cooler and dispose of bubble wrap/bags

6.16.5 Place containers on bench in the following order:

6.16.5.1 From left to right by sample ID in the order of the COC;

6.16.5.2 From back to front, tallest container (e.g., 1 L Amber Glass) to shortest (e.g., 125 mL plastic);

6.16.5.3 From back to front, glass bottles to plastic bottles;

6.16.5.4 From back to front of the same container type preserved to unpreserved (e.g., 500 mL Plastic HNO_3 behind 500 mL Plastic Unpreserved).

6.16.5.5 From back to front unfiltered to filtered (e.g., Total Metals behind Dissolved Metals);

6.16.5.6 Place VOA vials in green VOA racks by sample ID in the order of the COC Invert VOA vials and gently tap to make any headspace/bubbles come to the top. Measure the headspace//bubbles with a clear ruler and document any headspace/bubbles that is greater than 6mm on the VOA Headspace form(DV-F-0077) place a red sticker on the bottom of the VOA vial with the headspace/bubbles.

NOTE: Be sure to separate by VOA analysis type and in the same order for each sample (e.g., 8015 vials then 8260 vials for sample 1, 8015 then 8260 for sample 2, etc.) Any VOA samples must be iced while on the

counter during the login procedure. Immediately after unpacking the cooler, place VOA vials in a VOA rack and place a bag of ice or frozen gel pack on top of the containers to ensure temperature preservation

6.16.6 Once all containers are unpacked place them in cardboard boxes and cover in gel ice packs

6.16.6.1 Attempt to maintain previous layout as much as is reasonable. After one-half hour the samples must be moved to a cooler with icepacks or placed in the walk-in to maintain temperature preservation. It is recommended that a timer be used for each set of samples to monitor the time.

6.16.6.2 Aqueous samples for metals analysis do not need to be iced.

6.17 Fill out the Sample Receiving Checklist, recording the following information on the Sample Receiving Checklist:

6.17.1 Non-radioactive materials will have N/A for question 1.

6.17.2 The number of containers indicated on custody form matches the number received.

6.17.3 Client sample identifiers on the container labels **exactly** match identifiers on the custody form.

6.17.4 Samples are received within holding time.

6.17.5 Container labels are present, intact and legible.

6.17.6 Sample collection date and time are provided on both the COC and container labels.

6.17.7 Sample volumes, types of containers and preservatives meet the requirements of the Guidelines for Sample Bottles and Preservatives, (Attachment 5).

6.17.8 Client supplied extra volume for QC on at least one sample (Check *NO* if requested and not supplied; *NA* if not requested)

6.17.9 Determine if samples require splitting or compositing. If required, complete a Priority Form (Attachment 4).

6.17.10 Presence of multiple phases in any of the samples (e.g., solids and liquid or multiple liquid phases).

6.17.11 If there is no field for the field sampler's name on the COC question 5 is N/A

6.17.12 If there are no VOA Vials 12&13 are N/A

6.17.13 Stop at Login Checks Section

6.17.14 Initial on line at top right of questions field

6.18 Corrective Action for Sample Receipt Discrepancies

Any discrepancies must be noted on a Condition Upon Receipt Anomaly Report (Attachment 3). The CUR is placed in the project folder and reviewed by the PMA and or PM during login review. Clients are notified as necessary.

6.19 Sample Login

6.19.1 The Project Manager must be contacted before logging in any unusual matrices for correct log in instructions.

6.19.2 The COC "received by" field must be signed with TA-DEN and the signature of receiving analyst.

6.19.3 Samples are logged into the TestAmerica LIMS System (TALS). Refer to the User Guide in Attachment 7 or specific TALS instructions.

6.19.4 The Login application in TALS is divided into three different modules, Receipt, Login, and Job. Each module performs a distinct function in the login process.

6.19.4.1 The Receipt module allows the lab to receive samples, print container labels, and document the condition of the samples and cooler upon receipt. The act of receiving samples in TALS starts the login process.

6.19.4.2 In the Login module, sample information (client sample identifiers, sample matrix, sampling date/time) are entered for each of the received samples. A project containing the client requirements (created by the project manager prior to sample receipt) will be used to login samples. The project contains the method chains, project limits and reporting requirements for the samples. In the login module, methods are selected from the project in accordance with the chain of custody for each sample. By using the project as the client's template for login, the sample receiving group can log jobs more efficiently. It is important to note that even though a job is logged in and samples may be distributed to the lab for work, the data will not calculate until the login has been reviewed and approved by the project manager or designee.

6.19.4.3 The last module in the login application is the Job module. Each job is equivalent to a login when the login is first created. A login can then be split into several jobs (each job with a separate set of reporting requirements) or several logins can be joined to create one job (a sample delivery group). Each job contains a separate set of deliverables, client contacts, pricing, and turnaround times. The project manager will review the login as a whole and the pricing and deliverables for each job separately.

6.19.5 The User Guide (Attachment 7) describes how to receive a sample delivery, login a sample delivery, and work with jobs in TALS.

6.19.6 All containers will be designated a storage location based on test as listed below:

Method	Storage Location
624, 8260	MS Storage
6010, 6020, other nonrefrigerated samples	Metals Storage
8021, 8015_GRO, 602, 8011, EDB, DBCP, RSK-175, AK 101	GC-Storage
All other work for Denver	Main Walk-in – shelf number assigned each day
Subouts – non-volatile samples	Sub shelves in the Main Walk-in
Subouts – Volatile samples	Login VOA refrigerator

6.19.7 Generate labels according to the COC.

6.19.8 Sign the COC with the date and time that the cooler was received. Place the COC label (generated in Section 6.19.6) on the COC and scan into TALS.

6.20 Labeling

6.20.1 Remove containers from box covered in ice packs and lay-out per sample ID.

6.20.2 Place labels onto lids of container based on container type and sample ID. Avoid sticking onto other stickers such as preservation labels.

6.20.3 Labels for VOA Soils are **NOT** affixed directly onto the sample bottles but are attached with a rubber band.

6.20.4 Check TALS label for match to container label

- Sample ID
- Sample Time/Date
- Container Type

6.20.5 Once information is confirmed as matching place label on container side. Orient vertically so barcode on TALS label lays flat.

6.20.6 On containers too short for entire sticker to fit, e.g., 2 oz soil jars, still place sticker vertically.

6.20.7 DuPont PFOA stickers also go directly onto each PFOA container.

6.20.8 Samples are boxed by type (i.e., test group/storage location).

6.20.9 Separate by storage location. MS Storage in one box, Walk-in Shelf # in another box, Sub in another, etc. Ensure that all VOA water samples (including trip blanks, field blanks, etc.) are put in separate boxes from

VOA soil samples.

6.20.10 Label the front of all boxes with login number on colored sticker

6.20.11 Label front of all boxes with any potential sample archiving duration sticker indicated on checklist.

6.20.12 Label front of all boxes with any necessary radioactive materials sticker and radioactive materials stickers also go on every container for samples from known rad sites or samples determined to be radioactive.

6.21 Additional Preservation Checks

6.21.1 Place ice back on containers in the labeled boxes

6.21.2 The pH of any non-volatiles samples must be checked at time of receipt for DoD, DOE, and West Virginia work **ONLY, (**unless specified in the project.). For all other work mark NA for Item 19 on the Sample Receiving Checklist. (See Attachment 1a.)

6.21.2.1 Refer to "Guidelines for Sample Bottles and Preservatives" (Attachment 5) to determine what the pH should be.

6.21.2.2 Do not perform pH check on samples marked for volatile organics, oil and grease (Method 1664A or HEM) or TOC tests. These will be done later by VOA or Wet Chem analysts.

6.21.2.3 If the COC or other paperwork accompanying samples from DOE sites indicates that the samples may contain beryllium, beryllium oxide, and/or asbestos, do not open the sample container itself. These samples must be handled under appropriate ventilation to ensure contamination control and minimize possible worker exposure. If these are liquid samples that require pH checks, these checks must be performed in a hood. Note the sample handling requirement on the CUR so that the PM may add a method comment for the lab to handle samples in a fume hood.

6.21.2.4 For non-volatile tests requiring chemical preservation, open the bottle, remove a few tenths of a milliliter of sample with a disposable transfer pipette and touch the pipette tip to wide-range pH strips. Compare the color that develops immediately to the color chart.

6.21.2.5 Discard the pipette and any unused sample.

6.21.2.6 Mark the form indicating that the pH check was performed on all samples.

6.21.2.7 List the sample number and pH for any samples not meeting requirements on the CUR and contact the PM.

NOTE: Any analytical data resulting from improperly preserved samples must be accompanied by a statement in the narrative indicating the condition of the sample upon receipt and provide sufficient information to end data users regarding regulatory non-compliance.

).

6.21.3 Verify that the preservation is marked on VOA vials and confirm the test for which it was logged to ensure proper holding time is assigned.

6.21.4 Check whether or not non-volatiles samples require preservation with sodium thiosulfate as indicated in the project notes in TALS. If sodium thiosulfate is required, determine if the sample contains residual chlorine.

6.21.4.1 Samples that must be checked for residual chlorine include:

a) Drinking waters,

b) Process water samples suspected or known to contain chlorine,

c) Water samples from North Carolina, unless knowledge from the site indicates it is not required.

6.21.4.2 Samples should be checked for the presence of residual chlorine with starch iodide paper. Samples containing residual chlorine will develop a dark blue stain, caused by iodide being oxidized to iodine and reacting with the starch.

6.21.4.3 If samples are positive for residual chlorine, the PM must notify the client for instructions on how to proceed with the samples. If the client wishes to have the samples treated with sodium thiosulfate, the samples will be sent to the Wet Chemistry Department to perform the neutralization.

6.21.5 Complete the Login Checks, Archive Requirements and Labeling and Storage Checks sections of the Sample Receiving Checklist. See Section 6.22 for Sample Storage.

6.21.6 If the project includes any tests with short holding times fill out a Priority Form (Attachment 4), copies are made for each lab area receiving short holds. A copy is made for each short hold test in Wet Chem. Short hold samples are delivered to lab areas frequently throughout the day or the lab area sends a representative to pick up the samples in the Sample Receiving area. **A signature from the area accepting the samples is required before samples are transferred to the work area.**

NOTE: If no one is in the work area to receive the samples, the samples are stored as indicated in Section 6.22 until the samples can be signed for by an individual in the work area.

6.21.7 For jobs that have short holds or rapidly expiring samples Login staff must login release and login review these jobs.

6.21.8 A folder is created for the sample lot. A complete folder contains:

- A file folder labeled with the lot number
- Chain-of-custody forms
- Freight bills
- The completed Sample Checklist
- A CUR report, if used
- Priority Forms, if used
- Any paperwork the client supplied with the samples

6.21.9 Deliver the project folder to the PM's bin box in Sample Receiving for review and approval. The PM (or designee) collects the folders for review the login for the items outlined in WI-DV-036, *PM Login Review Items*.

6.21.10 The PM or designee works with the client, as needed, to resolve any items noted on the CUR or Sample Receiving checklist and communicates the resolution to the responsible parties.

6.21.11 Enter any data from RADACC forms into the RADDAC database. See SOP DV-RS-0001, *Purchase, Receipt Handling, and Identification of Radioactive Material.* Once the data are entered the form is placed in the RADACC form bin. These are delivered to the RSO weekly.

6.22 Sample Storage

6.22.1 Samples are stored in the walk-in cooler, except:

- Samples for volatile organics analysis (VOA) are taken directly to the VOA area refrigerators or temporarily placed on a cart in the Main Walk-in until delivery to the VOA areas.
- Water samples for metals analysis are picked up by an analyst from the Metals group.
- Known high level/high hazard samples are stored in Refrigerator ASG8.
- Samples for short-hold tests are temporarily placed on a cart in the Main walk-in until delivered to the lab. The lab analyzes these samples immediately and returns them to the storage location designated at log-in when done with the samples.

6.22.2 VOA soil samples preserved with DI water are stored in the freezer in the Sample Receiving area until transport to the VOA area. Verify by visual inspection at the base of the container that all samples to be put in the freezer are soil samples and that no water samples are present.

NOTE: Do not invert sample containers. Inspect vials without inverting.

6.22.2.1 Any water samples found must be reboxed and refrigerated.

6.22.2.2 After samples are placed in the freezer in either Sample Receiving or the VOA areas, complete the VOA Sample Freezer Verification Form (Attachment 6) found in the pocket on the freezer door. Signature or initials on this form indicate that samples have been visually inspected and only soil samples are placed in the freezer.

6.22.3 The shelf number where samples are stored in the walk-in cooler or the remote storage location is indicated on the sample labels. Verify that samples are put on the correct shelf.

6.22.4 Consult SOP DV-QA-0019, *Quarantine Soils Procedure*, for proper storage of samples or coolers labeled with "Quarantine Sample" stickers or other USDA labels. A special set of shelves in the walk-in cooler is marked for storing only quarantine samples.

6.22.5 Consult SOP DV-RS-0001, *Purchase, Receipt, Handling and Identification of Radioactive Material*, for the proper labeling and storage of samples received from radioactive sites or potentially radioactive sites.

6.22.6 Sample transfers are documented in TALS (See SOP DV-QA-0038, *Internal Chain of Custody and TALS*). Samples are kept in the walk-in cooler and the satellite areas until final results have been reported to the client. The samples are then moved to the sample archive area according to SOP DV-HS-0005, *Excess Sample Material Management*, unless special instructions state otherwise.

6.23 The ideal situation is not to roll coolers. Laboratory management will make its best effort to staff the login group with the appropriate number of people required to log in all coolers received daily. The average numbers of coolers an analyst should log each day is 15. If 150 coolers are received in a day then 10 people are needed. If the daily staff is only at 8 then management will try to find 2 more people to help out.

6.23.1 Although every effort is made to log all samples received on day of receipt, volume of receipts may dictate that some coolers are logged after the date of receipt. The COC must be signed on the day of receipt and must be signed with the receipt date and time, not the log-in date and time.

6.23.1.1 Any coolers that have not been logged are reviewed and evaluated at the end of the day to assess if they can be "rolled" i.e., no 48 hour analysis, short turn around times and rapidly expiring hold times.

6.23.1.2 As each cooler is reviewed, sign the COC, if it has not already been signed, with the date and time the cooler was received.

6.23.1.3 After cooler assessment is completed, an email with a list of all clients and number of coolers that were not logged is sent to the

Project Management group.

6.24 Subcontract Work

All sub-out work will be performed according to SOP DV-QA-0036, *Sub-out Work Sample Management and Chain of Custody*. These samples are placed on the Sub shelves in the Main Walk-in and VOA samples are placed in the Login VOA refrigerator until they are packaged for shipment.

6.25 Relogging and Relabeling Samples

6.25.1 If a job must be relogged the sample receiving staff will print a copy of the original chain of custody and ensure that all samples in the job are identified, located, relogged, relabeled and reshelved.

6.25.2 The relogging event must be documented on a CUR such that the original job number is linked to the new job number and this is attached to the new job in TALS.

6.26 Sample Archiving

6.26.1 Samples are archived and maintained per SOP DV-HS-0005.

6.26.2 Aqueous and soil samples and empty bottles will be taken from all storage areas and transferred to the sample archive area. If a job is cancelled, the archivist must contact the Sample Control Supervisor to assess when the samples can be archived. The samples **must** remain in cold storage until it can be confirmed that they are to be archived.

6.26.3 While samples are in archiving, they are under the custody of the Waste Management Group and documentation of the destruction of samples will be recorded per SOP DV-HS-0005.

6.27 Sample Returns

If samples are returned to the client before samples are archived, the Sample Receiving Group shall document the return shipment in TALS. (See SOP DV-QA-0038.)

7.0 RESPONSIBILITIES

7.1 Sample Receiving Personnel

7.1.1 The Sample Receiving personnel are responsible for the login of samples, including signing the Chain-of-Custody forms upon receipt of samples, completing the Sample Receiving Checklist, reading and complying with project login notes, noting short-holding-time parameters as identified on the Chain-of-Custody, identifying and documenting discrepancies on the Condition Upon Receipt Anomaly (CUR) form, promptly notifying Project Managers of all discrepancies, and maintaining the samples in the walk-in cooler.

7.1.2 Sample Receiving personnel are required to take Department of Transportation (DOT) General Awareness, Safety, Security, and Function Specific training in order to sign for hazardous materials deliveries. This training must be completed within 90 days from starting in Sample Receiving, and until the training is completed personnel must work under direct supervision.

7.2 Project Managers

7.2.1 The Project Manager (PM) must build a project in the TALS database before samples can be logged into the system. The project includes information about the types of samples expected, extraction/digestion methods, and analytical methods to be used.

7.2.2 If not already available to Sample Receiving, the PM provides Sample Receiving staff with the project number to use as the basis for logging in samples.

7.2.3 The PM or designee is responsible for the verification of sample login using WI-DV-0036, *PM Login Review Items*. The PM contacts the client with any issues noted by the sample receiving personnel.

8.0 WASTE MANAGEMENT

8.1 All waste will be disposed of in accordance with federal, state and local regulations. Where reasonably feasible, technological changes have been implemented in this procedure to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in Section 13 of the Environmental Health and Safety Manual, *Waste Management and Pollution Prevention*, and Policy DV-HS-001P, *Waste Management Plan*.

8.2 Receipt of samples can generate several kinds of waste.

8.2.1 Non-radioactive and non-hazardous waste

8.2.1.1 The primary waste is non-hazardous, non-radioactive packing material that is considered regular trash and disposed into the dumpster and taken to a landfill.

8.2.1.2 If a sample breaks in transit from the client to the laboratory, the packing material and material used to clean the spill will be considered hazardous if the sample was shipped as DOT hazardous.

8.2.1.3 Samples that are not shipped as DOT hazardous do not require special labeling or special notification to the Waste Management group. They are handled as described in SOP DV-HS-0005, *Excess Sample Material Management*.

8.2.2 Non-radioactive hazardous waste

Samples which are sent with prior knowledge of the hazardous constituents within a sample, need to be properly labeled by the client, i.e., PCB labels, etc. If a container carrying a known hazardous sample breaks in transit, all the material in that container must be assigned the same hazard classification as the broken sample. Sample Control will not disturb the contents of the container. Sample Control will contact Waste Management who will dispose of the broken contents and lab trash and rinse unbroken sample containers, trapping and disposing rinse water, per SOP DV-HS-0005.

8.2.3 Radioactive waste

Samples that are known or designated by the client to be potentially radioactive should be segregated and received following SOP DV-RS-0001, *Purchase, Receipt, Handling, and Identification of Radioactive Material*. If a container carrying known radioactive material (above DOT limits) breaks, all material in the shipping container must be considered radioactive. Contact the RSO (or designee) for instructions and guidance.

9.0 ATTACHMENTS

Attachment 1a: Example Sample Receiving Checklist
Attachment 1b: Example Alaska Sample Receiving Checklist
Attachment 2: Example TestAmerica Chain of Custody
Attachment 3: Example Condition Upon Receipt Anomaly Form
Attachment 4: Example Priority Form
Attachment 5: Example Guidelines for Sample Bottles and Preservatives
Attachment 6: Example VOA Sample Freezer Verification Form
Attachment 7: User Guide for Login of Samples into TALS

10.0 REVISION HISTORY

Revision 28 dated 11/05/2018
- Added headspace/bubbles info in section 6.16.5.6
- Updated copyright information

Revision 27 dated 7/31/2018
- Added comment to section 5.0 about receiving samples during non- working hours.

Revision 26 dated 12/31/2017
- Added comment to section 6.19.2 COC “received by” field must contain TA-DEN.

Revision 25 dated 12/08/2017
- Added comment to section 6.19.1 to check with Project Manager before logging in any unusual matrices.

Revision 24 dated 1/31/2017
- Added requirement for trip blanks for VOA analyses to Sample Acceptance Policy in Section 5.0

- Major revision to the procedure in Section 6.0. Reordered, clarified, added requirements.
- In Section 7.1 clarified that DOT training is required in order to sign for hazardous materials.
- Updated forms in the attachments to reflect procedural changes.

Revision 23 dated 6/30/2016

- Added new section 2.3 to reference SOP DV-QA-0019, Quarantine Soil Procedures
- Added new sections 3.2.1.4 and 6.21.6.3 and revised section 3.2.2.3 to address DOE requirement regarding samples with high levels of Be, BeO and asbestos.
- Added sentence to Section 6.7.1 regarding where to record IR temperature gun calibration
- Added note to section 6.9.5 regarding client notification for drinking water samples received out of temp
- Added VOA soil samples requiring freezing to Priority I list in Section 6.10.1
- Added new section 6.18 to address maintaining temperature preservation at login
- Added West Virginia to list of samples requiring pH verification at login, section 6.21.6
- Added bullet to Section 6.22.4 to address short term storage of short hold tests
- Added freezer storage in sample receiving area for VOA soils to Section 6.22.5
- Updated Attachment 1 – changed label on Y/N checkbox for pH checks from DoD/DOE

Revision 22 dated 12/31/2015

- Minor formatting and grammar corrections throughout
- Added requirement to list sample numbers when moving broken samples to Waste Group to Section 6.15
- Added language to Section 3.2.2.4 to require that communication with the waste management group should include the number and type of containers that require disposal

Revision 21 dated 09/30/2015

- Added note to section 6.7.2 regarding WV sample temperature requirements
- Re-arranged the order of sections 6.7.2.1 – 6.7.2.4 to clarify laboratory practices
- Added section the current section 6.11 regarding PM/PMA duties
- Added sections 6.13.2.1 – 6.13.2.3 regarding cooler assessment
- Section 6.13.4 was re-numbered to section 6.14 – all succeeding section re-numbered accordingly.
- Added section 6.18 regarding the use of gel packs

Revision 20 dated 09/09/2014

- Reordered section 6 to reflect current practice, including recording temperature on pre-printed label while cooler is in hood, opening any plastic bags holding samples while the cooler is in the hood to ensure no inadvertent exposures to personnel due to unnoted broken containers might occur
- Removed “cooler greet” from procedure as this is no longer in the process
- Revised Attachments 1a and 4 for ease of use and to incorporate procedure changes for recording cooler temperatures and other procedural changes made for efficiency.

Revision 19 dated 02/28/2014
- Removed detail regarding sub-out work from Section 6.19 and added reference to DV-QA-0036

Revision 18, dated 10/11/2013
- Added Alaska sample receiving checklist and option for PMs to provide client specific sample receiving checklist (Section 6.7)
- Added use of "Short Hold" stickers used by some clients (Section 6.8)
- Added description of login priorities in section 6.8
- Added requirement for PM or designee to verify assignment of login priorities (Section 6.10.5)
- Added Section 6.10.7 for cooler review at end of day
- Added requirement to verify cooler temperatures recorded on COC before unpacking cooler (Section 6.11)
- Added information regarding login priorities in section 6.12
- Added reference to DV-HS-0005 if coolers broken.
- Added sections 6.17.4-6.17.5 to generate and review bottles when affixing labels.
- Updated Attachments 1, 3, and 5.

Revision 17 dated 1/31/2013
- Added procedure steps for segregation of VOA soil and water steps and verification that only soil samples are frozen, Sections 6.16.2 and 6.16.6.
- Added new Attachment 6, "VOA Sample Freezer Verification Form" and renumbered remaining attachments.
- Replaced Attachments 1 and 4 with most recent revisions.

Revision 16 dated 9/28/2012
- Add additional detail about client notification and CUR instructions to section 6.7.4.4.
- Added additional clarification about how CUR's are communicated to the PM/PMA to section 16.4.1.
- Revised section 6.13.9 to perform pH check on non-volatile samples for DOD and DOE work only. All other samples will be checked at the bench.
- Revised section 6.16.4 that all water samples for metals (excluding mercury) are delivered directly to the lab (not refrigerated).
- Revised SOP reference in section 6.17.7 to Corporate SOP – "Subcontracting Procedures".
- Corrected typos/section references.

Revision 15 dated 1/31/2012
- Revised Sample Acceptance Policy (Section 5) for concurrence with QAM.
- Revised section 6 to better align with Sample Receiving Checklist.
- Clarified procedure for packing and shipping subcontract work (Section 6.19)
- Removed references to "multi-incremental sampling" or "MIS"
- Revised Attachment 1 (Sample Receiving Checklist).
- Replaced Attachment 4 (Short Holding Time form) and Attachment 5 (Rush Sheet) with new Priority form as Attachment 4 in this revision.
- Removed Attachment 6 (not referenced in text) and Attachment 10 (WI-DV-036) from previous version. Referred to WI by reference in text.
- Reordered and renumbered attachments
- Replaced Attachments 3 and 5 (as currently numbered) with updated forms.

Revision 14 dated 1/31/2011

- Updated language for consistency with TALS and current practice throughout
- Updated Attachment 1 and Attachment 4
- Added Attachment 10

Earlier revision histories have been archived and are available upon request.

Attachment 1a

Example Sample Receiving Checklist

TestAmerica Denver **Sample Receiving Checklist**
DV-QA-0003

Login #:_____ Date/Time Received:_____

Company Name & Sampling Site: _____

Time Zone: • EDT/EST • CDT/CST • MDT/MST • PDT/PST • OTHER State:

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR)

Temp_____ IR#_____ Temp_____ IR#_____ Temp_____ IR#_____ Temp_____ IR#_____

CF__-0.2____Initials_____ CF__-0.2____Initials_____ CF__-0.2____Initials_____ CF__-0.2____Initials_____

Date_____ Date_____ Date_____ Date_____

N/A	*Yes*	*No*		***Initials*** _____
☐	☐	☐	1.	Is radioactivity at or below background? BKG CPM:_____ CPM Reading: _____
☐	☐	☐	2a.	Is a custody seal present on the cooler?
☐	☐	☐	2b.	If yes, is the cooler's custody seal intact?
☐	☐	☐	2c.	Do cooler or samples appear to not have been compromised or tampered with?
☐	☐	☐	3a.	Were samples received on ice?
☐	☐	☐	3b.	Is cooler temperature acceptable?
☐	☐	☐	3c.	Has temperature been recorded?
☐	☐	☐	4.	Is COC present; filled out in ink and legible; and filled out with all pertinent information?
☐	☐	☐	5.	Is the Field Sampler's name present on the COC?
☐	☐	☐	6a.	Are there no discrepancies between the **sample IDs** and/or **collection date and time** on the containers and the COC?
☐	☐	☐	6b.	Are there no discrepancies between the container types and those listed on the COC?
☐	☐	☐	7.	Are samples received within Holding Time?
☐	☐	☐	8.	Do sample containers have legible labels?
☐	☐	☐	9.	Are all sample containers intact (not broken or leaking)?
☐	☐	☐	10a.	Are appropriate sample containers used?
☐	☐	☐	10b.	Are sample bottles completely filled? (Perchlorate bottles ≥ 1/3 head space)
☐	☐	☐	10c.	Is sufficient vol. for all requested analyses, incl. any requested MS/MSDs provided?
☐	☐	☐	11.	No splitting or compositing of samples required?
☐	☐	☐	12.	Do all VOA sample vials have no headspace or bubbles >6 mm (1/4") in diameter?
☐	☐	☐	13.	Were VOA vials labeled as preserved? ☐ HCl ☐ 0-6°C ☐ Sodium Thiosulfate ☐ Ascorbic Acid ☐ Other
☐	☐	☐	14.	Are all samples single phase? (i.e., no multiphasic samples are present.)

Login Checks: ***Initials*** _____

☐	☐	☐	15.	Was a Priority Form completed for any short holds or quick TATs?
☐	☐	☐	16.	Were any tests logged for subcontract?
☐	☐	☐	17.	Were special archiving instructions and login instructions indicated in the Project Notes?

Note Archive Requirements: _____

☐	☐	☐	18.	Were multiple Series logged for this job?

Labeling and Storage Checks: ***Initials*** _____

pH Checks Required? ☐ Yes ☐ No Residual chlorine check required: ☐ Yes ☐ No Quarantined:☐ Yes ☐ No

☐	☐	☐	19.	Was Sample Preservation verified and found to be correct? (excluding VOA, Oil & Grease, and TOC volumes)
☐	☐	☐	20.	Was Residual Chlorine checked and noted on the CUR if present?
☐	☐	☐	21.	If subcontract work was requested, was volume placed on sub shelf?
☐	☐	☐	22.	Were Terracore/Encores delivered to VOA lab?
☐	☐	☐	23.	Did the sample ID on TA label match the client's sample ID on container?
☐	☐	☐	24.	Were stickers for special archiving instructions affixed to each box?

DV-F-0057-1-16-2017-Rev 15 Page 1 of 1

Attachment 1b

Example Alaska Sample Receiving Checklist (page 1 of 2)

TestAmerica Denver **Alaska Sample Receiving Checklist** *(One per Cooler)*

Login #:_____

Receipt of cooler: *Initials*:_____ Date/Time Received:_____Contractor Cooler:_____ Lab Cooler:_____

Date/Time Cooler Opened:_____ Opened by:_____ Number of Coolers:_____

Company Name & Sampling Site: _____

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR)

Unpacking Checks: ***Initials*** _____ (Mark what temp. was taken from = Sample Bottle (S) or Temp. Blank (T)) ***IR Gun#*** _____

Corrected Temps (°C): _____(S or T) Uncorrected Temps (°C): _____(S or T) Correction Factor (°C) _____

N/A	Yes	No		
☐	☐	☐	1.	Did cooler come with Shipping slip (air bill)? Carrier name:_____ Air Bill number:_____
☐	☐	☐	2.	Is the shipper's packing slip attached to this form?
☐	☐	☐	3.	Were custody seals on outside of cooler? How many & Where:_____ Seal date: _____ Seal Name:_____
☐	☐	☐	4.	Were the custody seals signed and dated correctly?
☐	☐	☐	5.	Is the cooler's custody seal intact? Do cooler or samples appear to not have been compromised or tampered with?
☐	☐	☐	6.	Is radioactivity at or below background? BKG CPM:_____ CPM Reading: _____
☐	☐	☐	7.	Were samples received on ice? Was sufficient ice used? Describe type of packing in cooler: _____
☐	☐	☐	8.	Is cooler temperature acceptable? Has temperature been recorded?
☐	☐	☐	9.	Were custody papers in a plastic bag & taped inside the lid?
☐	☐	☐	10.	Is COC present; filled out in ink and legible; filled out with all pertinent information; Field Sampler's name present?
☐	☐	☐	11.	Were custody papers signed in the appropriate place?
☐	☐	☐	12.	Are samples received within Holding Time?
☐	☐	☐	13.	Were all bottles sealed in separate plastic bags?
☐	☐	☐	14.	Are there no discrepancies between the sample IDs on the containers and the COC?
☐	☐	☐	15.	Do sample containers have legible labels? Are all bottle labels complete?
☐	☐	☐	16.	Are all sample containers intact (not broken or leaking)?
☐	☐	☐	17.	Are appropriate sample containers used? Are sample bottles completely filled? (Perchlorate bottles ≥ 1/3 head space) Is sufficient vol. for all requested analyses, incl. any requested MS/MSDs provided?
☐	☐	☐	18.	No splitting or compositing of samples required?
☐	☐	☐	19.	Was Sample Preservation verified and found to be correct? (excluding VOA, Oil & Grease, and TOC volumes)
☐	☐	☐	20.	Do all VOA sample vials have no headspace or bubbles >6 mm (1/4") in diameter?
☐	☐	☐	21.	Adequate number/volume of VOA vials received?
☐	☐	☐	22.	Were VOA vials labeled as preserved? Preservative ☐ HCl ☐ 0-6°C ☐ Sodium Thiosulfate ☐ Ascorbic Acid
☐	☐	☐	23.	Are all samples single phase? (i.e., no multiphasic samples are present)
☐	☐	☐	24.	Was Residual Chlorine checked and noted on the CUR if present?

Login Checks: ***Initials*** _____

N/A	Yes	No		
☐	☐	☐	25.	Samples logged by:_____Date:_____Signature:_____
☐	☐	☐	26.	Was a Priority Form completed for any short holds or quick TATs?
☐	☐	☐	27.	Were any tests logged for subcontract?
☐	☐	☐	28.	Were special archiving instructions and login instructions indicated in the Project Notes?

G:\QA\Edit\FORMS\Sample Receiving\Alaska Sample Checklist_Rev 2.doc 1/31/2017

Attachment 1b

Example Alaska Sample Receiving Checklist (page 2 of 2)

TestAmerica Denver **Alaska Sample Receiving Checklist** *(One per Cooler)*

Note Archive Requirements: _____

☐ ☐ ☐ 29. Were multiple Series logged for this job?

☐ ☐ ☐ 30. Was the project manager called and status discussed? (If yes, give details on the bottom of this form)

Who was called?_____ By Whom?_____ (date)_____

Labeling and Storage Checks: ***Initials*** _____

N/A Yes No

☐ ☐ ☐ 31. If subcontract work was requested, was volume placed on sub shelf?

☐ ☐ ☐ 32. Did the sample ID on TA label match the client's sample ID on container? ***Verified by:*** _____

☐ ☐ ☐ 33. Were extra labels added to pre-tared containers?

☐ ☐ ☐ 34. Were Terracore/Encores delivered to VOA lab?

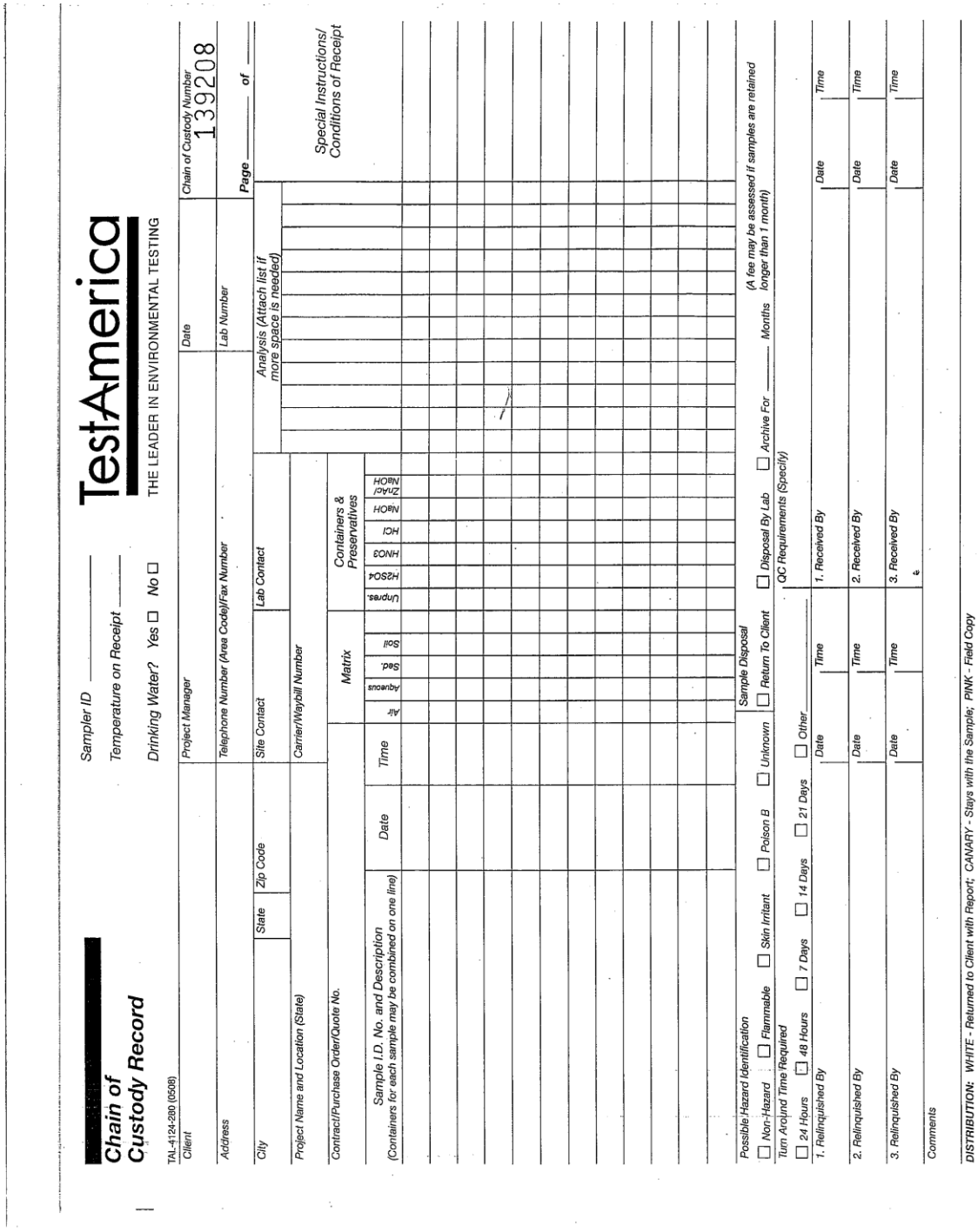
☐ ☐ ☐ 35. Were stickers for special archiving instructions affixed to each box?

SDG labels applied ***(initials):*** _____ ***Verified by:*** _____

G:\QA\Edit\FORMS\Sample Receiving\Alaska Sample Checklist_Rev 2.doc 1/31/2017

Attachment 2

Example TestAmerica Chain of Custody



Chain of Custody Record

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Sampler ID _____ Temperature on Receipt _____ Drinking Water? Yes ☐ No ☐

TAL-4124-280 (0508)

Client | Project Manager | Date | Chain of Custody Number 139208

Address | Telephone Number (Area Code)/Fax Number | Lab Number | Page _____ of _____

City | State | Zip Code | Site Contact | Lab Contact | Analysis (Attach list if more space is needed) | Special Instructions/ Conditions of Receipt

Project Name and Location (State) | Carrier/Waybill Number

Contract/Purchase Order/Quote No. | Matrix | Containers & Preservatives

Sample I.D. No. and Description (Containers for each sample may be combined on one line) | Date | Time | Air | Aqueous | Sed. | Soil | Unpres. | H2SO4 | HNO3 | HCl | NaOH | ZnAcl/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown ☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other _____ QC Requirements (Specify)

1. Relinquished By | Date | Time | 1. Received By | Date | Time
2. Relinquished By | Date | Time | 2. Received By | Date | Time
3. Relinquished By | Date | Time | 3. Received By | Date | Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Attachment 3

Example Condition Upon Receipt Anomaly Form

TestAmerica Denver
Condition Upon Receipt Anomaly Report (CUR)

Login No : _____ Date/Time: _____

Client : _____ Initiated by: _____

CONDITION/ANOMALY/VARIANCE (CHECK ALL THAT APPLY):

☐ **COOLERS** ☐Received, No Chain of Custody (COC) ☐Other:	☐ **CHAIN OF CUSTODY (COCs)** ☐Not relinquished by Client; No date/time Relinquished. ☐ Incomplete Information ☐Other:
☐ **CONTAINERS** ☐Leaking ☐Broken	☐ **TEMPERATURE (greater than 6° C)** ☐Cooler Temp
☐Without Labels ☐Extra ☐VOA Vials w/ Headspace or bubbles > 6 mm ☐Perchlorate without 1/3 headspace ☐Other:	☐ **CUSTODY SEALS (COOLER(S)/CONTAINER(S)** ☐None ☐ Not Intact ☐Other: _____
☐ **CONTAINER LABELS** ☐ Not the same ID/info as in COC ☐ ID COLLECTION ☐Time ☐Date ☐PRESERVATIVE ☐ Incomplete ☐ Markings/Info smeared or illegible ☐ Torn ☐ Other: _____	
☐ **SAMPLES** ☐ Samples NOT RECEIVED but listed on COC ☐Samples received but NOT LISTED on COC ☐Logged based on Label Information ☐Logged based on info from other samples on COC ☐ Logged according to Work Plan ☐Logged on HOLD UNTIL FURTHER NOTICE ☐Other: _____	☐ Trip Blank received, not on COC, _____ vials received ☐ Mislabeled as to tests, preservatives, etc. ☐ Holding time expired ☐ Improper container used ☐ Not preserved / Improper preservative used ☐ Improper pH _____ ☐ Lab to preserve sample ☐ Insufficient quantities for analysis

Comments: _____

Sample Control Review: _____ Date: _____

Revision 4
1/30/2017
G:\QA\Edit\FORMS\Sample Receiving\Att3_DV-QA-0003_CUR_1-30-2017.doc

Attachment 4

Example Priority Form

TestAmerica Denver

Priority Form

Log-In Number: _____ Project Manager: _____

Client: _____

	Initials:	Date/Time:
Receiving		
Dept. Rep. / Analyst		

Time Zone:

EDT/EST CDT/CST MDT/MST PDT/PST

Other:

	HT	Analysis	Min Volume needed (mL)	Method	Sample(s)	MS/MSD Required
	Cr+6	Chromium (VI) (24 h) [Circle Method]	100	3500-Cr B/D or 7196A		
Priority I		Turbidity	50	180.1		
		Biological Oxygen Demand	1000	5210 B		
		Carbonaceous BOD (cBOD)	1000	5210 B		
		Cyanide Preservation	100	335.4 / 4500-CN		
		Color	100	2120 B		
		Nitrite by Spec (COC May Only list Nitrate)	100	353.2 / 4500-NO_2 B		
		Orthophosphate by Spec.	50	365.1*		
		Nitrate by IC	50	300.0 / 9056		
		Nitrite by IC	50	300.0 / 9056		
		Orthophosphate by IC	50	300.0 / 9056*		
		Settleable Solids	1000	SM2540F		
Priority II		VOA 624 Unpreserved	40	624_5ml_UP		
		Dissolved Oxygen	100	4500-O G		
		Free Carbon Dioxide (CO_2)	100	4500-CO_2		
		Sulfite (SO_3^{2-})	100	4500-SO_3 B		
		pH (water)	100	4500-H B / 9040 / 9045		
		pH (soil Hanford)	5 g	9045C		
		Ferrous Iron	100	3500-FE D		

8260 Encores Terracores

☐ **Check if required**: Coring device un-extruded which requires extrusion and freezing within 48 hours.

☐ **Check if required**: A plug of dirt in an empty vial -- place in the freezer within 48 hours for preservation

Preserve: ☐

Preserve: ☐

Filter: ☐

Split: ☐

Composite: ☐

Tests	Samples		Rapidly Expiring	24 TAT	48 TAT	72 TAT	Other:

*Orthophosphate by methods 300.0 and 365.1 require field filtration within 15 minutes of collection.

\\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Sample Receiving\Att 4_DV-QA-0003_Priority Form_Rev 5_7-24-2018

Attachment 5

Example Guidelines for Sample Bottles and Preservatives (page 1 of 3)

TestAmerica Denver

SAMPLE BOTTLES AND PRESERVATIVES

(Unless otherwise noted all samples must be cooled to 4° Celsius)

References 40 CFR 136.3, Table II

	Parameter	Container	Chemical Preservatives
Aqueous Matricies	Alkalinity, BOD, Chloride, Color, Residual Chlorine, Chromium VI, Conductance, Fluorine, MBAS, Nitrite, Orthophosphate, Total Dissolved/Suspended Solids, Sulfate, Sulfite, pH, Nitrate	1000 mL poly HDPE (NM)	None
	Ammonia, COD, TKN, TON, Nitrate + Nitrite, Total Phosphate, TOC	500 mL glass (BR)	2 mL 50% Sulfuric Acid, pH <2
	Phenolics	500 mL glass (BR)	2 mL 50% Sulfuric Acid, pH <4
	TPH, Oil & Grease	Two (2) 1000 mL glass (BR)	4 mL 50% Sulfuric Acid, pH <2
	Metals (excluding Hg), Hardness	500 mL HDPE (NM)	5 mL 20% Nitric Acid, pH <2
	CLP Metals (excluding Hg), Hardness	1000 mL HDPE (NM)	10 mL 20% Nitric Acid , pH <2
	Radiochemistry	Two (2) 1000 mL HDPE (NM)	20 mL 20% Nitric Acid, pH <2
	Carbon-14 (C-14), tritium (H-3)	One (1) 1000 mL amber glass	None
	Total and/or Free Cyanide	250 mL HDPE (NM)	2 mL 50% Sodium Hydroxide, pH > 12
	CLP Total and/or Free Cyanide	500 mL HDPE (NM)	4 mL 50% Sodium Hydroxide, pH >12
	Sulfide	Two (2) 250 mL HDPE (NM)	1 mL 1 N Zinc Acetate **and** 1 mL 50% Sodium Hydroxide, pH > 9
	Volatile Hydrocarbons, GRO	Three (3) 40 mL glass	200 µL Hydrochloric Acid, pH < 2
	Purgeable Organics	Three (3) 40 mL glass	200 µL HCl (pH < 2), if sample is chlorinated add 100 µL 2% Sodium Thiosulfate
	Glycols *OR* Alcohols (DAI)	Two (2) 40 mL glass	None (no headspace)
	Base, Neutral, Acid Compounds, Dioxins	Two (2) 1000 mL amber glass (BR)	None
	Pesticides, PCBs	Two (2) 1000 mL amber glass (BR)	None
	Herbicides	Two (2) 1000 mL amber glass (BR)	None
	TOX	One (1) 1000 mL amber glass	4 mL 50% Sulfuric Acid (pH < 2), if sample is chlorinated add 100 µL 2% Sodium Thiosulfate. Should be eliminated of headspace.
	Extractable Hydrocarbons, DRO	Two (2) 1000 mL glass (BR)	None
	Perchlorate by Method 6860	One (1) 125 mL HDPE/syringe/filter	None, Minimum 1/3 volume as headspace
	Bulk Water Analysis	1/2 gallon or 1 gallon glass (WM)	None
	Aquatic Toxicology Testing	Three (3) 1gallon cubetainers	None
Solid Matricies	Organics, TPH, Metals, Radiochemistry, Oil & Grease	500 mL glass (WM)	None
	Wet Chemistry	250 mL glass (WM)	None
	Purgeable Organics	125 mL glass (WM)	None
TCLP/SLP Matricies	Purgeable Organics (Solid phase)	Two (2) 125 mL glass (WM)	None
	All other analytes (Solid phase)	1000 mL amber glass (WM)	None
	Purgeable Organics (Multiphase)	Two (2) 250 mL glass (WM)	None
	All other analytes (Multiphase)	Four (4) 1000 mL amber glass (WM)	None

NM - Narrow Mouth; BR - Boston Round; WM - Wide Mouth; HDPE - High Density Polyethylene; ⊘ - Does not require temperature preservation

G:\QA\Edit\forms\sample receiving\bottles and preservatives 10-11-2013.doc Rev. 3 - Revised: 10-11-2013

Attachment 5 (cont.)

Example Guidelines for Sample Bottles and Preservatives (page 2 of 3)

INSTRUCTIONS

The bottles contained in this kit have been specially pre-cleaned in order to ensure the integrity of your samples. In addition, any required preservatives have been pre-dispensed into the bottles. In order to further ensure sample integrity and to expedite handling within the laboratory, we ask that you read and follow these instructions:

1. Do not wash out the bottles. They are pre-cleaned and contain the preservatives needed to prevent deterioration of the samples.

Safety Note: BE CAREFUL! The preservatives added to the bottles are highly corrosive.

2. Avoid contamination. Do not mix the contents of different bottles or interchange caps.
3. Use a separate sample label for each bottle. You will notice a sticker with the preservative already attached to each bottle. Please do not cover this up; it helps the laboratory to identify preserved bottles.
4. If duplicates, spikes, and/or spike duplicates are required, please fill an extra set of bottles for each QC sample. If this presents a problem, contact the laboratory for instructions.
5. Determine if field filtering is required. Samples should be filtered **before** filling the bottles. Bottles containing samples which have been filtered in the field should be marked as such. This should be marked on the COC as well.
6. Sample bottles for volatile compounds (including TOX) must be filled completely, leaving no headspace. To check for headspace, turn the bottles upside down to make sure no bubbles are seen. Label each vial or bottle separately.
7. Some parameters have holding times of 48 hours or less. The samples must arrive at the lab within 24 hours of collection. The affected parameters are listed below. Please indicate on the chain-of-custody which (if any) are required.

BOD	CBOD	Orthophosphate by IC	MBAS/Surfactants
Nitrate by IC (300.0)	Nitrite by IC (300.0)	Orthophosphate by 353.2	Nitrate by 353.2
Color	Settleable Solids	Turbidity	Nitrite by 353.2
Nitrite by 4500-NO_2 B	Hexavalent Chromium	Fecal Coliform	Total Coliform

***NOTE**: Hexavalent Chromium and Fecal Coliform have a 24-hour holding time. If you are sampling for either of these tests, please keep the holding time in mind. It is best if the sampling takes place later in the day to avoid missing the holding time due to shipping delays.

8. Some parameters require preservation from the lab within 48-96 hours after the collection time. This includes 3030C metals, potentially dissolved metals, and encores. If you are planning on sending encores to the lab, please be sure to keep the hold time (must be preserved within 48 hours of collection) in mind when sending the samples to the lab. Be sure to let your Project Manager know when you expect the encores to arrive at the lab.
9. Use regular ice for cooling samples. "Blue Ice" packs placed inside plastic bags are recommended only when regular ice is not available.
10. In case problems arise with your sample shipment, please indicate the name and telephone number of a person who can be contacted. This person should be available at the time the samples are expected to arrive at the lab, including Saturdays.

If you have any questions, please call (303) 736-0100

G:\QA\Edit\forms\sample receiving\bottles and preservatives 10-11-2013.doc Rev. 3 - Revised: 10-11-2013

Attachment 5 (cont.)

Example Guidelines for Sample Bottles and Preservatives (page 3 of 3)

CHAIN OF CUSTODY INSTRUCTIONS

IF THE CUSTODY SEAL ON THE OUTSIDE OF THE SAMPLE BOTTLE KIT IS BROKEN UPON RECEIPT, **DO NOT CONTINUE!!!** THE BOTTLES MAY HAVE BEEN TAMPERED WITH DURING SHIPMENT. CALL YOUR PROJECT MANAGER AT (303) 736-0100 AND EXPLAIN THE SITUATION. THE PROJECT MANAGER WILL ADVISE YOU AS TO HOW TO CONTINUE.

Completely filling out the CHAIN OF CUSTODY record is very important. Please be sure to include ALL sample collection dates and times (military time only); the sample ID's (as you wish them to be reported on your final data report) and the analytical tests requested. Your company name, project contact and company address and phone number must be included on the CHAIN OF CUSTODY if the laboratory is to accurately perform and report the requested tests. Make sure that turn around times that differ from the quote, short holds, and any other special instructions are properly marked on the chain of custody to ensure the laboratory can identify such items. **We have attached a sample copy for your reference.**

In order to fully relinquish the samples to the laboratory, you must sign, date and record the time you transferred the samples to the laboratory. If you are shipping the samples to the laboratory, relinquish at the time you relinquish the samples to the shipper. You may mark the shipper under the comments section or the carrier/waybill number section of the chain of custody if appropriate.

After you have relinquished the Chain of Custody, keep the pink copy for your records and place the other two copies into the supplied plastic ziploc bag. This is important. If the ziploc bag is not closed, ice water may damage the chain of custody. Be sure to place the ziploc bag into the sample kit when you return the filled sample bottles to the laboratory.

Please feel free to contact your TestAmerica Project Manager if you have any questions on filling out this document.

G:\QA\Edit\forms\sample receiving\bottles and preservatives 10-11-2013.doc Rev. 3 - Revised: 10-11-2013

Attachment 6

Example VOA Sample Freezer Verification Form

VOA Sample Freezer Verification Form
Freezer ID:_____

JOB #	VERIFIED NO WATER SAMPLES PUT IN FREEZER BY:	Date Sample Placed in Freezer:	Time Sample Placed in Freezer:

G:\QA\Edit\FORMS\Sample Receiving\VOA Freezer Check_Rev 2.doc
Revision 2 12/06/2012

Attachment 7

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

User Guide for Logging Samples into TALS

To Start:

1. From the main menu, select *Sample Management* by double clicking the application.
2. From the Sample Management menu, select *Login* by double clicking the application.

To Receive a Sample Delivery Group

1. Click the [NEW] button on the toolbar located at the top of the screen.
1. Click [Yes] to create a new Login.

Receipt - Info

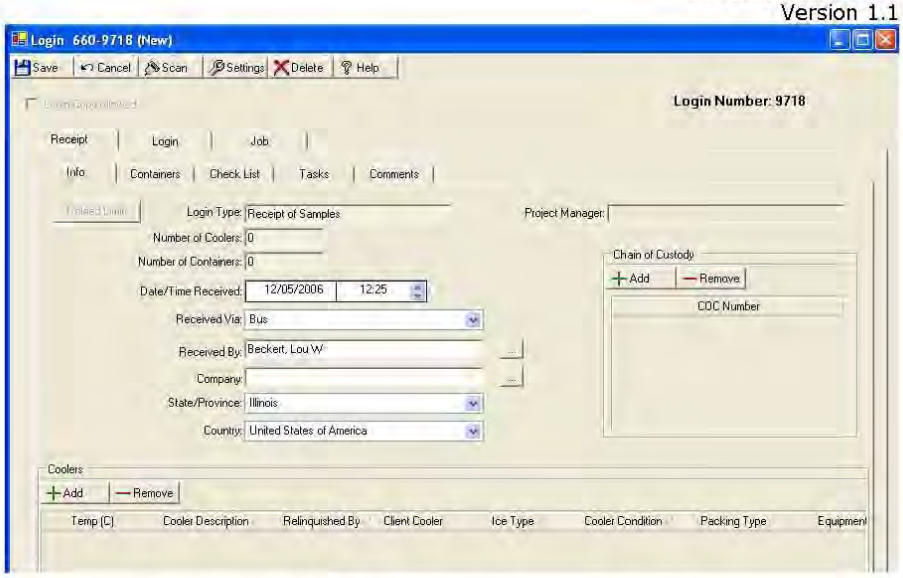
When samples arrive at the laboratory, they must first be received in the LIMS. By keeping these modules separate, the sample receiving group can start the login, receive the samples and print the labels for the sample. Rush samples can then be moved directly into their respective labs to begin work before the samples are completely logged. Until the sample login is complete, the samples will not relate to the login. All samples MUST be received before the login can be completed.

1. In each field below, enter the following:

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



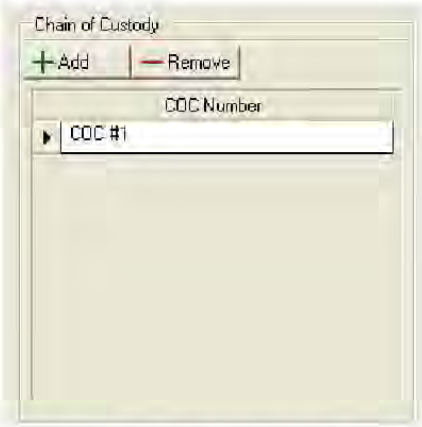
- **Login Type** – this defaults to Login when a new login is chosen
- **Number of Coolers** – this will be filled in with the information supplied in the cooler section
- **Number of Containers** – this will be filled in with the information supplied in the container tab
- **Date/Time Received** – this will default to the time the login was created. To change this date/time to the actual receipt date/time noted on the COC, use the drop down/up arrows in each field
- **Received Via** – mode of delivery of the samples (courier, client pickup, etc)
- **Received By** – this is the individual who received the samples and signed the chain of custody.
- **Company** – this is the client who sent the samples. This is an optional field
  - Note: *If a company is chosen in this field, the **PROJECT LOOKUP** on the **LOGIN TAB** will automatically be filtered to display active projects from only that company.*
- **State/Province** - Enter the State where samples were collected – this is a required field
- **Country** – this defaults the United States but must be changed if the samples were actually taken outside of the United States.

2. Click [Add] in the "Chain of Custody" section to enter the Client's Chain of Custody numbers.
   - Note*: Entering one (1) chain if custody will automatically add the COC number to all containers. If more than one COC is entered, a COC must be selected manually for each container. This may be done on the Containers tab*

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



3. Click [Add] in the "Coolers" section of the screen to enter the Cooler information:

- **Temp (C)** - the internal temperature of the cooler upon receipt
- **Cooler Description** –a description of the physical cooler, whether it be a thermal cooler, insulated box or other container
- **Relinquished By** –the individual that signed the Chain of Custody and relinquished the custody of the samples to the laboratory sample custodian
- **Client Cooler** – Check this box if this is a client provided cooler (not an STL cooler)
- **Ice Type** – type of ice that the samples were packed in (drop down list)
- **Cooler Condition** – the physical condition of the cooler upon arrival
- **Packing Type** – the type of cushioning material used to keep the samples from breaking in transport
- **Equipment** – any equipment (sampling equipment, balances, palm pilots, etc) packaged with the samples
- **Comments** – free form text field to enter any additional information about the cooler

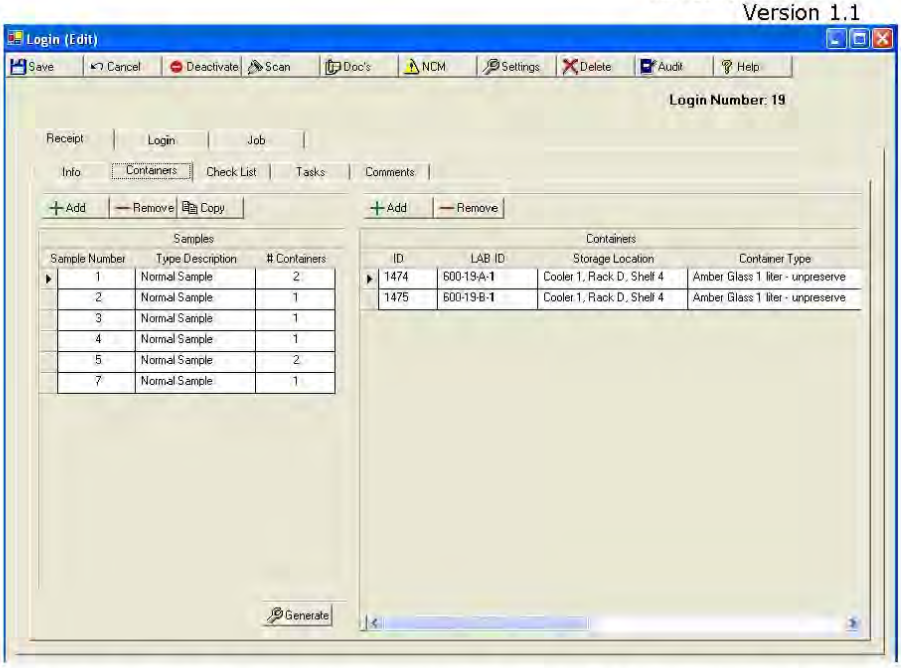
Receipt - Containers

1. Click [**Add**] to enter the number of SAMPLES RECEIVED in the SAMPLES Section of the form.
2. Enter the total number of containers received per sample in the space provided in the **#Containers** field.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



3. Click [**Generate**] to create the container records in the CONTAINERS Section of the form.
   - Containers can also be added manually for each sample by selecting the sample in the Samples grid and then clicking the [Add] button on the Containers grid. The box below will be displayed to enter the number of containers desired for the sample.

4. After generating the CONTAINER records, fill in the following information in each of the fields in the container grid (fields are represented from left to right - scroll to the right to see the remaining fields)
   - **ID** - this is the unique container id assigned by the LIMS - no entry is allowed in this field
   - **Lab ID** - this is a combination of the login location, the login number, the alpha occurrence of the container for that sample (A = 1, B = 2, etc.) and the sample number itself. The ID and Lab ID both indicate the same container and can be used interchangeably. No entry is allowed in this field

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

- **Storage Location** – storage locations are set up in the system defaults and populate the drop down list used in this field
- **Container Type** - use the drop-down arrow to select from a valid values list of containers
- **Container Volume**- will auto-fill based on the selection in Container Type
- **Container Units** – will auto-fill based on the selection in Container Type
- **Condition** – condition of container when received. Enter only exceptions to a condition of good.
- **COC** – use the drop down arrow to select available Chain of Custodies. If no COCs were entered on the info tab, this field will be blank. If there is only one COC, the field will be auto-filled
- **Cooler** – select from the drop down list if more than one cooler was received or the field will be auto-filled if there is only one cooler.
- **Tag** – CLP sample containers may arrive with a tag. Enter the tag id in this field

Note: *This information must be completed for all containers/all samples.*

Some shortcuts are available:

A) *Simultaneously pressing the **<CTRL>** key and the **<DOWN>** arrow will automatically fill in the field below the field the cursor is on (as long as the new field is blank).*

B) *Some fields allow filling in every row of a column automatically. Simply highlight the HEADER of a column (Storage Location for example). This will turn the entire column BLUE. Right-Click in the blue and select a value from the valid values list. All fields in this column will now have the selected value.*

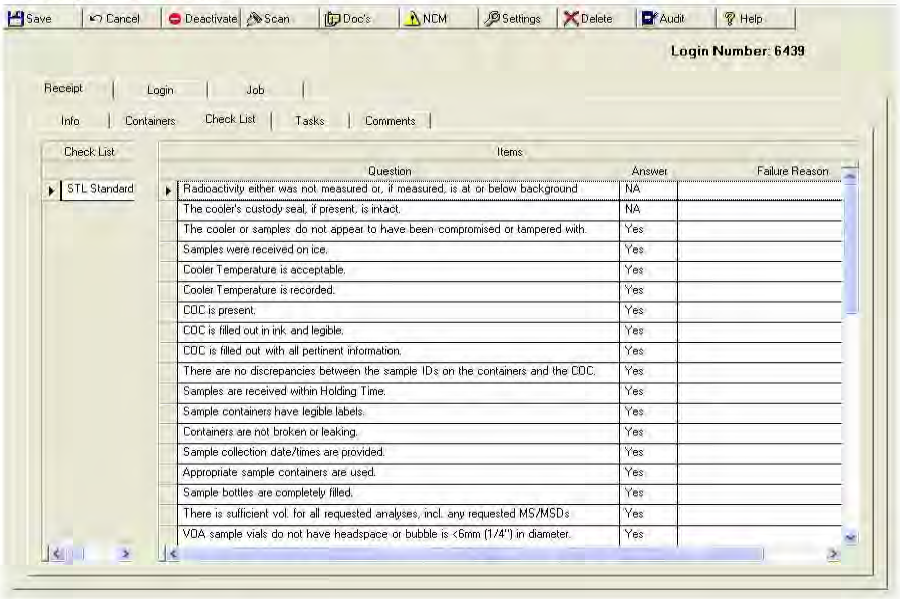
C) *Use the **[COPY]** button in the SAMPLES Section of the form. Simply fill out all of the information in the CONTAINERS Section of the form for one sample. Click on COPY. Choose the completed sample on the left-side of the form ("copy from") by clicking the left-most grey square (causing that row to be completely highlighted) and then choose the samples to "copy to" in the right-side of the form by clicking the left-most grey square of the row (causing that row to be completely highlighted). For more samples, simply hold the mouse button down when selecting the first sample and "drag" the highlight down the list of samples until all of the samples to copy are highlighted. Click [OK] and the container information will be copied to the new records.* **Note**: *The copy function only works if the samples have the same number of containers.*

Attachment 7 (cont.)

Login

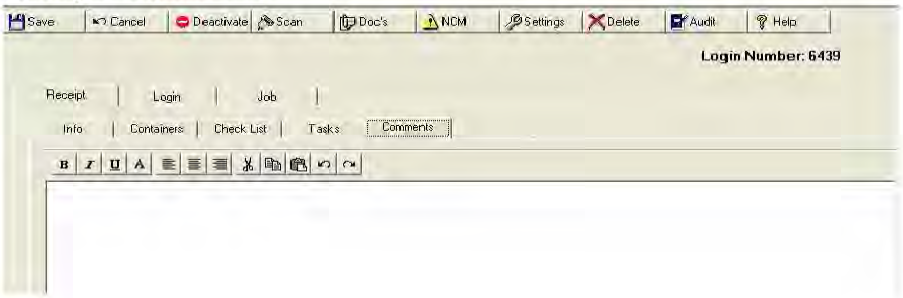
Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

Receipt - Checklist



1. Answer all questions.
2. A Failure Reason must be entered for all negative responses
3. If the question does not apply to this set of samples (i.e. a question pertaining to solid samples for a shipment of only waters) N/A may be used as a response.
4. To quickly enter the most common answer for all questions, highlight the HEADER of the ANSWER COLUMN. This will turn the entire column BLUE. Right-Click in the blue highlight and select an answer (Yes, No, N/A). All fields in this column will now have the selected response.

Receipt - Comments



Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

Free-form area to enter any comments, notes, etc. These can appear on backlogs if desired.

Save

1. Click Save from the menu across the top of the screen to save the receipt information entered to this point.

To Login a Sample Delivery Group

1. Locate the Login by either clicking on the Find button (which will bring up a list of all the logins for this location or that have work for this location) or the Login # button where the login number can be entered directly. Either choice will load the login.
2. In the Login tab, click the [**Add**] button next to the PROJECT NUMBER field. If a COMPANY was chosen on the RECEIPT TAB, the list of projects will be filtered based on that company. Otherwise a complete list [of Active Projects] will be displayed. Filter the list based on the pop-up screen and select the appropriate project.
3. Click [OK]
    - Note: If the wrong PROJECT is selected, this must be changed before continuing. Save the login, click edit and then click on the [**Change**] button and follow the instructions.

Login - Samples

1. Enter the information for each Client Sample. Some fields will auto-filled based on information from the previous tabs and the Project. Update information as needed.
    - **Sample Number** – assigned by the system as each sample is added. If samples are removed from the login, the sample number does not renumber
    - **Type Description** – type can be normal sample (client sample), or a qc sample (ms, msd or sample duplicate)
    - **Customer Sample ID** - Enter the Client Sample ID accurately, as it was entered on the COC. If the Project has "Sites" associated with it, and the PM elected to verify against the site samples list, this field will NOT be free-form, but will be a drop-down list to select the appropriate Client Sample ID.
    - **Sample Matrix** - select the Sample Matrix from the drop-down list. This is the sample matrix that the lab will see on their backlogs and in Analyst Desktop. This is the matrix that is used in the reference data as well. If the client needs the matrix to be displayed on the report and EDD as something more specific such as 'ground water', use the alternate sample matrix field.
    - **Sample Date** – enter the sample date for each sample as it is entered on the COC. Click the down arrow to bring up a calendar to select the date from.

Attachment 7 (cont.)

- **Sample Time** – enter the sample time as entered on the COC. To enter the date in military time (24 hour clock), change the format by clicking on the 

button at the top of the screen, then select 24 hour.
- **Time Zone** – change the time zone to the zone where the samples were taken
- **Hazard Level** – For laboratories with a Hazard Level Permit, enter the hazard level of the samples as received.
- **Alternate Sample Matrix** – as mentioned above, the client may require that a more descriptive sample matrix appear on their deliverables. Alternate Sample Matrices are added in the Site Sample module and are available as a drop down in the Login module.
- **Action Limit Set** - If ONE Action Limit Set has been associated with this PROJECT, the list name will automatically fill in. If more than one exists, you must choose the appropriate list form the drop-down choices.
- **Sample Comments** – enter any additional information about the sample that may need to be conveyed to the analytical group.

Client Requested QC Samples

If sample QC is received from the client and it is necessary to create this QC at login (MS/MSD/Duplicates, etc.) follow this procedure:

1. Select the sample by clicking on the sample Number or Type Description field.
2. Right Click to pull up the **Sample Options** menu and select **Add QC SAMPLES.**
3. Choose the appropriate QC types from the popup box which will appear and click [Add].
4. Populate the appropriate information for the QC samples
5. Click back to the **LOGIN TAB – CONTAINERS SUBTAB** to add the appropriate information for the containers received specifically for the QC.

Login – Groups

The analyses associated with each sample are contained in LOGIN GROUPS. Sample Receiving will build these groups from the information contained in the PROJECT.

The groups can be created a variety of ways, ranging from easy to complex. The most straight-forward way is to organize your groups in relationship to the samples and the analyses requested on each sample.

For example:

If sample #1 and #3 are both waters and are requesting GCMS VOA8260 Standard List and Lead ... create a LOGIN GROUP with these two parameters and associate this group to these samples.

If sample #2 is only getting Lead ... create another LOGIN GROUP that only contains Lead and associate this group to this sample.

Attachment 7 (cont.)

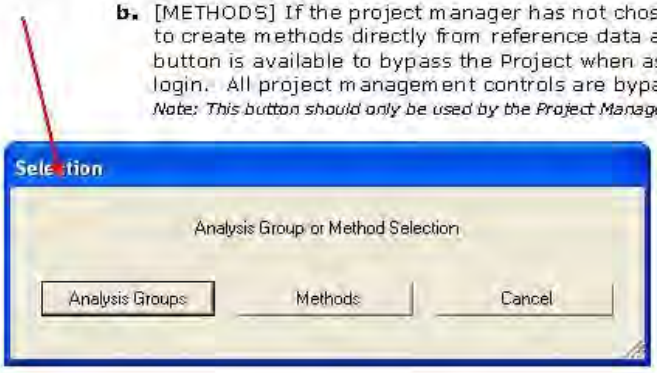
Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

In this example, you would have two login groups (with the correct analyses on each sample).

To CREATE A NEW LOGIN GROUP:

1. Click on the **[NEW]** Button.
2. From the pop-up menu click **[Analysis Groups].**
   - **a.** [CANCEL] to cancel action
   - **b.** [METHODS] If the project manager has not chosen to block the ability to create methods directly from reference data at Login, the Methods button is available to bypass the Project when assigning methods to a login. All project management controls are bypassed at this point. *Note: This button should only be used by the Project Manager.*



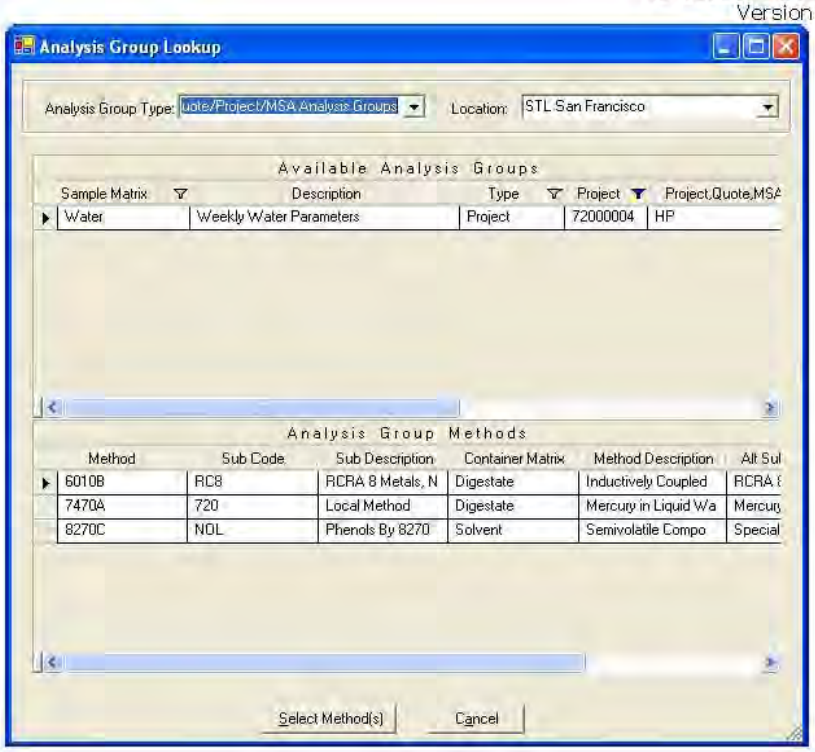
You are now viewing all of the methods/groups the Project Manager has created in the PROJECT.

1. (Paying attention to the Matrix and the Group Description) highlight the Group that contains the methods of interest.
2. Once you choose a group, the associated METHODS will appear in the lower grid of this pop up.
3. Highlight the method(s) of choice and click on **[Select Method]**. You now have a LOGIN GROUP with the chosen Methods and their corresponding preps.
4. Click **[Add]** to add more methods to this LOGIN GROUP.
5. (or) Click **[New]** to create a new LOGIN GROUP.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



A. You may view each LOGIN GROUP created by highlighting the LOGIN GROUP in the "Login Groups" grid by clicking the left-most grey square and highlighting the entire row.
B. Once highlighted, the corresponding methods/preps will be displayed in the grid to the right.
C. The grid will be titled with the LOGIN GROUP NAME (which automatically comes from the PROJECT, but can be renamed by you simply by typing over the title in the LOGIN GROUPS grid).
D. Columns in the LOGIN GROUP [DETAIL] GRID:
    i) Method Code – the internal ID of the method
    ii) Method Location - the originating location of the method
    iii) Method Description – detailed description of the method
    iv) Alt Sub-Desc – this field is customized in the PROJECT to indicate to the lab if the list has been modified from it's original state.
    v) Destination – if method is being sent to another facility to be analyzed, this field will have the information of that location, otherwise, it will be your current location.
    vi) TAT – The Turnaround Time of the Method

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

vii) Condition – the current condition of the method (Active, Cancelled, On Hold)
viii) Status – the current status of the method (Ready, Batched, 1st Level Reviewed, etc.)
ix) Phase – currently not used.
x) Sub-Desc – the default description of the sub-list chosen at creation.
xi) Holding Time Calculation – Method used to calculate holding time
xii) VTSR – if checked, the holding time will be calculated off of the RECEIPT DATE and follow the holding time setup for this occurrence.
xiii) Up Rpt Limit Type/Low Rpt Limit Type – these fields work together to advise you how the method will be reported. The "Upper Limit" is the value at which the sample will be reported (usually the RL), while the "Lower Limit" is the [lowest] value at which the system will report a value (usually the MDL).
xiv) Container Matrix – the matrix of the sample expected for this method
xv) Reporting Basis – the basis of reporting this method such as a "Dissolved" Metal versus a "Total" Metal.
xvi) Calibration Group – currently not used
xvii) Calibration Curve – currently not used
xviii) Dry Wt Adjust – Will this method be corrected for Dry Weight?
xix) # Tics – How many TICs do you want to report for this method? This would be the MAX amount of TICs reported.
xx) Method Comments – free flow comments
xxi) Reporting Rules – currently used for dual column analyses to indicate which rule to follow when reporting both columns.
xxii) Do Not Report – currently used to inhibit a method from printing on the final report, although the method is needed in the login for additional analyses (such as reporting the calculated result of HARDNESS, but not wanting to report the individual analyses required to achieve the hardness calculation)

Note: *A "grey background" in the METHODS/PREP window indicates this login group is not associated to the sample highlighted in the "SAMPLE GROUPS" grid. A "white background" indicates this group is associated to the sample highlighted. Additionally, the LOGIN GROUP in the "Sample Groups" grid will turn blue to indicate which LOGIN GROUP has the focus.*

6. Once the LOGIN GROUP(S) has been created, you will associate the LOGIN GROUP to the correct sample(s).
   A. Simply put a check-mark in the square corresponding to the correct sample and the correct login group.
   B. A shortcut is available here as well. Simply highlight the HEADER of the LOGIN GROUP in the "Sample Group" grid (shown above by the "1" and "2") by right-clicking, then left-click on your selection.
7. To **CANCEL** a method (or to place a method **ON HOLD**), highlight the appropriate SAMPLE in the sample grid, and the appropriate LOGIN Group.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

a. Change the CONDITION of the method from ACTIVE to either CANCEL or ON HOLD as needed.
b. Changing the CONDITION of a prep method will automatically update the condition of the analytical method.

Login – Limits

This tab will be reviewed by the Project Manager to assure all of the appropriate limits have been loaded into the LOGIN.

Login – Questions

This tab may have information that needs completion. If a deliverable requires the collection of additional information that is normally not captured in the LIMS, a question will be used to hold this additional information. The answer to the question is the data that will be stored for the required field.

Example:

Question: **What is the sample depth listed on the Chain of Custody?**
Answer: **4.5**

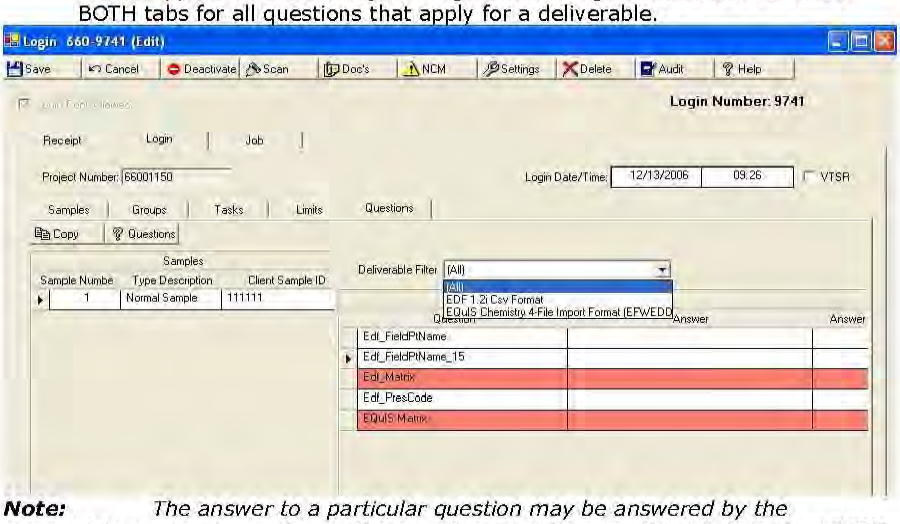
- Each deliverable may have its own set of questions. To view the questions for a deliverable, use the 'Deliverable Filter' to select the deliverable name from the deliverables that have been added to the job. After selection, the list of 'questions' will appear in the box below the Deliverable.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

- Required questions are highlighted in '**Red**'. Answers to these questions are required by the deliverable and/or the client and must be completed. Questions that are not highlighted are optional
- Also note – when questions are created in the deliverable, they are designated as either Login Sample Questions OR as Job Questions. Questions that are located under the Login –Questions tab are questions based on the Login and/or Samples for this login only. Questions located under the Job – Questions tab are based on the Job (which may include multiple logins) and will be applied to the entire job – regardless of login. Make sure to check BOTH tabs for all questions that apply for a deliverable.



Note: *The answer to a particular question may be answered by the appropriate group, depending on the question. Just because this tab is in the LOGIN Module doesn't imply that the answer will come from Sample Receiving.*

Job – Job

A JOB is a set of samples from a single Login or multiple Logins. Samples are reported and invoiced as JOBS.

The JOB Tab is primarily a Project Manager function. This information is copied down from the associated Project.

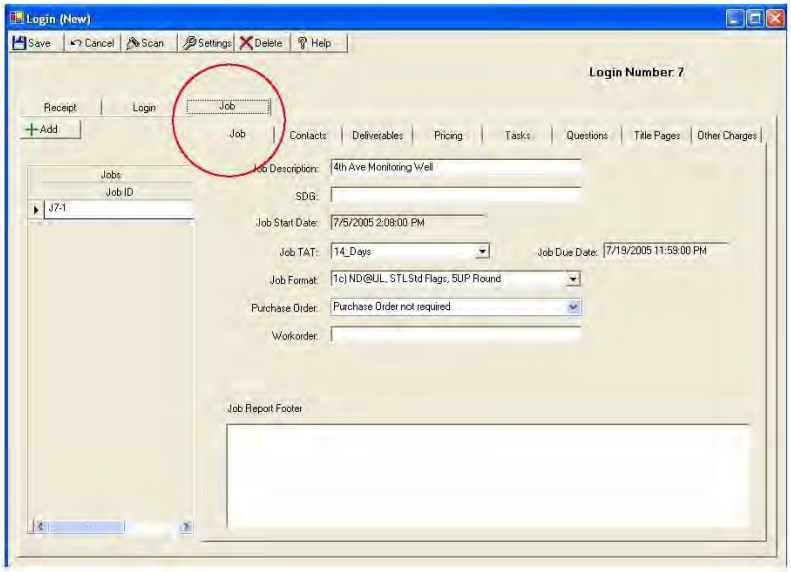
1. Job Description – carried over from Project
2. SDG – and alternate naming convention for joining multiple LOGINS into a JOB for reporting.
3. Job Start Date – carried over from Project
4. Job TAT – turnaround time defaulted from Project. Turnaround Time can be modified per Login and Job.
5. Job Due Date – calculated from turnaround time specified on Project.
6. Job Format – the default formatter from Project. Formatter can be changed per Login and Job.
7. Purchase Order – carried over from Project. PO can be changed per Login and Job.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1

8. Workorder - carried over from Project. Workorder can be changed per Login and Job.



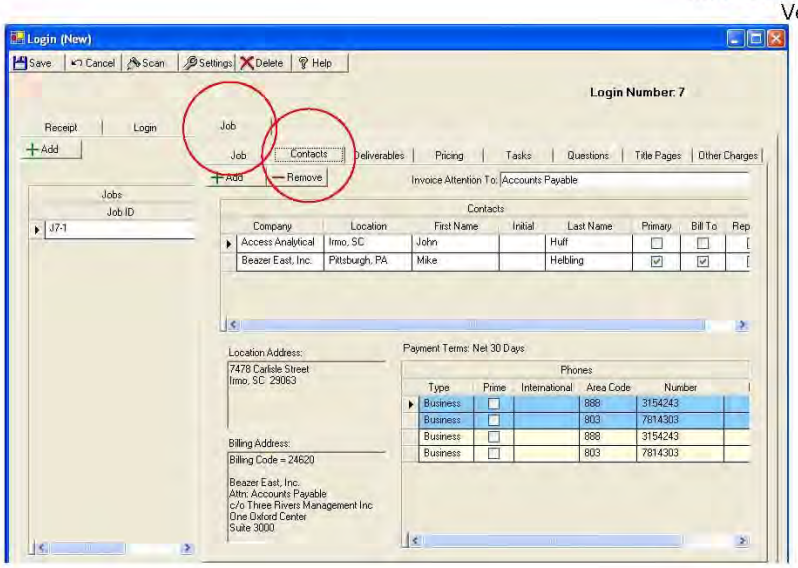
Job – Contacts

The Contact information is copied down from the Project exactly as built. The information can be modified per job. Additional contacts may be added as needed.

Attachment 7 (cont.)

Login

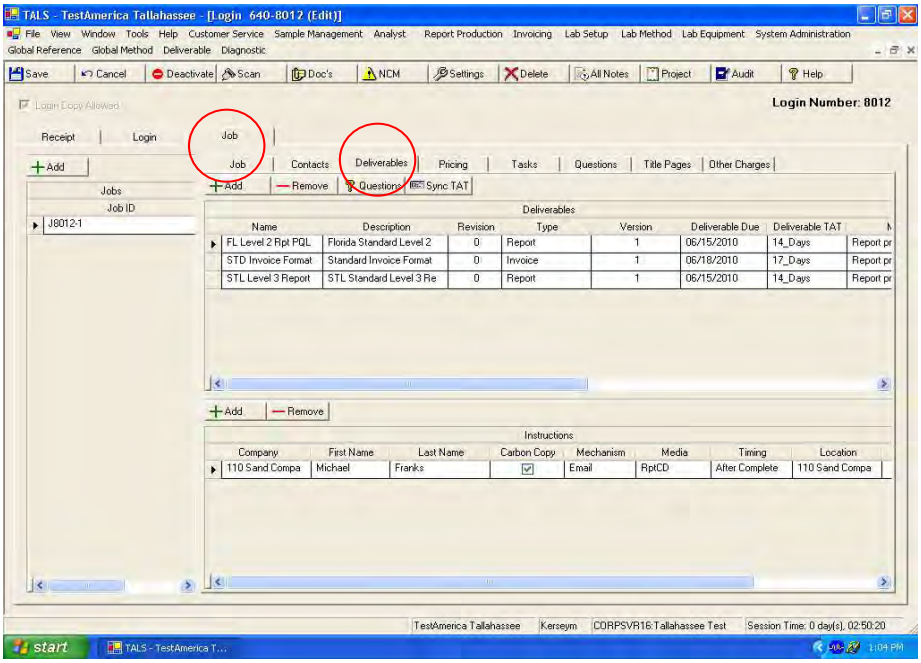
Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



Job – Deliverables

The Deliverable information is copied down from the Project exactly as built. The information can be modified per job. Additional Deliverables may be added as needed.

Attachment 7 (cont.)



Job – Pricing

The Pricing is copied down from the Project. Pricing can be modified and additional JOBS can be added, if needed.

To add another JOB to a Login:

1. Click [Add]
2. A job can contain any combination of methods/samples/logins. You will assign these on the Pricing tab
3. Sort the Pricing grid by any combination of the headers. Sort and Filter the list to obtain the desired set of samples that you want on another job.
4. Left click the Job ID header to activate the column. Right-click the JOB ID header and choose the job # to assign desired job to samples/methods in the sorted grid.

Job – Task

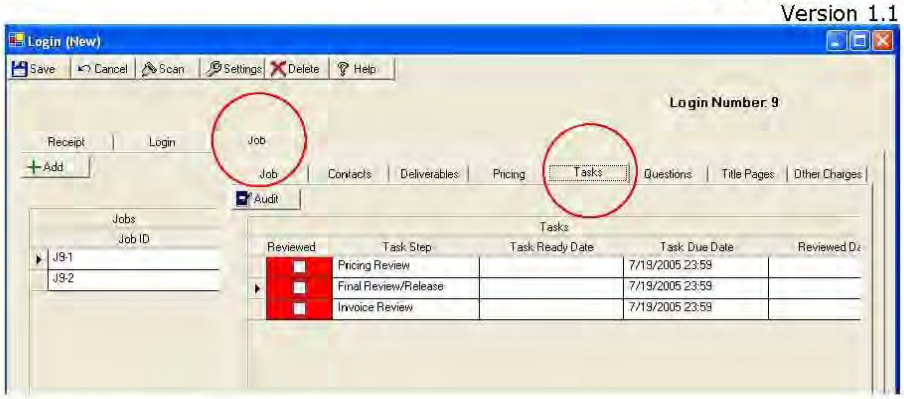
1. As Tasks are completed (simply by placing a checkmark in the space provided next to the task), the date/time and user are recorded.
2. Tasks are hierarchal, and the previous Task must be completed before completing the next task.

Note: The Login Review Task must be checked on the Login – Task tab prior to setting the review tasks in the Job tab.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



Job – Questions

This tab may have information that needs completion. If a deliverable requires the collection of additional information that is normally not captured in the LIMS, a question will be used to hold this additional information. The answer to the question is the data that will be stored for the required field.

Example:

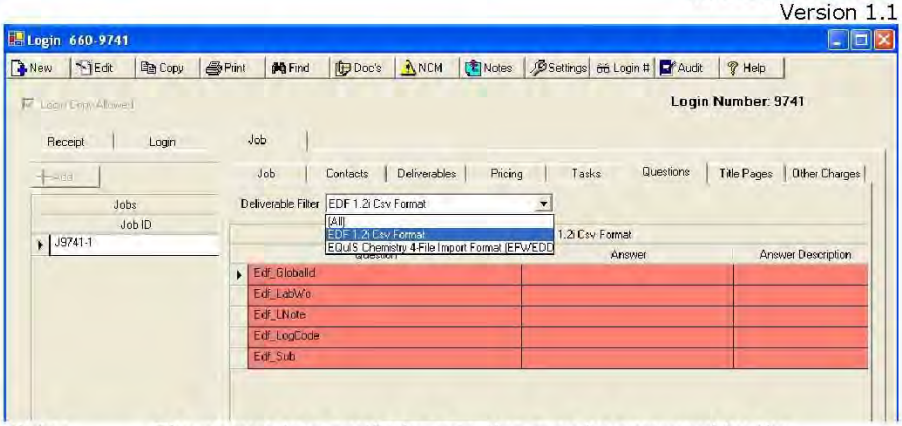
Question: **What is the sample depth listed on the Chain of Custody?**
Answer: **4.5**

- Each deliverable may have its own set of questions. To view the questions for a deliverable, use the 'Deliverable Filter' to select the deliverable name from the deliverables that have been added to the job. After selection, the list of 'questions' will appear in the box below the Deliverable.
- Required questions are highlighted in 'Red'. Answers to these questions are required by the deliverable and/or the client and must be completed. Questions that are not highlighted are optional
- Also note – when questions are created in the deliverable, they are designated as either Login Sample Questions OR as Job Questions. Questions that are located under the Login –Questions tab are questions based on the Login and/or Samples for this login only. Questions located under the Job – Questions tab are based on the Job (which may include multiple logins) and will be applied to the entire job – regardless of login. Make sure to check BOTH tabs for all questions that apply for a deliverable.

Attachment 7 (cont.)

Login

Issued: 01.31.06
Re-issued: 00.00.00
Version 1.1



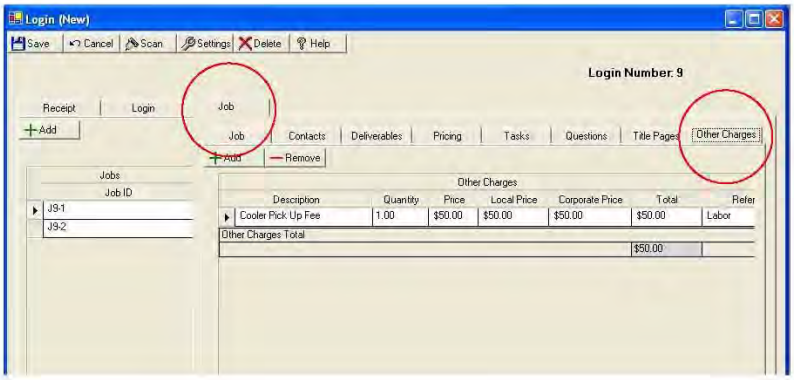
Note: *The answer to a particular question may be answered by the appropriate group, depending on the question.*

Job – Title Pages

Not used.

Job – Other Charges

Non-Analytical charges are copied from the Project. [**Add**] / [**Remove**] **Other Charges** as needed.



SAVE the Login.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Selecting and Using Balances

Approvals (Signature/Date):

Doug Gomer 6/27/19
Doug Gomer Date
Environmental Health and Safety Coordinator

Roxanne Sullivan 6/27/19
Roxanne Sullivan Date
Quality Assurance Manager

Charles Newton 6/27/19
Charles Newton Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 PURPOSE AND SCOPE

1.1 This procedure describes requirements, responsibilities, and instructions for using analytical and top-loading balances. Specifically this procedure provides guidance for the selection of balances, initial set up of balances, daily calibration checks, acceptance criteria for balance performance, and trouble shooting and corrective actions for balance performance problems.

1.2 This procedure implements guidance presented in ASTM D 4753-02, *Standard Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing*.

1.3 This procedure applies to all TestAmerica Denver operations that require the use of a balance, either top loading or analytical, to weigh samples, reagents, and/or standards, as part of performing an analytical method, or to perform calibrations of volumetric glassware and pipettes.

2.0 SAFETY

2.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual, and this document.

2.2 Specific Safety Concerns or Requirements

2.2.1 In almost all cases, there are no hazards associated with using a balance to weigh standard weights to verify a balance calibration. Balances are used in analytical laboratory areas, and as such, safety requirements for the area apply. Balances are typically used to weigh chemicals in the course of performing an analytical method. Therefore, the safety section of the applicable SOP shall apply when using a balance as part of an analytical method.

2.2.2 In some cases, it may be necessary to eliminate static in an enclosed balance chamber. The device most commonly used is a Staticmaster® ionizer, manufactured by Amstat Industries, Inc. This device consists of Po-210, a radioisotope that emits alpha particles, pressure welded into a thin strip of gold and silver foil. Amstat also produces ionizing brushes that contain the Po-210 radioisotope. The following precautions apply when using a static eliminator that contains a radioactive source:

2.2.2.1 Only a radiation worker may handle the device that contains the radioactive source. All other workers must not handle the source, but may use the balance.

2.2.2.2 Nitrile or latex gloves must be worn when handling the source.

2.2.2.3 The balance must have a label showing that radioactive material is located inside the balance chamber.

2.2.2.4 The source must be entered into the TestAmerica Denver source registry.

2.2.2.5 The source must be given to the Radiation Safety Officer (RSO) when no longer being used.

2.2.3 In general, when working in a laboratory area, eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

2.3 Primary Materials Used

There are no materials used while performing this procedure that have a serious or significant hazard rating.

NOTE: The Po-210 radioactive source described in Section 2.2.2 above is not hazardous when used as intended. Alpha radiation, while a strong air ionizer, presents no external radiation hazard. It is incapable of penetrating an ordinary sheet of paper or a person's epidermal layer of skin. Air stops alpha particles at a distance of about two inches. The isotope is pressure welded into a multi-layer gold and silver foil that is insoluble and inert in most chemicals. Because the isotope is an integral part of the foil, it is vibration and impact resistant.

3.0 DEFINITIONS

3.1 The following definitions are taken from ASTM D 4753-02, Standard Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing.

3.1.1 Balance: An instrument for determining the mass of an object by the action of gravity on the object.

3.1.2 Capacity: The maximum mass recommended by the manufacturer, disregarding any additional capability supplied by a taring device.

3.1.3 Readability: The value of the smallest unit of mass that can be read without estimation over the given range of measurement, either directly or by use of a vernier or micrometer.

3.1.4 Sensitivity: The ratio of the deflection (ΔL) of the balance indicator or self-indicating display to the mass (ΔM) causing the deflection; $S = \Delta L/\Delta M$ at a given mass.

3.1.5 Standard Mass: An object of specified mass and construction used with balances, and for the verification of balances and other masses.

NOTE: **For the purpose of this SOP, the terms "mass" and "weight" are synonymous. Therefore a standard mass is also a standard weight. Likewise, the act of weighing an object is the same as determining the mass of an object.**

3.1.6 Tolerance: A value fixing the limit of allowable error or departure from true performance or value.

3.2 Analytical Balance: For the purpose of this SOP, an analytical balance is equipped with an enclosure that protects the balance pan from air currents, and has a readability of 0.0001 g or better.

3.3 Top-Loading Balance: For the purpose of this SOP, a top-loading balance typically consists of a balance pan mounted on a box containing the balance mechanism and an electronic display, and has a readability of 0.1, 0.01, or 0.001 g.

3.4 Daily Calibration Check: The verification of a balance calibration that is performed each day prior to first use by weighing standard masses and comparing the measured value to the certified value to demonstrate that the balance is performing within established tolerance limits.

3.5 QA Department Weights: ASTM Class 1 weight sets stored in the QA area. These weights are not used for working proposes or daily checks and are marked "QA" on their cases. The QA Department Weights are re-certified by an approved vendor at least every 5 years (See Section 5.7).

NOTE: Currently the approved vendor for recertifying ASTM Class 1 weights is Quality Control Services, 2340 SE 11th Avenue, Portland, OR 97214.

3.6 Lab Working Weights: ASTM Class 1 weight sets used for daily lab checks and stored in the lab. The Lab Working Weights are checked annually (± one month), by the QA Department, against the QA Department Weights (see Section 5.8). They are also re-certified by an approved vendor at least every 5 years.

3.7 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 EQUIPMENT AND SUPPLIES

4.1 Analytical or top-loading balance that is appropriate for the application for which it is used. The balance must have a current service label. If the label indicates that service is overdue do not use the balance and notify the Group Leader and/or QA group.

4.2 Weights

Each weight or set of weights that is used must have a current calibration. The weight sets are labeled with the last calibration date and expiration date. If the calibration is overdue or calibration label missing, do not use the weights. Notify

the Department Supervisor or Department Manager and/or QA group.

4.3 Logbook for recording daily calibration checks: Logbooks have been designed specifically for either an analytical balance or top-loading balance (see Attachments 1 & 2), and contain tables of control limits that are appropriate for the type of balance (see Attachments 3 & 4). The logbook must have the balance ID documented on the cover page and each page that a daily calibration check is performed.

5.0 PROCEDURE

5.1 Balance Selection

5.1.1 Determine how the balance will be used, e.g., to weigh reagents, standards, and/or samples for a specific analytical method; to calibrate pipettes; to calibrate volumetric glassware; etc. A balance must have a capacity sufficient to accommodate all anticipated test weights, and its performance must be adequate so as not to be a cause of test error.

5.1.2 Determine the range, capacity, readability, and accuracy needed for the specific weighing application. This may be dictated in a standard method or may rely on the accuracy needed for the specific measurement. For balances that are used for many applications, the application with the most stringent quality requirements will dictate the balance selection.

Example 1: Standard Method 2320 B for alkalinity specifies weighing 2.5 ± 0.2 g of a standard to the nearest mg. This implies that the balance must have a range that includes 2.5 g, a capacity greater than 2.5 g, and a readability of 0.001 g (1 mg).

Example 2: When using a balance to calibrate the volume of a 200 µL pipette, the balance must be capable of weighing to the nearest 0.0001 g according to SOP DV-QA-0008, *Volumetric Verification*. In addition, the accuracy of the balance must be such that a 2% deviation in volume can be evaluated. In this specific case, assuming a density of 1.000 for water, 200 µL of water should weigh 0.200 g. The balance must be capable of detecting a 2% deviation in 0.200 g, or 0.004 g. This implies that the balance must have a range that includes 0.200 g, a capacity greater than 0.200 g, and a readability of 0.0001 g to meet the readability requirement specified in DV-QA-0008. A balance could have a readability of 0.001 g and still meet the accuracy requirement of 2%.

5.2 Balance Implementation

5.2.1 Install and set up a balance in accordance with the manufacturer's instructions.

5.2.2 Notify the QA group about the new balance in order to get the balance calibrated by the appropriate vendor (Section 6.1.4). Provide the balance make, serial number, readability and manufacturer's manual to QA.

NOTE: The balance **MUST NOT** be used until it has been calibrated by the appropriate balance service vendor and approved for service.

5.2.3 Request a new logbook from the QA group.

5.2.4 Perform an initial set of balance calibration verification measurements as described in Section 5.3.

5.2.5 Document the range of the balance in the balance calibration verification logbook. Analysts must confirm the verified range prior to use to ensure it is adequate for their mass needs. (See Attachments 1 and 2).

5.2.6 If any problems are encountered during set up, notify the QA group, who will consult with the balance service vendor to solve the problem.

5.2.7 Return the old logbook to QA for archiving.

5.3 Balance Calibration Verification

The verification is performed on a balance that is in service and under in-service conditions. Calibration verifications are comparable to ASTM "in-use testing" and are intended to indicate the quality of usual and customary mass determinations that are made or may be made when using the balance. Balance calibration verifications must be performed daily prior to first use of the balance.

5.3.1 Select a set of three standard masses that bracket the range of masses typically measured. This range **MUST** include the weight of the container/vessel containing what is being measured, even if this is done using the tare on the balance.

NOTE: **The range over which the balance is used is indicated in the logbook.**

5.3.2 Verify that the balance has a current service label and that the standard masses have a current calibration label.

5.3.3 If the balance service is not current, then do not use the balance. If the calibration of the standard masses is not current, then do not use the standard masses. Notify the Group Leader and the QA group.

5.3.4 For instructions on how to operate the balance, consult the owner's manual.

5.3.5 Verify the balance is level using the integrated bull's-eye level. Adjust the feet of the balance to level it if necessary. If a balance is moved the leveling must be checked before the balance calibration can be verified. Record the level check in the *Balance Level (Y/N)* column of the balance

calibration logbook (see attachments 1 and 2).

5.3.6 Remove the first standard mass from its box using either plastic tweezers or clean gloves, and place it on the balance pan.

NOTE: Never touch the standard mass without gloves. This may result in inaccurate readings due to skin oils being transferred to the standard mass.

5.3.6.1 For top-loading balances, allow the balance to stabilize and read the weight directly from the balance display.

5.3.6.2 For analytical balances, close all doors, allow the balance to stabilize, and read the weight directly from the balance display.

5.3.7 Document the weight reading in the applicable balance calibration verification logbook (see Attachments 1 and 2).

5.3.7.1 Record ALL the digits that are displayed.

5.3.7.2 To determine if the balance is in control consult the acceptance criteria table found either at the top of the logbook page (example given in Attachment 2) or in the front of the logbook (example given in Attachment 3).

5.3.7.3 The logbook shall contain the following information for each daily calibration verification:

- **Date,**
- **Weight Serial number,**
- **Certified value of each mass used,**
- **Balance reading, including ALL digits displayed,**
- **Initials of person performing the calibration check, and**
- **Balance level check (Yes/No)**
- **Indication of acceptability (Acceptable? Yes/No).**

5.3.8 Remove the standard mass from the balance using either plastic tweezers or clean gloves, and place it carefully back in its box.

5.3.9 If the balance fails the calibration check, and immediate corrective actions indicate that the balance is malfunctioning (see Section 5.6), then all affected samples, standards, and QC samples weighed since the last successful check must be repeated and the balance tagged as Out-of-Service as indicated in Section 5.6.3.3. Notify the QA department that the balance requires service and determine if a spare balance with the correct specifications is available for use. Note in the logbook the balance that was taken out of service, and record the serial number/ID of the new balance.

5.4 Acceptance Criteria for Analytical Balances

5.4.1 The acceptance criteria specified by revisions 4.2, 5.0 and 5.1 of the Department of Defense (DoD) Quality Systems Manual (QSM) have been implemented by TestAmerica Denver for all analytical balances.

5.4.2 The daily calibration verification must be ±0.1% or ±0.5 mg, whichever is larger, of the certified value of the standard mass measured for.

5.4.3 Control limits for typical standard masses weighed on an analytical balance are tabulated in Attachment 3.

5.5 Acceptance Criteria for Top-Loading Balances

5.5.1 The acceptance criteria specified in ASTM D 4753-02 have been implemented by TestAmerica Denver for all top-loading balances. These criteria are at least as stringent as the criteria specified by revisions 4.2, 5.0 and 5.1 of the Department of Defense (DoD) Quality Systems Manual (QSM).

5.5.2 The control limits depend on the readability of the balance used and are summarized below:

Top-Loading Balance Control Limits

Readability, g	Test Mass Range, g	Basic Tolerance
0.001	**< 2**	**± 0.002 g**
	≥ 2	**± 0.1 %**
0.01	**< 20**	**± 0.02 g**
	≥ 20	**± 0.1 %**
0.1	**< 200**	**± 0.2 g**
	≥ 200	**± 0.1 %**

5.5.3 Control limits for typical standard masses weighed on a top-loading balance are tabulated in Attachment 4.

5.6 Balance Problems and Corrective Action

5.6.1 If a balance does not behave normally or fails the calibration check, repeat the calibration verification measurements using all three standard masses.

5.6.2 If the balance fails twice, notify the Group Leader and/or the QA group, and document the problem in the logbook.

5.6.3 Corrective action, as described below, shall be performed by the Group Leader, a qualified analyst, or a member of the QA staff. Document all actions taken and results in the logbook.

5.6.3.1 Perform troubleshooting according to the owner's manual. Typically this includes checking the following:

- **balance has power, is plugged in or has fresh batteries,**
- **pan is free of obstructions,**
- **balance is level,**
- **table is free of vibrations,**
- **area is free of drafts or winds,**
- **area is free of extreme temperatures, and**
- **balance has warmed up sufficiently, if it was just turned on.**

5.6.3.2 To determine whether the balance problem is the result of a balance failure or environmental conditions, either the QA group or the Group Leader can opt to perform maintenance testing as described in ASTM D4753-02.

- **Place the balance in an environment that meets the manufacturer's recommendations, e.g., where vibration, air currents, and temperature extremes are minimized.**
- **Perform the balance calibration verification as described in Section 5.3.**
- **If the balance meets acceptance criteria under these conditions, then consider changing the environmental conditions under which the balance is normally used or try a different balance.**
- **If the balance fails the calibration checks under these conditions, then arrange to have the balance serviced by the approved balance service vendor. (See Section 6.1.4)**

5.6.3.3 If the balance is not restored to proper operation immediately, then post an Out-of-Service form on the balance to warn other people that it cannot be used. Also note in the logbook that the balance was removed from service.

5.6.3.4 Inform the Group Leader and/or the QA group of the repeated failure. The Group Leader and/or the QA group will determine if the balance will need to be serviced or replaced.

5.6.3.5 Following service, perform the required calibration verification as described in Section 5.3. If the calibration verification is successful, then note the return to service in the logbook.

NOTE: **The balance cannot be put back in service until the calibration check is successfully performed.**

5.6.3.6 If the balance has been replaced, document on the cover and at first use in the logbook the date the balance ID changed or request a new logbook unique to the new balance.

5.7 QA Department Weights Calibration Verification

5.7.1 The QA Department Weights used in the laboratory to perform the verification of the Lab Working Weights are recertified every 5 years by a third party. Physical records of the certifications are maintained in the QA Group. Certification dates are tracked in the Weight List spreadsheet found at \\tacorp\Corp\QA\QA_Facilities\Denver-QA\Equipment\Support-Equipment\Weights (see Attachment 6).

5.7.2 All weight sets used to perform the calibration verification as described in Section 5.3 are also recertified by a third party calibration service every 5 years in compliance with Wisconsin regulation NR 149.44 (3) (g) (2).

5.7.3 The laboratory inspects any and all weights that were outside of laboratory control upon receipt for any visual damage.

5.7.4 As good laboratory practice, the laboratory performs a comparison between the weights received and a set of weights previously calibrated on an annual basis to confirm the masses received were not damaged in transit. The comparison will be performed as the annual verification (see section 5.8.2) and stored at \\tacorp\Corp\QA\QA_Facilities\Denver-QA\Equipment\Support-Equipment\Weights\Annual Weight Verification.

5.7.5 Once verified, the QA Department Weights are considered valid for 5 years and are used only to verify the Lab Working Weights on an annual basis.

NOTE: Currently the approved vendor for recertifying ASTM Class 1 weights is Quality Control Services, 2340 SE 11th Avenue, Portland, OR 97214.

5.8 Lab Working Weight Calibration Verification

5.8.1 The Lab Working Weights that are used to perform the daily verification of balances are verified on an annual basis (± one month) against the QA Department Weights maintained in the QA department. They are also recertified by a third party calibration service every 5 years (see Section 5.7). Certification dates are tracked in the Weight List spreadsheet found at \\tacorp\Corp\QA\QA_Facilities\Denver-QA\Equipment\Support-Equipment\Weights (see Attachment 6).

5.8.2 Annual Verification

The criteria listed in the Annual Weight Comparison Template are recommended criteria based on the criteria for the daily balance calibration check specified in the DoD QSM. The QA Manager has the discretion to determine if the results of the comparison check are sufficient to meet laboratory needs and requirements. The annual verification is performed on a balance that is in service and under in-service conditions (see Section 5.3 for details). The verification process is as follows:

5.8.2.1 Use an analytical balance for weight measurements less than or equal to 10 g and a top loading balance for measurements greater than 10 g.

5.8.2.2 Tare the balance then place the weight from the lab set on the plate. Record the output of the weight measurement from the balance to the same number of decimal places displayed on the balance.

5.8.2.3 Repeat with the corresponding weight from the QA set.

5.8.2.4 Enter the results into the Annual Weight Comparison Template (see Attachment 5) and evaluate against the criteria.

5.8.2.5 If the difference in weights between sets is within the recommended criteria, "Pass" will appear in the evaluation column.

5.8.2.6 Place a calibration label on the weight set noting date performed and date due (1 year from date performed).

5.8.2.7 If criteria are not met, notify the QA Manager. The QAM or designee will determine an appropriate course of action and record the action in the record.

5.8.3 The criteria listed in the Annual Weight Comparison Template are recommended criteria based on the criteria for the daily balance calibration check specified in the DoD QSM. The QA Manager has the discretion to determine if the results of the comparison check are sufficient to meet laboratory needs and requirements.

5.8.4 All observations must be recorded at time of measurement. Observations may be recorded electronically if the balance is attached to a computer. Alternatively, print a copy of the Annual Weight Comparison Template form and record the original observations, balance ID, analyst and date performed. Transcribe this information into the online template to calculate the differences and the evaluations. When data entry is complete, print this form and attach it to the printed copy of original observations. If manual entry was performed, review of data entry must be performed by a second member of the QA team and documented on the form. File the record by month and year in the folder \\tacorp\Corp\QA\QA_Facilities\Denver-QA\Equipment\Support-Equipment\Weights\Annual Weight Verification.

6.0 RESPONSIBILITIES

6.1 QA Department

6.1.1 The QA Manager and QA staff are responsible for maintaining the standard weights that are used for daily calibration check on each balance and the NIST calibration weights.

6.1.2 The QA staff ensures that each set of Class 1 (ASTM) and Class S (NIST) standard masses used to check the accuracy of a balance is verified annually and recertified every 5 years as described in Section 5.8.

6.1.3 The QA staff ensures that each set of NIST certified Class 1 (ASTM) and Class S (NIST) standard masses used to annually verify the laboratory working calibration weights is recertified at a minimum of every five years by an approved vendor.

NOTE: **The currently approved vendor for providing and recertifying standard masses is Quality Control Services, Portland, Oregon.**

6.1.4 The QA staff are responsible for maintaining physical records of certification for all QA and lab working weights. Records of certification are scanned upon receipt to \\tacorp\Corp\QA\QA_Facilities\Denver-QA\Equipment\Support-Equipment\Weights\Weight Certification Records.

6.1.5 The QA group is responsible for ensuring that each active balance is serviced annually by an approved balance service vendor, and that the vendor attaches a label to each balance indicating current service.

NOTE: **The currently approved vendor for servicing balances is QA Balance Services, Inc., Aurora, Colorado.**

6.1.6 The QA group may also perform additional maintenance testing of a balance that fails daily calibration verification checks to determine whether service is warranted.

6.1.7 The QA group is responsible for verifying weights that have been sent out for calibration are in acceptable working order. This is done by performing a balance verification as stated in Section 5.7 upon receipt of the weights from the vendor before the weights are placed into service.

6.2 Group Leaders

6.2.1 Group Leaders are responsible for selecting the appropriate balance, in terms of capacity, readability, and accuracy, for each analytical application.

6.2.2 Each Group Leader is responsible for supervising the analysts who perform the daily balance calibration checks and ensuring the checks are performed prior to use of the balance each day. Each Group Leader shall review, initial, and date the balance logbooks at least bimonthly.

6.2.3 Group Leaders and analysts are responsible for notifying the QA group whenever any of the following occurs:

- A balance is moved to a different location or group in the laboratory,
- A balance is no longer operating correctly and needs service,
- A balance is taken out of service, or
- Standard weights are moved to a different location or group in the laboratory other than the normal sharing of weights between groups.
- An internal calibration is required.

NOTE: If a balance contains a Staticmaster ® the RSO must also be notified if the balance is moved, taken out of service or sent out for service.

6.3 Analysts

6.3.1 Under the direction of the Group Leader, analysts perform the daily balance calibration checks. Analysts shall report any balance problems to the Group Leader and the QA group. Analysts shall record daily calibration verification checks in the applicable logbook. The Group Leader shall ensure that balance problems, trouble shooting, corrective actions, and return to service are recorded in balance logbooks, as appropriate.

6.3.2 Each analyst who uses a balance must ensure that a daily calibration check has been performed that day, either by performing and recording the check, or by verifying that the check has already been performed and recorded for that day. A Group Leader or someone from the QA group must be notified if this check is not being performed as required. A balance shall not be used if the daily check has not been performed.

6.3.3 When additional calibration checks are prescribed by a specific method or program (e.g., a closing calibration check following all sample measurements), then the analyst shall ensure that the required checks are performed as prescribed.

6.3.4 Analysts and group leaders are responsible for properly using balances, keeping the balance pan clean, keeping standard masses clean, and protecting standard masses from damage.

7.0 REFERENCES / CROSS-REFERENCES

7.1 ASTM D4753-02, *Standard Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing*.

7.2 Wisconsin Administrative Code s. 35.93, NR 149.44 (3) (g) (2)

8.0 ATTACHMENTS

Attachment 1: Analytical Balance Calibration Verification Log
Attachment 2: Top-Loading Balance Calibration Verification Log
Attachment 3: Analytical Balance Control Limits
Attachment 4: Top-Loading Balance Control Limits
Attachment 5: Annual Weight Comparison Template
Attachment 6: Weight List (Example)

9.0 REVISION HISTORY

Revision 15, 30 June 2019
- Annual Review
- Updated copy right information

Revision 14, 30 June 2018
- Annual Review
- Added balance ID on each logbook page requirement to section 4.3

Revision 13, 30 June 2017
- Annual Review
- Added clarification to section 3.4, 5.3 and 6.2.2 regarding daily balance calibration verification requirements.
- Added clarification to section 5.3.1 regarding including tare weight when bracketing range of masses.
- Added balance level check logbook requirements to section 5.3.7.3.
- Updated section 5.3.9 to notify QA when balance is out of service.
- Added section 5.6.3.6 concerning logbook requirements for balance replacements.
- Added data entry review in section 5.8.4.
- Added weight certification record documentation requirements to section 6.1.4.
- Added notification requirements in the event an internal calibration is required to section 6.2.3.
- Edited electronic file paths throughout.

Revision 12, 31 August 2016
- Added clarification to Section 5.8.2.1 regarding which balances to use for annual verifications
- Added requirement to record balance level check in logbook to Section 5.3.5
- Updated Attachments 1-3 to match current logbook setup

Revision 11, 31 October 2015
- Added requirement to Section 4.2.3 that all weight sets need to be calibrated every five years by a third party
- Added same requirement to Sections 5.8.1 and 6.1.2
- Added Section 3.7 regarding definitions of analytical terms
- Deleted Sections 3.5 and 3.6, unused definitions
- Added definitions of different weight sets to Section 3
- Changed name of NIST weights to QA department weights
- Added Section 5.3.5 regarding leveling the balance
- Corrected language in Section 5.8.4
- Deleted Section 5.7.5 as it does not apply
- Deleted Section 5.7.6 as it does not apply
- Updated Attachment 5
- Added Attachment 6
- Added Section 5.2.7 requiring return of logbooks to QA
- Minor grammar and formatting changes throughout

Revision 10, 30 June 2015
- Added section 5.7.2 weight sets used in calibration need to be calibrated every five years by a third party

Revision 9, dated 31 August 2014
- Annual Review
- Updated/corrected all section references
- Added references to working weights and NIST weights
- Updated section 5.2.2 required information

- Deleted previous section 5.3.5
- Added sections 5.7 and 5.8
- Added section 7
- Added attachment 5

Revision 8, dated 31 August 2013
- Annual Review
- Switched sections 4 and 5 to be in the appropriate order
- Added note to section 5.2.2
- Added statement concerning the need to include any containers/vessel to the weight range of the balance to section 5.3.1
- Updated section 5.4.1 to reflect versions 4.2 and 5 of the DoD QSM.
- Added DOD QSM reference to section 5.5.1
- Revised section 6 for clarity
- Updated section 6.2.2 to reflect bi-monthly reviews
- Added note to section 6.2.3 to notify RSO if balance with Staticbuster® is moved
- Updated Attachments 1-4

Revision 7.4, dated 31 August 2012
- Annual Review

Revision 7.3, dated 31 August 2011
- Annual Review
- Corrected various formatting and grammatical errors.
- Added a new section 4.2.2.

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Analytical Balance Calibration Verification Log (Example)

ANALYTICAL BALANCE CALIBRATION LOG
SOP NUMBER: DV-QA-0014 **TestAmerica Denver**

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Balance ID: 24850570

Balances must be level before calibrating. Check the levelling bubble attached to the balance and adjust with the levelling screws if necessary before proceeding.

Date	Weight Serial Number	Weight Used	Balance Reading (g)	Initals	Balance Level (Y/N)	Acceptable? (Y/N) Comments

Attachment 2.

Top-Loading Balance Calibration Verification Log (Example)

Top-Loading Balance Calibration Verification Log

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

DV-QA-0014 TestAmerica Denver

Balance ID: 24750836

ACCEPTANCE CRITERIA:	**0.001 READABILITY:**	For <2 g, true mass ± 0.002 g: for ≥ 2 g, true mass ± 0.1%
	0.01 READABILITY:	For <20 g, true mass ± 0.02 g: for ≥ 20 g, true mass ± 0.1%
	0.1 READABILITY:	For <200 g, true mass ± 0.2 g: for ≥ 200 g, true mass ± 0.1%

Balances must be level before calibrating. Check the levelling bubble attached to the balance and adjust with the levelling screws if necessary before proceeding.

Date	Weight Serial Number	Weight Used	Balance Reading (g)	Initials	Balance Level (Y/N)	Acceptable? (Y/N)/Comments

Attachment 3.

Analytical Balance Control Limits

TestAmerica Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Analytical Balance Control Limits

DV-QA-0014

Results of the daily balance check must be within 0.1% or 0.5 mg of the expected value, whichever is larger.

Weight Used (grams)	0.1% of Weight (mg)	0.1% of Weight (grams)	0.1% Lower Limit* (grams)	0.5 mg Lower Limit* (grams)	0.1% Upper Limit* (grams)	0.5 mg Upper Limit* (grams)
0.002	0.002	0.000002		0.00195		0.00205
0.010	0.01	0.00001		0.0095		0.0105
0.020	0.02	0.00002		0.0195		0.0205
0.050	0.05	0.00005		0.0495		0.0505
0.10	0.10	0.00010		0.0995		0.1005
0.20	0.20	0.00020		0.1995		0.2005
0.50	0.50	0.00050	0.4995	0.4995	0.5005	0.5005
1.0	1.00	0.0010	0.999		1.001	
2.0	2.00	0.0020	1.998		2.002	
5.0	5.00	0.0050	4.995		5.005	
10.0	10.00	0.010	9.99		10.01	
20.0	20.00	0.020	19.98		20.02	
50.0	50.00	0.050	49.95		50.05	
100.0	100.00	0.10	99.9		100.1	
200.0	200.00	0.20	199.8		200.2	
500.0	500.00	0.50	499.5		500.5	
1000	1000.00	1.00	999		1001	

Balance Capability Needed to Meet Control Limits

Weight Range	Balance Reading	Balance Sensitivity (gram)
0.01 to 0.1	4 places	0.0001
0.1 to 1	4 places	0.0001
1 to 10	3 places	0.001
10 to 100	2 places	0.01
100 to 1000	1 place	0.1

Attachment 4.

Top-Loading Balance Control Limits

TestAmerica Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Top-Loading Balance Control Limits

DV-QA-0014

Readability = 0.001 g (3 decimal places in the display)					
Weight Used (gram)	0.1% of Weight (gram)	0.1% Lower Limit (gram)	0.002 g Lower Limit (gram)	0.1% Upper Limit (gram)	0.002 g Upper Limit (gram)
0.002	0.000002		0.00018		0.004
0.010	0.00001		0.008		0.012
0.020	0.00002		0.018		0.022
0.050	0.00005		0.048		0.052
0.10	0.0001		0.098		0.102
0.20	0.0002		0.198		0.202
0.50	0.0005		0.498		0.502
1.0	0.001		0.998		1.002
2.0	0.002	1.998	1.998	2.002	2.002
5.0	0.005	4.995		5.005	
10.0	0.010	9.990		10.010	
20.0	0.020	19.980		20.020	
50.0	0.050	49.950		50.050	
100.0	0.100	99.900		100.100	
200.0	0.200	199.800		200.200	
500.0	0.500	499.500		500.500	
1000	1.000	999.000		1001.000	
2000	2.000	1998.000		2002.000	

Readability = 0.01 g (2 decimal places in the display)					
Weight Used (gram)	0.1% of Weight (gram)	0.1% Lower Limit (gram)	0.002 g Lower Limit (gram)	0.1% Upper Limit (gram)	0.002 g Upper Limit (gram)
0.01	0.00001		-0.01		0.03
0.02	0.00002		0.00		0.04
0.05	0.00005		0.03		0.07
0.10	0.0001		0.08		0.12
0.20	0.0002		0.18		0.22
0.50	0.0005		0.48		0.52
1.0	0.001		0.98		1.02
2.0	0.002		1.98		2.02
5.0	0.005		4.98		5.02
10.0	0.01		9.98		10.02
20.0	0.02	19.980	19.980	20.02	20.02
50.0	0.05	49.950		50.05	
100.0	0.10	99.900		100.10	
200.0	0.20	199.800		200.20	
500.0	0.50	499.500		500.50	
1000	1.00	999.000		1001.00	
2000	2.00	1998.000		2002.00	

Readability = 0.1 g (1 decimal places in the display)					
Weight Used (gram)	0.1% of Weight (gram)	0.1% Lower Limit (gram)	0.002 g Lower Limit (gram)	0.1% Upper Limit (gram)	0.002 g Upper Limit (gram)
0.1	0.0001		-0.01		0.3
0.2	0.0002		0.0		0.4
0.5	0.0005		0.3		0.7
1.0	0.001		0.8		1.2
2.0	0.002		1.8		2.2
5.0	0.005		4.8		5.2
10.0	0.01		9.8		10.2
20.0	0.02		19.8		20.2
50.0	0.05		49.8		50.2
100.0	0.10		99.8		100.2
200.0	0.20	199.8	199.8	200.2	200.2
500.0	0.50	499.5		500.5	
1000.0	1.00	999.0		1001.0	
2000.0	2.00	1998.0		2002.0	

Attachment 5.

Annual Weight Comparison Template

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Record of Annual Comparison Check between Lab Working Weight Set and QA Department Weight Set

Analyst	Date	Lab Set	QA Set
		Set 1	Set 2

Traceability records for the weight sets are maintained in the QA department.

		Observations					
Weight Mass	Conversion (g)	Balance ID	Lab Set (g)	QA Set (g)	Difference (g)	Criteria (g)	Evaluation
2 mg	0.0020				0.0000	0.0005	Pass
10 mg	0.0100				0.0000	0.0005	Pass
20 mg	0.0200				0.0000	0.0005	Pass
100 mg	0.1000				0.0000	0.0005	Pass
1 g	NA				0.0000	0.0010	Pass
2 g	NA				0.000	0.002	Pass
5 g	NA				0.000	0.005	Pass
10 g	NA				0.000	0.010	Pass
20 g	NA				0.00	0.02	Pass
50 g	NA				0.00	0.05	Pass
100 g	NA				0.00	0.10	Pass
200 g	NA				0.0	0.2	Pass
500 g	NA				0.0	0.5	Pass
1 Kg	1000				0.0	1.0	Pass
2 Kg	2000				0.0	2.0	Pass
3 Kg	3000				0.0	3.0	Pass

Work Instruction:

Use an analytical balance for weight measurements < 1 g and a top loading balance for measurements greater than 1 g. Tare the balance then place the weight from the lab set on the plate. Record the output of the weight measurement from the balance to the same number of decimal places displayed on the screen. Repeat with the corresponding weight from the QA set. Enter the results into this workbook and evaluate against the criteria. If the difference in weights between sets is within the recommended criteria, enter "Pass" in the evaluation column. Place a calibration label on the weight set noting date performed and date due (1 year from date performed). If criteria are not met, notify the QA Manager. The QAM will determine an appropriate course of action and record the action in the record.

Criteria:

The criteria listed in this workbook is recommended criteria based on the criteria for the daily balance calibration check specified in the DoD QSM. The QA Manager has the discretion to determine if the results of the monthly comparison check are sufficient to meet laboratory needs and requirements.

Frequency of Check:

The standard weights used in the laboratory to perform the balance calibration check are recertified every 5 years by a third party. As good lab practice, the laboratory performs a comparison between the weights used daily and a set of weights kept in QA on an annual basis to confirm the weight set used daily is adequate for use between recertification events.

Record Keeping:

All observations must be recorded at time of measurement. Observations may be recorded electronically if the balance is attached to a compouter. Alternatively, print a copy of this form and record the original observations, balance ID, analyst and date performed. Transcribe this information into this workbook to calculate the difference and enter the evaluation. When data entry is complete, print this form and attach it to the printed copy of original observations. File the record, however generated, using the system established such that the record is readily available for review.

Attachment 6.

Weight List (Example)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Sub-out Work Sample Management and Chain of Custody

Approvals (Signature/Date):

Alyssa Gomez 10.4.18
Alyssa Gomez — Date
Sample Receiving Supervisor

Doug Gomer 10/4/18
Doug Gomer — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 10/4/18
Roxanne K. Sullivan — Date
Quality Assurance Manager

FOR

10/5/18
Richard Clinkscales — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 PURPOSE

1.1 This procedure describes the procedure for the packaging and shipping of samples to sub-contract laboratories. The samples must be shipped in a manner that is consistent with preservation requirements and prevents contamination and breakage. They must be shipped in a manner consistent with the Federal Department of Transportation (DOT) requirements.

2.0 SCOPE

2.1 TestAmerica Denver has one sample shipment area. Both radiological and non-radiological samples may be shipped from this area. Personnel working in the Sample Control area are required to complete radiation safety training, as described in the Radiation Safety Manual (RSM), before handling radioactive samples. See SOP DV-RS-0001, *Purchase, Receipt, Handling and Identification of Radioactive Material*, for the procedures specific to incoming radioactive samples.

2.2 This procedure describes the Chain-of-Custody requirements from receipt of samples via a Chain of Custody form until samples are packaged and sent in coolers to the respective sub-contract laboratory.

2.3 The procedure is summarized in Work Instruction WI-DV-0084, *Sample Sub-out Checks*. FedEx System instructions listed in Attachment 1 are included in the work instruction.

3.0 SAFETY

3.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual and the Radiation Safety Manual.

3.2 Specific Safety Concerns

Most of the chemical preservatives discussed in the next section are corrosive to human skin, eyes, and other tissues. Because the laboratory purchases bottles with small amounts of preservative chemicals already added, there is little potential for significant spills. However, it is important to handle open or broken bottles that contain preservatives with great caution, including the following:

3.2.1 Use the appropriate personal protection equipment (i.e., nitrile or latex gloves, safety glasses with side shields, and lab coat).

3.2.2 Use the appropriate spill containment and absorbent materials.

3.2.3 Contact the Waste Coordinator if unsure of how to clean up or dispose of materials from reagent spills.

3.2.4 When working with broken glass cut resistant gloves must be worn.

3.3 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	Hazards	Exposure Limit[(2)]	Signs and Symptoms of Exposure
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Sodium Bisulfate	Corrosive Oxidizer Dehydrator Poison	N/A	Forms sulfuric acid when mixed with water. See symptoms under sulfuric acid, below.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of sodium hydroxide dust will cause irritation of the nasal and respiratory system.
Mono-chloroacetic Acid	Inhalation Hazard Corrosive	0.3 ppm (TWA) 1.0 ppm (STEL)	Extremely destructive to mucous tissues and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Skin contact can result in redness, pain, and severe burn.

Material [1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Nitric Acid Solution, <20%	Corrosive Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Skin contact can result redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 DEFINITIONS

4.1 Chain-of-Custody Form: A critical legal document, which records collection, possession, and transfer of samples from one person or party to another.

4.2 Cooler Seals: Seals used to demonstrate that custody has been maintained and that unauthorized personnel have not had access to the samples.

4.3 Temp Blank: A blank that accompanies the bottles and samples that is used to test the cooler temperature at time of receipt.

4.4 Materials of Trade: Per 49 CFR 173.6, the samples that are transported from the field to the laboratory for analysis are classified as a "Material of Trade." Under the provisions of 49 CFR 173.6, "Materials of Trade" are not subject to the provisions of the hazardous materials shipping regulations as long as the following provisions are met:

4.4.1 The material is transported by TestAmerica employees, the client's employees, or private courier hired by TestAmerica or the client.

4.4.2 The total gross aggregate weight of the sample package does not exceed the limits set forth in the citation. Individuals need to check the regulatory citation since the total mass varies by shipping class and packing group.

4.4.3 The total gross aggregate weight of all packages containing known hazardous materials does not exceed 440 pounds.

4.4.4 The materials are packaged in accordance with the citation. Packaging for each classification of material may vary slightly. However, in general the packages must be leak tight for liquids and gases, sift proof for solids, be securely closed, secured against movement, and protected against damage.

4.4.5 The outer packages must be marked with either a common name or a proper shipping name.

NOTE: This citation does not apply to explosive (Class 1) or radioactive (Class 7) materials.

5.0 PROCEDURE

5.1 TestAmerica has a list of approved laboratories that may be used for testing not performed by this lab (the approval process is described in SOP No. CA-L-S-002, "Subcontracting Procedures"). The following procedures also apply to sending work to other TestAmerica labs.

5.2 The PM indicates any subcontract lab arrangements in the project.

5.3 Samples awaiting shipment should be kept refrigerated. Most samples can be placed in the main walk-in once logged; however samples for Volatile Organic Analysis should be placed in the Volatile area satellite refrigerator or in a cooler with ice to prevent contamination.

5.4 Items to verify as the samples are prepared for shipment are summarized in WI-DV-0084.

5.5 A subcontract COC is generated from TALS and must accompany the samples sent out to other labs. This COC must clearly indicate sample identities and the tests that must be performed. If known hazardous or radioactive materials are being shipped, the samples must be labeled per DOT regulations and the information included with the COC. If the samples have a USDA label or quarantine sample label, the receiving laboratory of the shipment must also have a soil import permit. Samples cannot be shipped until the receiving lab provides a copy of their permit and the required USDA label.

5.6 Once the PM group provides the sub-out COC; samples are pulled and checked against the COC.

5.6.1 Verify that the number of sample containers match the number listed on the sub-out COC.

5.6.2 Verify that the sample IDs match the sample IDs on the sub-out COC.

5.7 Packing the Cooler

Ensure that the bottles are wrapped and packaged with sufficient packing material to prevent breakage. If the bottle order does not completely fill the cooler, add extra packing material to fill any unused space and prevent bottle movement during shipping. Pack each cooler as follows:

5.7.1 Place absorbent material (e.g., Power-Sorb Pads or equivalent) in the cooler to contain a spill if breakage should occur. There must be enough absorbing material to absorb all the preservative in all the sample bottles in the cooler.

5.7.2 Place a layer of foam packing material or bubble wrap on top of the pads. Use enough material to completely cover the bottom and sides of the cooler, with enough extra to cover the top after the cooler is filled.

5.7.3 Place a plastic cooler liner in the cooler.

5.7.4 Place the individual bottles, VOA vials, or jars in appropriately sized bubble bags as necessary. All glass containers require bubble bags, plastic containers do not unless specifically requested.

5.7.5 Place the bottles in Ziploc bags.

Note: All bottles (plastic and glass) need to be in Ziploc bags. Make sure that the Ziploc bags are sealed correctly.

5.7.6 Place the bagged sample containers into the lined cooler. Once all of the bottles are in place, tie the open end of the liner into a knot over the top of the bottles.

5.7.7 Cover the liner with an extra layer of foam and add extra foam as needed to prevent sample container movement during shipping.

5.8 Sub-out Sample Cooler Thermal Preservation

All samples aside from those logged solely for Metals water analysis require thermal preservation. Loose ice cubes or dry ice will be used as appropriate to provide cooling during sample transit. Ice packs or “Blue Ice” shall not be used.

5.8.1 Ice

After the bagged samples have been added to the cooler following the instructions in Sections 5.7.6, loose ice is added to cover the samples before tying off the cooler liner. Ice is added to the sample cooler in proportion to the cooler size, with anywhere from ½ gallon to 4 gallons of ice being added. Make sure the lid of the cooler is still able to close completely.

5.8.2 Dry Ice

In instances where samples must be shipped offsite but must remain frozen during shipment (e.g., DI water preserved soils for volatile organic analysis) dry ice must be used to keep the sample containers frozen. Dry ice is added in a similar fashion to water ice, however, in order to prevent the build up of pressure in the cooler from sublimation, the cooler must be vented. Venting is accomplished by using a drill to make a hole in the lid of the cooler. Make sure the hole is clear of tape or labels before shipping. Also, a dry ice DOT label must be affixed to the top of the cooler before it

can be shipped.

5.9 The individual packing the cooler signs the COC to relinquish the samples.

5.10 A temperature blank should be included in all coolers sent with subcontracted work. The signed subcontract COC is included in the cooler and a custody seal is affixed to the cooler along with any other shipping markings as required.

5.11 The FedEx shipping label is generated (see Attachment 1 or WI-DV-0084). The address on the shipping label must match the address on the copy of the COC. Saturday delivery must be selected and Saturday delivery labels must be placed on the cooler when shipped on Friday. The temperature control label is placed on or near the FedEx tag with "Refrigerate" marked (iced samples only).

5.12 When the samples have been shipped, a copy of the COC and the tracking number for the shipment is forwarded to the PM. The PM's project file will include records of any shipments of hazardous or radioactive materials. A second copy of the COC is retained in the Sample Receiving area for reference. Copies of forms documenting shipments of radioactive materials will be sent to the RSO.

6.0 ATTACHMENTS

Attachment 1: FedEx system instructions

7.0 REVISION HISTORY

Revision 5, dated 10/05/2018
- Annual Review

Revision 4, dated 9/30/2017
- Annual Review
- Added note to Section 5.7.5 & updated WI-DV-0084 accordingly

Revision 3, dated 9/30/2016
- Corrected Work Instruction reference in Section 2.3
- Added detail to Section 5.6 to emphasize verification that shipment is correct and complete
- Revised Section 5.10 that only samples that require thermal preservation must be iced
- Added use of FedEx temperature control label to Section 5.13
- Added instruction in Attachment 1 to enter Department Code into FedEx System (new Section 7, renumbered remainder of section)
- Revised WI-DV-0084 to reflect changes in this SOP
- Annual review

Revision 2, dated 9/30/2015
- Annual review.

Revision 1, dated 9/30/2014
- Removed Attachment 1 and converted contents to Work Instruction WI-DV-0084, Sample Sub-Out Checks.
- Added new Section 5.4 to reference WI-DV-0084; renumbered remaining sections.

Revision 0, dated 1/31/2014

- Initial Implementation

Attachment 1

FedEx System Instructions

1. Enter the name of the laboratory in the Receipt ID Field. Press tab
2. If the address does not populate enter the address manually
3. If sending multiple coolers to the same lab enter the number of coolers in the number of packages filed (need this in master tracking number)
4. Service type – std overnight; except on Friday – priority overnight
    - At this point if priority over night is chosen on Friday the box for Saturday delivery needs to be approved. Must check this box. Affix Saturday delivery labels to cooler.
5. Package Type – your packing
6. Press override PREFs (at bottom of screen)
7. Enter Department Code (280320) in the Reference section
8. Change bill from sender to receiving.
    - If account number field is populated proceed
    - If account number field is not populated – change back to sender if we do not have the receiving account number
9. Press ship to generate the shipping label.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Soil and Waste pH [SW9045C & SW9045D]

Approvals (Signature/Date):

Andrew Allen — 8/17/18 — Date
Technical Specialist

Connie Jewell — 8/17/18 — Date
Department Supervisor

Doug Gomer — 8/17/18 — Date
Health & Safety Manager/Coordinator

Roxanne Sullivan — 8/17/18 — Date
Quality Assurance Manager

Richard Clinkscales — 8/17/18 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This is an electrometric procedure for measuring pH in solid samples. This method is applicable to the analysis of soils, sediments, sludges, or non-aqueous liquids. It does not apply to multiphase wastes where the aqueous phase constitutes more than 20% of the sample. See DV-WC-0031 for the determination of pH in multiphase wastes

1.2 A detection limit (MDL) for pH has not been defined, however, for reporting purposes this laboratory uses 0.1 pH units as the RL and MDL.

1.3 This method is applicable to all ranges of pH.

2.0 Summary of Method

The sample is mixed with reagent water. The pH meter, glass electrode, and reference electrode (or single combination electrode) are standardized against five reference buffer solutions of known pH bracketing the pH expected to be found in the sample. The sample measurement is made by immersing the electrodes into the sample solution and taking a reading from the meter.

3.0 Definitions

3.1 **pH** - At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Because of ionic interactions in all but very dilute solutions, it is necessary to use the “activity” of an ion and not its molar concentration. The use of the term pH assumes that the activity of the hydrogen ion is being considered. The approximate *equivalence* to molarity can be presumed only in very dilute solutions. A logarithmic scale is used to accommodate the wide range of ionic activities.

3.2 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 The pH response of most glass electrodes is imperfect at both ends of the scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of salts and strong acids, which have a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of the proper electrodes for these conditions. For example, sodium may interfere at pH > 10, and is controlled by using a “low sodium error” electrode.

4.2 Temperature fluctuations will cause measurement errors.

4.3 Coatings of oil and particulate matter may impair electrode response.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1 (as per the Corporate Environmental Health and Safety Manual), laboratory coat, and nitrile or latex gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 pH Meter with temperature compensation ability. Details of the pH meter and electrode currently in use can be found at R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision).

6.1.2 Glass electrode with reference electrode. A calomel, silver-silver chloride or other reference electrode of constant potential may be used; or use a combination electrode that incorporates both measuring and reference functions.

6.1.3 Analytical balance capable of weighing to the nearest 0.1 gram. The balance is checked for accuracy each day it is used in accordance with SOP DV-QA-0014.

6.1.4 Shaker table.

6.2 Supplies

6.2.1 40 dram vials with snap cap or a container large enough to hold sample and cover electrodes.

6.2.2 Glass wool, if oily wastes are to be tested.

6.2.3 50 mL graduated cylinder

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 **Reagent water**: Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

7.3 **pH Buffers: 2, 4, 7, 10 and 12:** Use commercially available solutions that have been validated by comparison to NIST standards. The solution aliquots used to calibrate the pH meter must be replenished each day of use.

7.4 **ICV Buffer Solution:** A pH 7 buffer solution from a second source provider, obtained commercially and traceable to NIST standards. The ICV solution aliquot used must be replenished each day of use.

7.5 **Laboratory Control Sample (LCS) Solution:** The LCS solution must be certified for pH and is commercially available. The pH 7 buffer from Section 7.3 is normally used as the LCS. Due to the nature of pH determination, a solid matrix is not used.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation [1]	Holding Time [2,3]	Reference
Solid	4 oz. glass or plastic	50 g	Cool, ≤ 6 °C	None	SW-846

[1] 40 CFR Part 136.3 and SW-846 indicate no preservation is required for pH. It is intended by both programs that the samples be analyzed in the field. TestAmerica Denver typically refrigerates these samples because the aliquot tested is taken from a sample container that is used for other tests that do require refrigeration.

[2] pH is intended to be a field measurement and samples are to be analyzed immediately per SW-846. The laboratory attempts to measure pH as soon as possible upon receipt. All laboratory analyzed samples are flagged as out of hold.

[3] Samples **must** be analyzed the same day that the extraction is performed.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 or 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC - The following quality control samples are prepared with each batch of samples.

9.2.1 Laboratory Control Sample (LCS/LCSD)

One LCS/LCSD is required with each batch of samples processed, not to exceed 20 samples. See Section 7.5.

Acceptance Criteria: The LCS must be within ± 0.05 pH units of the true value.

Corrective Action: If the LCS is not within the control limits, rerun all associated samples.

9.2.2 Duplicate Samples

One duplicate sample must be analyzed with each batch of samples processed not to exceed 20 samples.

Acceptance Criteria: The two results should agree within ± 0.10 pH units.

Corrective Action: If the difference is greater than ± 0.10 repeat the analysis. If the difference still exceeds the control limit the data will be flagged as outside of the limit.

9.2.3 Method blanks and matrix spikes are not applicable to pH.

9.3 Instrument QC

9.3.1 Initial Calibration Verification

Record the expected pH, manufacturer, and lot number of the verification buffer used for a second source pH 7.0 buffer solution. Analyze the second source pH 7 buffer solution.

Acceptance Criteria: The second source ICV buffer solution should read within ± 0.05 pH units of the true value.

Corrective Action: If this criterion is not met, the problem should be identified, corrected, and the meter recalibrated.

9.3.2 Continuing Calibration Verification

A pH 7.0 buffer check is required after every 10 or fewer samples and at the end of the run. The CCV is the same pH 7 buffer solution used in the initial calibration.

Acceptance Criteria: The pH buffer checks must be within ± 0.05 units of the true value.

Corrective Action: If the pH 7.0 buffer check is outside of the control limits, rerun all samples since the last acceptable pH 7.0 buffer check.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective described.

10.3 Sample Preparation

10.3.1 Weigh a minimum of 40 g of sample into a 40 dram vial, add 40 mL reagent water using a graduated cylinder and cap the vial. A sample which is not a soil but another material may possibly react violently on the addition of water. In such cases, add the water to the solid in a hood. If the sample shows any signs of heat or gas evolution, do not cap the vial as pressure may build up

10.3.1.1 If the samples are oily, filter through glass wool to remove oil. Retain the aqueous phase for analysis.

10.3.1.2 If the sample is hygroscopic and absorbs all the reagent water, add an additional 20 mL of reagent water to the extraction vessel and mix to incorporate. Note the increased water volume on the benchsheet.

10.3.1.3 If the sample is a waste and additional water is needed, add an additional 40 mL of reagent water.

10.3.2 Mix on shaker table for 5 minutes.

10.3.3 Let the sample settle undisturbed for a minimum of one hour.

10.4 Calibration

10.4.1 Follow the operating instructions supplied by the manufacturer of the pH meter. See WI-DV-0034 for more information concerning meter calibration.

10.4.2 Record the instrument ID, pH probe ID, thermometer probe ID, and reagent IDs in the batch record in TALS.

10.4.3 When using the Thermo Five Star pH meter, all results are to be temperature corrected to 25 °C using the Automatic Temperature Compensation function available with the instrument.

NOTE: Methods 9045C and 9045D state: "The sample temperatures must be within ± 2 °C of the calibrated buffers or temperature corrected." All samples are automatically corrected for temperature by the instrument.

10.4.4 Calibrate the pH meter using five buffers at pH 2.0, 4.0, 7.0, 10.0 and 12.0. The buffers should be fresh for each day of use.

10.4.4.1 Pour a small amount of the buffer solutions into separate disposable medicine cups, adding enough solution to cover the bulb at the bottom of the pH electrode. Add a stirring bar to each cup and place on a stirring plate. Turn on the plate stir function at a low level.

10.4.4.2 Allow the pH to stabilize and record the reading in the "Initial Cal Reading" box in the pH probe calibration logbook.

10.4.4.3 Follow the instructions for calibration as described in WI-DV-0034, pH Meter Calibration.

10.4.4.4 After each calibration point has been saved, allow the probe to remain in the buffer until the pH reading stabilizes. Record this reading in the "Final Cal Reading" box in the pH probe calibration logbook before moving to the next buffer solution.

10.4.4.5 The reading of the buffer solutions must be within ± 0.05 pH units of the certified buffer solution values. If they are not, recalibrate.

10.4.4.6 After the calibration is complete, record the slope in the instrument logbook. The source methods do not provide criteria for acceptance of the slope. The Orion 5 Star pH meter manual sets the acceptable slope range from 85% to 115%. If the slope falls outside this range, maintenance is required (see Sections 10.6 and 10.7).

10.4.4.7 Record the manufacturer and lot number of the buffers used in the pH probe calibration logbook. See Attachment 1.

10.4.5 Verify the calibration using a buffer solution (ICV). See Sections 7.4 and 9.3.1.

10.4.5.1 Record the pH, manufacturer, and lot number of the verification buffer used.

10.4.5.2 The reading of the buffer solution should be within ± 0.05 pH units of the true value. If this criterion is not met, the problem should be identified, corrected, and the meter recalibrated.

NOTE: Internal standard calibration is not an appropriate technique for the determination of pH.

10.5 Sample Analysis

10.5.1 Samples must be analyzed on the day they are prepared.

10.5.2 Analyze one LCS and one sample duplicate per batch of 20 samples.

10.5.3 Insert the electrode into the aqueous layer just far enough to cover the electrode bulb and junction. Do not allow the electrode to come into direct contact with oil.

10.5.4 Allow the reading to stabilize.

10.5.5 The pH reading, temperature, and time are recorded directly in TALS.

NOTE: Methods 9045C and 9045D require the sample temperature to be reported with each pH result. All sample temperatures are recorded on the instrument raw data. TALS reports the pH as pH adjusted to 25 °C to account for the temperature correction performed by the instrument.

10.5.6 Rinse the electrodes well between measurements.

10.5.7 A pH 7.0 buffer check (CCV) is required after every 10 or fewer samples (excluding the LCS/LCSD) and at the end of the run. See Section 9.3.2.

10.5.8 Record the balance ID, pH meter ID, the pH probe ID and the pH thermometer ID in the batch record.

10.5.9 Follow the instructions supplied with the electrodes for storage after use. Record daily maintenance in the pH Calibration and Maintenance Log. See Attachment 1.

10.6 Troubleshooting

10.6.1 Slow response or a wavering response is indicative of a dirty or oil-coated pH probe or that the probe is not properly connected to the meter. Samples high in dissolved CO_2 can cause the pH to change as the sample is stirred.

10.6.2 No temperature displayed may be a result of the temperature probe not being properly connected to the meter.

10.6.3 Using plastic disposable beakers and a magnetic stir plate and stir bar may generate static electricity that could affect stability. Turn off the stir plate, unplug and allow to sit for a few minutes.

10.7 Maintenance

10.7.1 Clean the electrode as needed following manufacturer's instructions.

10.7.2 See Section 20 of the Denver Quality Assurance Manual for maintenance procedures.

11.0 Calculations / Data Reduction

11.1 There are no calculations. This is a direct reading method. Data are manually entered in TALS at time of measurement.

11.2 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no MDL study for pH. The laboratory reports samples to the nearest 0.1 pH units and uses this increment as the MDL for reporting purposes.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration. The pH 7 Buffer solution (Section 7.5) is typically used.

12.2.2 The pH of each aliquot must be within 0.05 units of the true value.

12.2.3 If the analyte does not meet the acceptance criteria, the test must be repeated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Further details concerning demonstrations of proficiency are described in DV-QA-0024.

12.3 Training Requirements

The group leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use, has the required experience, and has successfully analyzed initial demonstration samples (see SOP DV-QA-0024 for details).

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on

quantity needed, and prepare reagents based on anticipated usage and reagent stability).

13.2 This method does not contain any specific modifications that serve to prevent or minimize pollution.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Acidic sample waste generated by the analysis – Aqueous Acidic (F).

14.2.2 Alkaline sample waste generated by the analysis – Aqueous Alkaline (E).

14.2.3 Exhausted soil samples utilized in the analysis – Soils (S)

14.2.4 Exhausted acidic and/or alkaline buffer solutions utilized in the analysis and expired standards and reagents – Contact the Waste Coordinator for guidance.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 9045D, “Soil and Waste pH”, Revision 4, November 2004.

15.1.2 Method 9045C, “Soil and Waste pH”, SW-846, Revision 3, 1995.

15.2 “Soil pH (Hydrogen Ion Activity)”, Methods of Soil Analysis, Second Edition, American Society of Agronomy, 1982.

16.0 Method Modifications

Item	Method	Modification
1	9045C/9045D	Temperature is not reported with the pH result. Sample pH is reported as pH adj. to 25°C.
2	9045C/9045D	Sample size used in this SOP is 40 g solid sample to 40 mL reagent water. This is the same proportion, though larger, than that stated in the source methods. The larger sample size helps ensure a more representative result. The source methods further state that additional dilutions may be made if the sample is hygroscopic. Additional water is added to the sample rather than starting with a new sample aliquot.
3	9045C/9045D	The source method has conflicting statements regarding use of additional dilutions for waste and starting with new aliquot and added double the volume of water. The laboratory adds additional reagent water starting with sample to water ratio of 1:3 rather than 1:2.

17.0 Attachments

Attachment 1: Example pH Calibration and Maintenance Log
Attachment 2: Example TALS Benchsheet

18.0 Revision History

- Revision 15, dated 31 August 2018
  - Annual Review

- Revision 14, dated 31 July 2017
  - Annual Review
  - Minor grammar and formatting changes throughout

- Revision 13, dated 08 July 2016
  - Revised Sections 10.4.4.1 – 10.4.4.6 for clarity and to match the new logbook format with regard to the calibration procedure

- Revision 12, dated 21 April 2016
  - Added reference to master list in Section 6.1.1
  - Updated Section 7.2 to reflect laboratory definition of reagent water
  - Added language to Section 8 to clarify holding time requirements
  - Reworded Section 9.3.1 due to redundancy
  - Changed the title of Section 10.4 to “Calibration”
  - Reworded Sections 10.4.4.1 through 10.4.4.5 to clarify calibration procedure
  - Added slope requirement to Section 10.4.4
  - Changed LIMS to TALS throughout
  - Minor grammar and formatting changes throughout

- Revision 11, dated 13 October 2015
  - Updated Section 1.2 to reflect pH RL and MDL used by the laboratory
  - Updated Section 7.4 to include a comment regarding daily replenishment
  - Updated Section 9.1 to be consistent verbiage across SOPs as applicable
  - Added requirement 10 section 10.4.4.1 to record pH result of calibration

- Updated Attachment 1 to reflect current pH logbook

- Revision 10, dated 31 January 2015
  - Moved Sections 10.5.2 and 10.5.3 to 10.4.2 and 10.4.3
  - Added Section 10.4.4.3 to require recording of slope to meet ORELAP requirement
  - Updated Attachment 1

- Revision 9, dated 31 July 2014
  - Revised Section 6 to reflect current practice
  - Revised Section 10.3 to add additional water when sample is hygroscopic rather than starting with new sample aliquot and to record the volume of water added.
  - Removed reference to recording slope from the calibration in Section 10.4.1.1. The source method does not address this and there is no established acceptance limit for the slope. Revised Attachment 1 to reflect change in documentation requirements.
  - Added Sections 10.6 and 10.7, Troubleshooting and Maintenance, respectively.
  - Revised Section 12.2 to reflect use of pH 7 Buffer solution as QC sample.
  - Expanded Method Modification #2 with more detail and added Method Modification #3.
  - Source method review.

- Revision 8, dated 31 July 2013
  - Added section 3.2
  - Added section 10.5.9
  - Revised section 11.1 to note data are manually entered
  - Annual Review

- Revision 7, dated 27 July 2012
  - Removed HCl and cleaning procedure for probe.
  - Revised Section 5 based on removal of HCl.
  - Replaced magnetic stirrer with shaker table and removed magnetic stirrers in Section 6.
  - Updated Section 7 to include reagent water and second source ICV buffer at pH 7.0.
  - Revised Section 8
  - Revised Section 9.1, 10.1, 10.2 to reflect current practice
  - Revised calibration procedure (Section 10.4)
  - Moved procedural note for cleaning electrode in Section 4 to Section 10.5.3
  - Removed Attachment 2 and added statement about data review with reference to removed checklist. (Section 11.2)
  - Source method review
  - Formatting and editorial changes throughout

- Revision 6.5 dated 31 January 2012
  - Annual Technical review
  - Updated Section 9.1.1 to read LCS/LCSD

- Revision 6.4 dated 11 February 2011
  - Annual Technical review
  - Updated Attachments 1 & 2

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Example pH Calibration and Maintenance Log

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Denver

Calibration and Maintenance Log

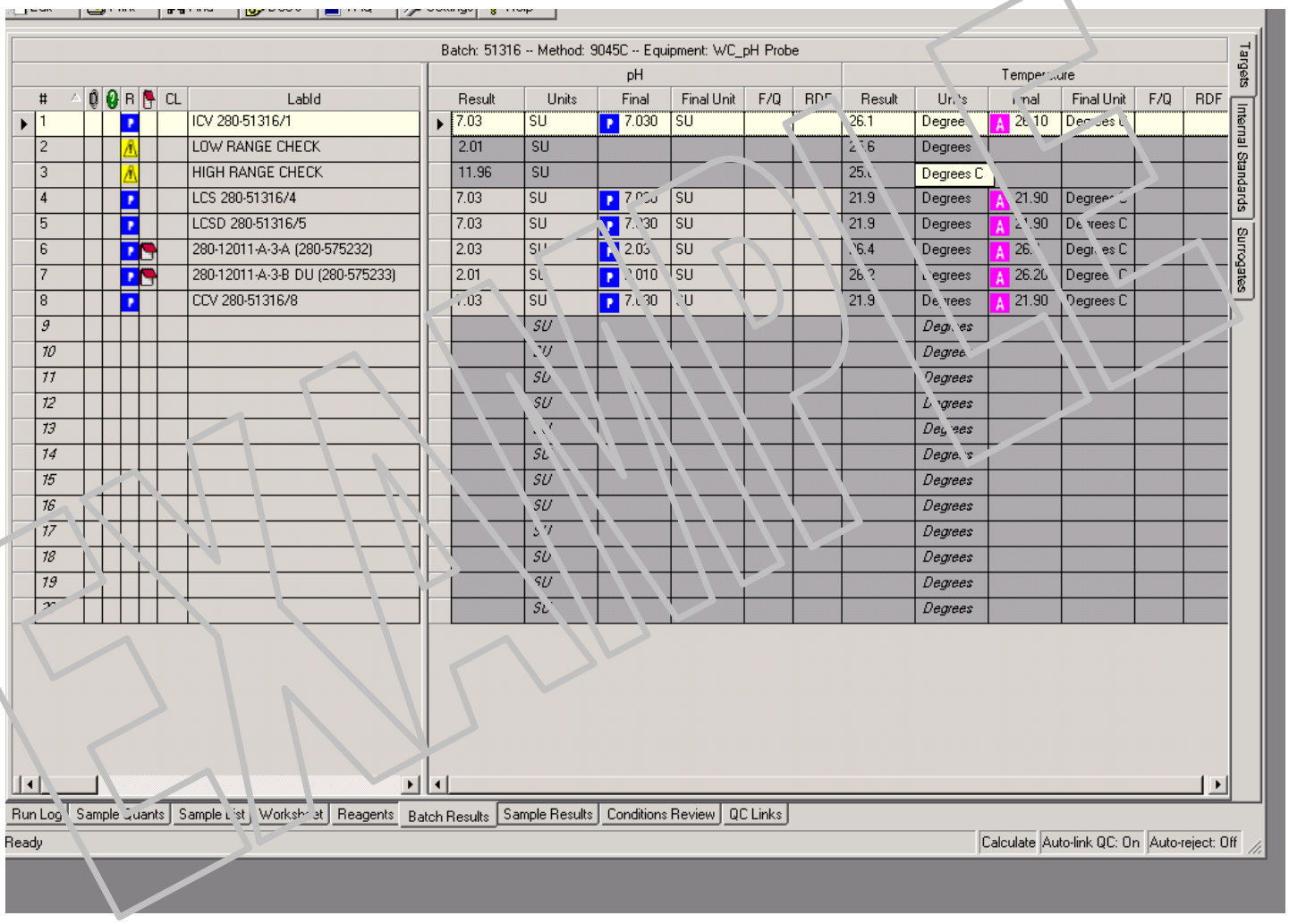
Wet Chemistry / pH Probe

Daily Maintenance	Day	Sat	Sun	Mon	Tue	Wed	Thu	Fri
No maintenance required when instrument is not in use.	Date/Time:							
	Analyst:							
1) Inspect the probe for scratches or cracks.								
2) Probe solution refilled.								
3) Store probe in storage solution.								
4) Wipe off apparatus and clean up any spills.								
Calibration Standards:	2.0 Buffer Lot #: Expiration Date:							
	2.0 Buffer Calibration Reading							
	4.0 Buffer Lot #: Expiration Date:							
	4.0 Buffer Calibration Reading							
	7.0 Buffer Lot #: Expiration Date:							
	7.0 Buffer Calibration Reading							
	10.0 Buffer Lot #: Expiration Date:							
	10.0 Buffer Calibration Reading							
	12.0 Buffer Lot #: Expiration Date:							
	12.0 Buffer Calibration Reading							
Calibration Slope:								
Instrument Removed From Service: Y / N	Initials:	Date:	Return to Service with Passing Calibration				Initials:	Date:

Additional Maintenance/Comments:

Attachment 2.

Example TALS Benchsheet



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Percent Moisture in Soils and Wastes

[ASTM D2216, CLP ILM05.3, SW 3550C]

Approvals (Signature/Date):

Andrew Allen 10/30/17 Technical Specialist	Doug Gomer 10/30/17 Health & Safety Manager / Coordinator
Roxanne Sullivan 10/30/17 Quality Assurance Manager	William S. Cicero 10/30/17 Laboratory Director

Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This standard operating procedure (SOP) provides instructions for determining percent moisture (or percent solids) in samples and is based on CLP ILM05.3, ASTM D 2216-05, and SW-846 Method 3550C.

1.2 This method is applicable to soils and sludges. Wipe samples do not require % moisture. Please see Group leader or Project Manager before proceeding with % moisture for any other unusual matrices For the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids, refer to SOP DV-WC-0064.

1.3 The practical range for the determination of percent moisture is from 0.4% to 99.6%, based on the uncertainty of the top-loading balance used.

2.0 Summary of Method

2.1 The percent moisture of a solid or semi-solid sample is determined gravimetrically by weighing a 15 g aliquot of the homogenized "wet" sample, letting the sample dry in an oven at 100 ± 5 °C for a minimum of 12 hours and weighing the remaining "dry" residue.

2.2 The moisture lost by the sample is calculated as the difference between the "wet" mass and the "dry" mass. Percent moisture and percent solids are calculated relative to the original "wet" sample mass.

3.0 Definitions

3.1 Percent Moisture: The amount of water in a solid or semi-solid material that is lost as a result of drying the material in an oven at a defined temperature, typically 100 ºC, and expressed as a percent of the original sample mass. The moisture determined in this manner is not necessarily the total water content of a material. For example, drying a sample at 100 ºC will not remove waters of hydration.

3.2 Percent Solids: The amount of solid in a solid or semi-solid material that is retained after drying the material in an oven at a defined temperature, typically 100 ºC, and expressed as a percent of the original sample mass. The solid determined in this manner is not necessarily the total solid content of a material. For example, drying a sample at 100 ºC will not remove waters of hydration.

3.3 Refer to the Glossary of the *TestAmerica Denver Quality Assurance Manual* (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 The principal source of error for this method is failure to obtain a representative sample. Non-representative particulates, e.g., rocks or leaves in a soil sample, should be excluded from the sample if it is determined that their inclusion is not

desired in the final result. Any observations of odd sample matrices or exclusions of sample fractions must be recorded by the analyst.

4.2 Positive biases may result from, among other things, evaporative losses, loss of water of crystallization, and loss of volatile organic matter during drying.

4.3 Negative biases may result from the presence of significant amounts of oil and grease, as well as the absorption of moisture after drying.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Be careful when placing samples into and taking samples out of the drying oven (100 ºC). Wear insulated gloves or use tongs when handling hot weighing dishes.

5.4 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. **Note: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

6.1 Aluminum weigh pans, disposable.

6.2 Disposable wooden spatula.

6.3 Top-loading balance, 0.01 g readability.

6.4 Drying oven, capable of maintaining a temperature of 100 ± 5 °C.

6.5 Insulated gloves or tongs.

6.6 Desiccator.

6.7 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents and Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

DRIERITE desiccant (anhydrous calcium sulfate): Available from commercial sources.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples are to be collected in a glass or plastic bottle with a tight fitting cap and refrigerated to ≤ 6 °C.

8.2 There is no regulatory holding time for this parameter; however, analysis should begin as soon as possible.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.1 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-

0031. This is in addition to the corrective actions described in the following sections.

10.0 QC Samples

10.1.1 A method blank is not applicable to percent moisture determinations and is not required by the source methods.

10.1.2 Laboratory control samples are not applicable to percent moisture determinations and are not required by the source methods.

10.1.3 One sample duplicate is required with each batch of 20 or fewer samples.

Acceptance Criteria: When the moisture content of the sample is greater than 10%, the relative percent difference (RPD) between the sample and sample duplicate must be ≤ 20%.

Corrective Action: If the RPD for the duplicate samples exceeds the established limit, then the sample must be reanalyzed, if any sample is remaining. If no sample is remaining, then the data must be appropriately flagged.

10.1.4 Matrix spikes and matrix spike duplicates are not applicable to percent moisture determinations and are not required by the source methods.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Calibration

11.3.1 The top loading balance is calibrated annually by an outside vendor as described in SOP DV-QA-0014.

11.3.2 Verify the accuracy of the balance daily before use as described in SOP DV-QA-0014. Daily calibration verifications are recorded in a balance logbook.

11.4 Sample Analysis

11.4.1 Perform all weighings as quickly as possible. Wet samples lose mass through evaporation of water. Dried samples can be very hygroscopic and absorb moisture from the air.

11.4.2 Mass measurements are electronically captured for direct entry into TALS. The TALS Worksheet performs all calculations. If necessary, the data may be directly entered manually into the Worksheet tab in TALS. Weights should not be recorded on secondary sheets for later entry.

11.4.3 For each sample in the batch, label the aluminum weigh pan with the sample number. Label an additional pan for the sample duplicate.

11.4.4 Weigh each pan on the balance to determine the mass (tare weight) of the pan. Record the tare weight to the nearest 0.01 g for each pan. Collect all of the pans for each batch onto a single aluminum tray.

11.4.5 Homogenize the sample by stirring and pulverizing any large chunks.

11.4.6 Place a 13 - 17 g representative subsample into the weigh pan. Record the "wet" sample mass as displayed by the balance.

11.4.7 Note any observations of the sample condition on the benchsheet (e.g., “rocky”).

11.4.8 Carefully place the tray of pans into the drying oven that has been pre-heated to 100 ± 5 ºC. Allow the samples to dry for at least 12 hours. Record the time the samples were placed into the oven, oven temperature when the samples were placed in the oven (observed temperature and corrected temperature), the time they were removed (to document the drying time), and the temperature of the oven (observed temperature and corrected temperature). Also record the thermometer ID and balance ID. (See Attachment 1 for example entries in the Batch Editor.)

NOTE: Samples may be dried for less than 12 hours if it can be demonstrated that a constant mass is obtained. In this case, data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with at least 1 hour of drying time in each cycle. Constant mass is defined as a loss in mass of no greater than 0.01 g between the starting weight and the final weight of the last cycle.

11.4.9 At the end of the drying time, remove the tray of pans from the oven and immediately place it in a desiccator. Record the time samples are placed in the desiccator. Allow the samples to cool to room temperature (at least 1 hour). Record the time the samples are removed from the desicator.

NOTE: The desiccant is blue in color when dry. When the indicator color changes to purplish-pink, the desiccant must be replaced.

11.4.10 If the sample appears oily, generate an NCM to document the anomaly: "Results for sample (indicate sample number) may be inaccurate - sample appeared oily after drying."

11.4.11 Weigh the dried sample and record the mass to the nearest 0.01 g (see Attachment 2).

11.5 Troubleshooting and Maintenance

11.5.1 DRIERITE in the desiccators should be changed when it turns purplish-pink.

11.5.2 If the oven temperature cannot be maintained within range, notify facility maintenance.

12.0 Calculations / Data Reduction

12.1 Raw data are entered directly into TALS and calculations are performed by the Worksheet tab in the analytical batch. The following calculations represent those performed by TALS and can be used to manually verify these calculations.

12.2 Calculation of Solids Fraction

The fraction of solids in the original sample is used to correct the concentration of other measured analytes in a wet sample for the dry sample weight. The fraction of solids in a sample is calculated as follows:

$$\text{solids fraction} = \frac{D - T}{W - T} \qquad \textit{Equation 1}$$

Where:

D = Gross weight (mass) of the dried sample and the weigh pan (grams).
T = Tare weight (mass) of the weigh pan (grams)
W = Gross weight (mass) of the wet sample and the weigh pan (grams).

12.3 Calculation for Percent Solids

The percent solids in a sample is calculated by multiplying the fraction of solids by 100%, as follows:

$$\%\,\text{solids} = \frac{D - T}{W - T} \times 100\% \qquad \textit{Equation 2}$$

12.4 Calculation for Percent Moisture

TALS uses the percent moisture value to correct reported wet-weight analyte concentrations to a dry-weight basis. The percent moisture in a sample is calculated by subtracting the percent solids from 100%, as follows:

$$\%\,\text{moisture} = 100\% - \left\lfloor \frac{D-T}{W-T} \times 100\% \right\rfloor \qquad \textit{Equation 3}$$

12.5 The relative percent difference is calculated for the sample and sample duplicate determinations using the formula:

$$\%\,RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2}\, x\, 100\% \qquad \textit{Equation 4}$$

Where R_1 and R_2 are the calculated % moisture results for the sample and sample duplicate.

12.6 When reporting chemical concentrations on a dry-weight basis, divide the wet weight concentration by the fraction of solids in the sample, as follows:

$$\text{Dry Weight Concentration} = \frac{\text{Wet Weight Concentration}}{\text{Solids Fraction}} \qquad \textit{Equation 5}$$

12.7 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for a copy of the checklist and for more detail on the review process.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial method detection limit study is not performed for percent moisture. The lower limit of quantitation is dictated by the accuracy and sensitivity of the balance used. This test uses a top loading balance with a readability of 0.01 g. The balance must pass daily calibration verification within ± 0.02 g for weights less than 20 g. If the daily calibration verification control limits represent the maximum "noise" level of the balance, then three times this level, or 0.06 g, would be a reasonable lower limit of quantitation. This translates to a practical measurement range of 0.4 to 99.6 % moisture.

13.2 Demonstration of Capabilities

13.2.1 Since a spiked aliquot is not appropriate for this procedure, initial and continuing demonstration of capability is documented by collecting data for a completed batch. An acceptable IDOC is determined by meeting any method required batch QC.

13.2.2 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024, *Training*.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Solid Sample Waste – Waste Stream S

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 USEPA Contract Laboratory Program Statement of Work for Inorganic Analytes, Multi-Media, Multi-Concentration, ILMO 5.3, March 2004, Exhibit D, Section 1.6, *Percent Solids Determination Procedure*.

16.2 ASTM D 2216, *Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*, ASTM International, March 1, 2005.

16.3 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550C, *Ultrasonic Extraction*, Revision 3, December 1996.

17.0 Method Modifications:

Item	Method	Modification
1	ILMO5.3 ASTM D 2216 3550C	ILMO5.3 and 3550C specify a sample aliquot of 5 to 10 grams. ASTM D 2216 lists a minimum sample size of 20 grams, but allows discretion for the use of smaller aliquots. The 15-gram aliquot specified in this SOP is appropriate for the size of the weigh pan used.
2	ILMO5.3, ASTM D 2216 3550C	ILMO5.3 specifies a drying temperature of 103 to 105 ºC. ASTM D 2216 specifies a drying temperature of 110 ± 5 ºC. Method 3550C states a drying temperature of 105°C. These temperature specifications are based on the boiling point of water at sea level, which is 100 ºC. The boiling point of water at the altitude of the laboratory location is 95 ºC. Therefore, the acceptable drying temperature range for this SOP has been set at 100 ± 5 ºC to provide results comparable to performing this method at sea level. This is justified by the discussion in ASTM D 2216, which notes that materials containing significant amounts of hydrated water (e.g., gypsum) may present a special problem as these materials can slowly dehydrate at the standard drying temperature of 110 ºC and at very low relative humidity.
3	ILMO5.3	ILMO5.3 allows a single weighing of the dried sample, but only if the sample is dried for a period of at least 12 hours and no longer than 24 hours. If the sample is dried for a period of time less than 12 hours, then it must be demonstrated that a constant weight has been achieved by repeating the drying, cooling, and weighing cycle at least twice. This SOP prescribes a drying time of at least 12 hours. Samples may be left in the ovens over the weekend. This is because the determination of moisture content is not affected by longer drying periods.
4	ILMO5.3, ASTM D 2216	Because of the sample aliquot size and the type of balance used, the practical range of the method is limited to 0.4 to 99.6 weight percent.
5	ILMO5.3, ASTM D 2216	Aluminum weigh dishes are used instead of porcelain crucibles as a matter of convenience. The porcelain dish is not necessary, because the determination of volatile solids, which requires heating in a muffle furnace at 550 ºC is not included in the scope of this SOP.
6	ILMO5.3	Method ILM05.3 requires the use of a lid. The lid is not necessary, because the determination of volatile solids in not included in the scope of this SOP. This is consistent with ASTM D 2216 which states the lid is optional.
7	3550C	Method 3550C states to weigh the sample for dry weight determination immediately following the weighing of the aliquot for extraction. In this laboratory, the dry weight determination is done separately from the preparation of that solid sample for any other tests. The dry weight determined is used for dry weight correction of all tests on that sample. The aliquot used for the determination of dry weight is taken from a container that is clearly marked with the client and lab sample IDs for that particular suite of tests. Depending upon the clients' practices, the sample for dry weight may or may not come from the same container used for one of the other tests for that sample..

18.0 Attachments

Attachment 1: Example Batch Editor Screen
Attachment 2: Example Gravimetric Calculation Benchsheet

19.0 Revision History

- Revision 9, dated 31 October 2017
  - Added statement to section 2.1 about different matrices.
- Revision 8, dated 31 January 2017
  - Annual Review
- Revision 7, dated 29 February 2016
  - Annual Review
  - Corrections for structure and grammar throughout
  - Removed pre-2011 revision history
  - Added new Section 3.2, definition of percent solids
  - Removed reference to hazard table in Section 5.4
  - Added language to Section 10.4.2 for clarity
  - Reworded Section 10.4.8 for clarity
  - Changed Section 10.4.9 to align with standard SOP language
  - Changed LIMS to TALS throughout

- Revision 6, dated 28 February 2015
  - Revised section 10.4.6 to specify range of weights for sample
  - Revised Section 10.4.8 to list all items to be documented in the batch record (observed and corrected temperatures both in an out of oven, time in and out of oven)
  - Revised section 10.4.9 to include recording of times in and out of the desiccator
  - Added new Attachment 1 to provide example of documentation of equipment, oven temperatures, and times.
  - Annual Technical Review

- Revision 5, dated 28 February 2014
  - Added Section 10.5
  - Annual technical review

- Revision 4, dated 28 February 2013
  - Added Method 3550C as a reference and throughout SOP as needed
  - Added section 3.2
  - Revised section 9.1, 10.1, and 10.2 to reflect current practice
  - Revised section 10.4.2 to reflect that electronic transfer of weights to the LIMS is the preferred practice for recording data
  - Moved section on data review from section 10 to 11.7 for consistency with other documents
  - Revised section 12.2 to describe the appropriate documentation of an IDOC for a non-spiked test
  - Revised Method modifications 1 and 2 to include Method 3550C
  - Added Method Modification 7 to address determination of percent moisture as an independent test from other preparations of that sample for analysis
  - Annual technical review
  - Formatting and grammatical changes throughout

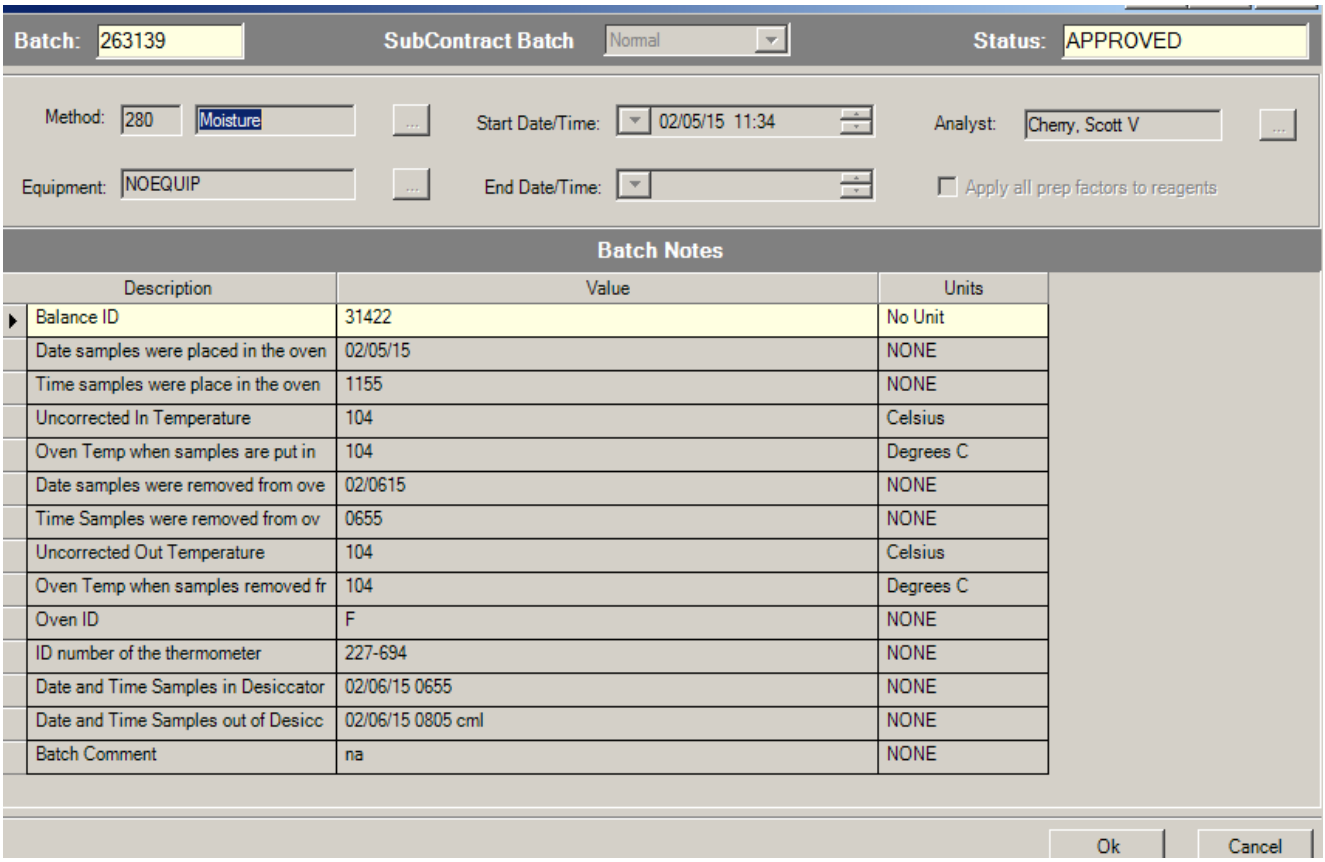
- Revision 3.4, dated 28 February 2012
  - Source method review
  - Added gloves and disposable spatulas to supplies list.
  - Added calculation for % RPD
  - Removed statement that percent moisture of a sample is calculated and entered into the LIMS as raw data are input into the LIMS and result calculated by the LIMS
  - Removed Method EPA 160.3 from references and method modifications. (Previously removed in Revision 3.)
  - Revised method modifications to address sample size, drying temperature and optional use of lid per ASTM D 2216.
  - Removed Attachment 2 and inserted discussion of data review process in section 10.5 with reference to data review SOP and form.

- Revision 3.3, dated 04 March 2011
  - Annual Technical Review.
  - Updated Attachments 1 & 2

Earlier revision histories have been archived and are available upon request

Attachment 1.

Example Batch Editor Screen



Batch: 263139 SubContract Batch Normal Status: APPROVED

Method: 280 Moisture ... Start Date/Time: 02/05/15 11:34 Analyst: Cherry, Scott V ...

Equipment: NOEQUIP ... End Date/Time: Apply all prep factors to reagents

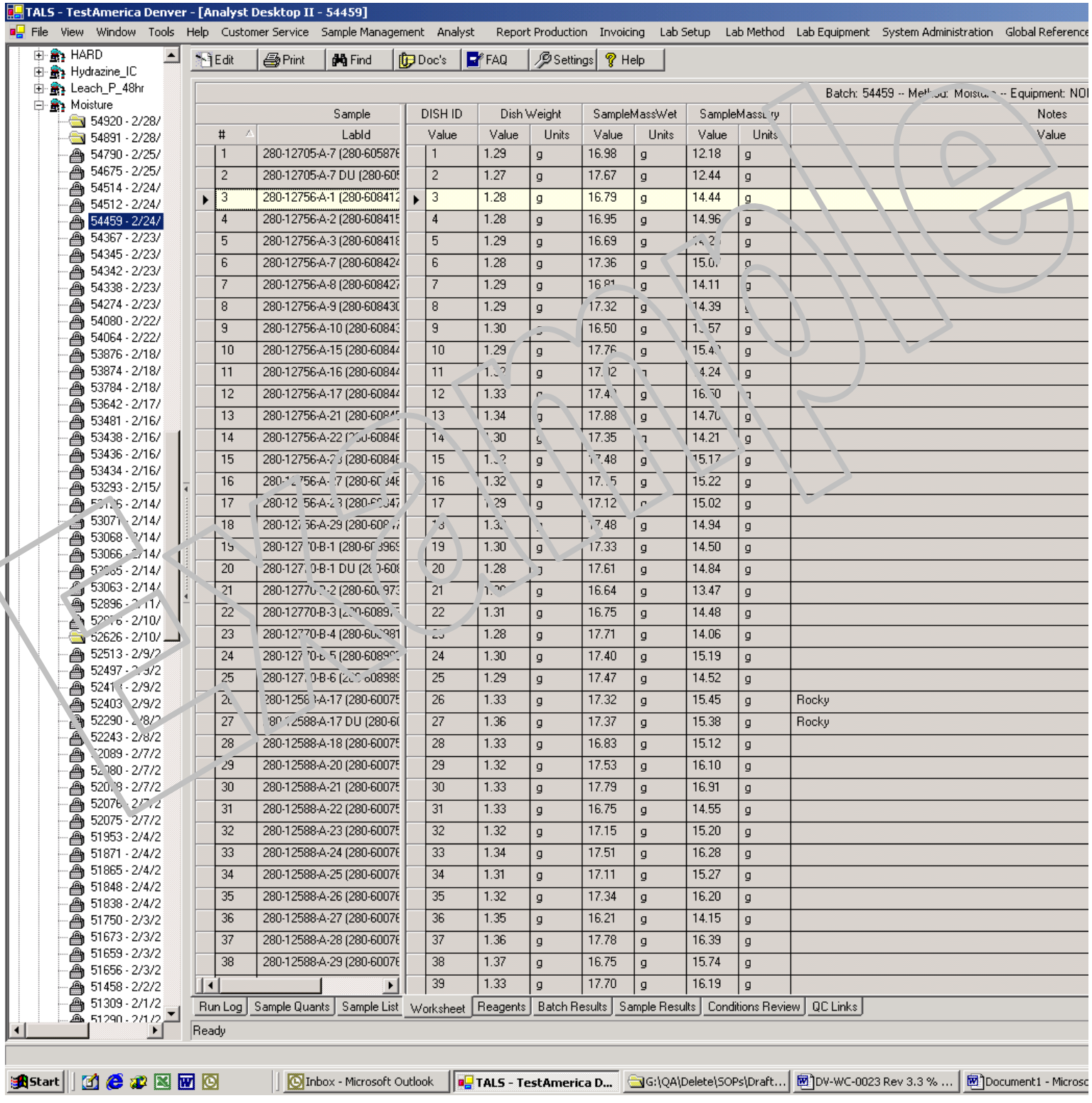
Batch Notes

Description	Value	Units
Balance ID	31422	No Unit
Date samples were placed in the oven	02/05/15	NONE
Time samples were place in the oven	1155	NONE
Uncorrected In Temperature	104	Celsius
Oven Temp when samples are put in	104	Degrees C
Date samples were removed from ove	02/0615	NONE
Time Samples were removed from ov	0655	NONE
Uncorrected Out Temperature	104	Celsius
Oven Temp when samples removed fr	104	Degrees C
Oven ID	F	NONE
ID number of the thermometer	227-694	NONE
Date and Time Samples in Desiccator	02/06/15 0655	NONE
Date and Time Samples out of Desicc	02/06/15 0805 cml	NONE
Batch Comment	na	NONE

Ok Cancel

Attachment 2.

Example Gravimetric Calculation Benchsheet



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Manual and Automated pH [SM 4500-H+ B, SW 9040B & C]

Approvals (Signature/Date):

Connie Jewell — 6/27/19 — Date
Department Supervisor

Doug Gomer — 6/27/19 — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan — 6/27/19 — Date
Quality Assurance Manager

Charles Newton — 6/27/19 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This is an electrometric procedure for measuring pH in aqueous samples. This method is applicable to the analysis of drinking, surface, and saline waters, and acid rain. It also applies to multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of the waste. Corrosivity of concentrated acids and bases cannot be measured by this procedure.

1.2 Liquid samples which are not miscible with water or solids must be analyzed by DV-WC-0001, Soil and Waste pH.

1.3 A detection limit (MDL) for pH has not been defined, however, for reporting purposes this laboratory uses 0.1 pH units as the RL and MDL.

1.4 This method is applicable to all ranges of pH.

2.0 Summary of Method

The pH meter, glass electrode, and reference electrode (or single combination electrode) are standardized using five reference buffer solutions of known pH bracketing the pH expected to be found in the sample. The sample measurement is made by immersing the electrodes into the test solution and taking a reading from the meter.

3.0 Definitions

3.1 **pH** - At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Because of ionic interactions in all but very dilute solutions, it is necessary to use the “activity” of an ion and not its molar concentration. The use of the term pH assumes that the activity of the hydrogen ion is being considered. The approximate *equivalence* to molarity can be presumed only in very dilute solutions. A logarithmic scale is used to accommodate the wide range of ionic activities.

3.2 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 The pH response of most glass electrodes is imperfect at both ends of the scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of salts and strong acids having a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of the proper electrodes for these conditions. For example, sodium may interfere at pH > 10, and is controlled by using a “low sodium error” electrode.

4.2 Coatings of oil and particulate matter may impair electrode response.

4.3 Temperature variations will change the pH of the samples and also affect electrode response. Electronic temperature correction may be used to correct for electrode response.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1 (as per the Corporate Environmental Health and Safety Manual), laboratory coat, and nitrile or latex gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS (formerly known as MSDS) for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Manual pH

6.1.1.1 pH meter including temperature compensation ability. Details of the pH meter and electrode currently in use can be found at R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision).

6.1.1.2 Glass electrode with reference electrode--a calomel, silver-silver chloride or other reference electrode of constant potential may be used; or use a combination electrode that incorporates both measuring and reference functions.

6.1.1.3 Magnetic stirrer and Teflon-coated stir bars.

6.1.2 Automated pH

Man-Tech Autotitrator (WC-AT3), consisting of:

- Burivar I/2 Buret Module
- Titrasip Titration Module
- PC-Tis Interface Module
- PC running PC-Titrate software

6.2 Supplies

6.2.1 Tubes to fit autosampler, (these must be thoroughly rinsed to remove all traces of salt if reused).

6.2.2 Pipette calibrated to 5 mL, and disposable tips.

6.2.3 Disposable beakers.

6.2.4 Miscellaneous laboratory apparatus and glassware.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE: TALS reagent codes are given in parentheses.

7.2 pH Buffers, 2, 4, 7, 10, and 12: These buffers should be obtained commercially and traceable to NIST standards. Other buffers can be used as appropriate to bracket the range of each sample. (TALS Reagent codes: pH 2.0 Buffer, pH 4.0 Buffer, pH 7.0 Buffer, pH 10 Buffer, pH 12 Buffer). The solution aliquots used to calibrate the pH meter must be replenished each day of use.

7.3 ICV Buffer Solution (pH 7.0 ICV): A pH 7 buffer solution from a second source provider, obtained commercially and traceable to NIST standards. The ICV solution aliquot used must be replenished each day of use.

7.4 Laboratory Control Sample (LCS) Solution (pH 7.0 Buffer): The LCS solution must be certified for pH and is commercially available. The pH 7 buffer from Section 7.2 is normally used as the LCS.

7.5 3 M Potassium Chloride: This solution is purchased from Thermo.

7.6 Reagent water: Water obtained from the laboratory DI system. For information on the DI system see SOP DV-QA-0026, *DI Water Monitoring*.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation[1]	Holding Time[2]	Reference
Water	Glass or plastic	100 mL	None	None	SW-846
Water	Glass or plastic	100 mL	None	Analyze within 15 minutes	40 CFR Part 136.3

[1] 40 CFR Part 136.3 and SW-846 indicate no preservation is required for pH. It is intended by both programs that the samples be analyzed in the field. TestAmerica Denver typically refrigerates these samples because the aliquot tested is taken from a sample container that is used for other tests that do require refrigeration.

[2] pH is intended to be a field measurement. The laboratory attempts to measure pH as soon as possible upon receipt. All laboratory analyzed samples are flagged as out of hold.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the

analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC - The following quality control samples are prepared with each batch of samples.

9.2.1 Laboratory Control Sample (LCS):

One LCS is required with each batch of samples processed, not to exceed 20 samples.

Acceptance Criteria: The LCS must be within ± 0.05 pH units of the true value. Note: This limit is presented in TALS as 99-101% based on the use of the pH 7 buffer.

Corrective Action: If the LCS is not within the control limits, rerun all associated samples.

9.2.2 Duplicate Samples:

One duplicate must be analyzed with each batch of samples processed, not to exceed 20 samples.

Acceptance Criteria: The two results should agree within ± 0.10 pH units.

Corrective Action: If the difference is greater than ± 0.10 repeat the analysis. If the difference still exceeds the control limit the data will be flagged as outside of the limit.

9.2.3 Method blanks and matrix spikes are not applicable to pH.

9.3 Instrument QC

9.3.1 Initial Calibration Verification

Record the expected pH, manufacturer, and lot number of the verification buffer used for a second source pH 7.0 buffer solution. Analyze the second source pH 7 buffer solution.

Acceptance Criteria: The ICV buffer must read within ± 0.05 pH units of the true value.

Corrective Action: If this criterion is not met, the problem should be identified, corrected, and the meter recalibrated.

9.3.2 Continuing Calibration Verification

A pH 7.0 buffer check is required after every 10 or fewer samples and at the end of the run. The CCV is the same pH 7 buffer solution used in the initial calibration.

Acceptance Criteria: The CCV pH buffer checks must be within ± 0.05 units of the true value.

Corrective Action: If the pH 7.0 buffer check is outside of the control limits, rerun all samples since the last acceptable pH 7.0 buffer check.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Manual Sample Analysis

10.3.1 Follow the operating instructions supplied by the manufacturer of the pH meter. See WI-DV-0034 for more information concerning meter calibration.

10.3.2 Record instrument ID, pH probe ID, thermometer probe ID, and reagent IDs in the batch record in TALS.

10.3.3 When using the Thermo Five Star pH meter, all results are to be temperature corrected to 25 °C using the Automatic Temperature Compensation function available with the instrument.

NOTE: Methods 9040 B & C state “The sample temperatures must be within ± 2 °C of the calibrated buffers or temperature corrected.” All samples are automatically corrected for temperature by the instrument.

10.3.4 Calibrate the pH meter using five buffers at pH 2.0, 4.0, 7.0, 10.0, and 12.0. The aliquots of buffers should be fresh for each day of use.

10.3.4.1 Pour a small amount of the buffer solutions into separate disposable medicine cups, adding enough solution to cover the bulb at the bottom of the pH electrode. Add a stirring bar to each

cup and place on a stirring plate. Turn on the plate stir function at a low level.

10.3.4.2 Allow the pH to stabilize and record the reading in the "Initial Cal Reading" box in the pH probe calibration logbook.

10.3.4.3 Follow the instructions for calibration as described in WI-DV-0034, pH Meter Calibration.

10.3.4.4 After each calibration point has been saved, allow the probe to remain in the buffer until the pH reading stabilizes. Record this reading in the "Final Cal Reading" box in the pH probe calibration logbook before moving to the next buffer solution.

10.3.4.5 The reading of the buffer solutions must be within ± 0.05 pH units of the certified buffer solution values. If they are not, recalibrate.

10.3.4.6 After the calibration is complete, record the slope in the instrument logbook. The source methods do not provide criteria for acceptance of the slope. See the pH meter manual for acceptable slope range. If the slope falls outside this range, maintenance is required (see Sections 10.6 and 10.7).

10.3.4.7 Record the manufacturer and lot number of the buffers used in the pH probe calibration logbook. See Attachment 1.

NOTE: Internal standard is not an appropriate calibration technique for the determination of pH.

10.3.5 Verify the calibration using a buffer solution (ICV). See Sections 7.3 and 9.3.1.

10.3.5.1 Record the pH, manufacturer, and lot number of the verification buffer used.

10.3.5.2 The reading of the buffer solution should be within ± 0.05 pH units of the true value. If this criteria is not met, the problem should be identified, corrected, and the meter recalibrated.

NOTE: Internal standard calibration is not an appropriate technique for the determination of pH.

10.3.6 Analyze one LCS and one sample duplicate per batch of 20 samples.

10.3.7 Pour enough sample into a beaker to cover the electrodes and place on the magnetic stirrer. Stirring should be fast enough to provide homogeneity and keep solids suspended, but should not disturb the air-water interface. Acid rain samples should not be stirred.

10.3.8 Immerse the electrodes in the sample and allow the reading to stabilize. The pH is recorded to the nearest 0.01 pH.

10.3.9 Repeat measurement on successive volumes of sample until the values differ by less than 0.1 pH units. Two or three volume changes are usually sufficient.

10.3.10 The pH reading, temperature, and time are recorded directly in TALS by the analyst at the time of measurement.

NOTE: Methods 9040B & C require the sample temperature to be reported with each pH result. All sample temperatures are recorded on the instrument raw data. TALS reports the pH as pH adjusted to 25 °C to account for the temperature correction performed by the instrument. If the pH of a sample is greater than 12, for 9040C, the temperature of the sample must be 25 +/- 1 degree C. Document if it is not.

10.3.11 Rinse the electrodes with a stream of reagent water in between samples.

10.3.12 Rinse the magnetic stir bars with reagent water in between samples.

10.3.13 Follow the instructions supplied with the electrodes for storage after use. Record daily maintenance in the pH Calibration and Maintenance Log. See Attachment 1.

10.4 Automated Sample Analysis using the Man-Tech Autotitrator

10.4.1 The pH meter is calibrated each day of operation.

10.4.2 Be sure the reference electrode has been filled with 3 M potassium chloride.

10.4.3 Calibrate the pH meter using pH 2, 4, 7, 10, and 12 buffers.

10.4.3.1 The reading of the buffer solutions must be within ± 0.05 pH units of the certified buffer solution values. If they are not, recalibrate.

10.4.3.2 Fill the first seven tubes in the autosampler with the following order of samples: pH 2 buffer, pH 4 buffer, pH 7 buffer, pH 10 buffer, pH 12 buffer, and reagent water.

10.4.3.3 Click on the button “PH CALIBRATION” and follow the screens to calibrate.

10.4.4 When calibration has finished, go to titrator and choose “examine calibrations.” Print the calibration if instrument states it is valid. If the calibration is not valid, recalibrate.

10.4.5 Check the calibration using a buffer solution (ICV). See Sections 7.3 and 9.3.1.

10.4.5.1 Record the pH, manufacturer, and lot number of the verification buffer used.

10.4.5.2 The reading of the buffer solution should be within ± 0.05 pH units of the true value. If this criteria is not met, the problem should be identified, corrected, and the meter recalibrated.

NOTE: Internal standard calibration is not an appropriate technique for the determination of pH.

10.4.6 Load field samples into the autosampler and initiate the analysis.

10.5 Troubleshooting

10.5.1 Slow response or a wavering response is indicative of a dirty or oil-coated pH probe or that the probe is not properly connected to the meter. Samples high in dissolved CO_2 can cause the pH to change as the sample is stirred.

10.5.2 No temperature displayed may be a result of the temperature probe not being properly connected to the meter.

10.5.3 Using plastic disposable beakers and a magnetic stir plate and stir bar may generate static electricity that could affect stability. Turn off the stir plate, unplug and allow to sit for a few minutes.

10.6 Maintenance

10.6.1 Clean the electrode as needed following manufacturer's instructions.

10.6.2 See Section 20 of the Denver Quality Assurance Manual for maintenance procedures.

11.0 Calculations / Data Reduction

11.1 For Manual pH, data are entered directly into the worksheet in TALS at the time of observation. There is no transcription of data.

11.2 For Automated pH, the data are recorded by the instrument and uploaded to TALS when the run is complete. There is no transcription of data.

11.3 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no MDL study for pH. The laboratory reports samples to the nearest 0.1 pH units and uses this increment as the MDL for reporting purposes.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1.1 Four aliquots of the QC check sample (LCS) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.1.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.1.4 Further details concerning demonstrations of proficiency are described in DV-QA-0024.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use, has the required experience, and has successfully analyzed initial demonstration samples (see SOP DV-QA-0024 for details).

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

13.2 This method does not contain any specific modifications that serve to prevent or minimize pollution.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Acidic or neutral sample waste – Waste Stream F

14.2.3 Basic sample waste – Waste Stream E

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 9040B, "pH Electrometric Measurement", Revision 2, January 1995.

15.1.2 Method 9040C, "pH Electrometric Measurement", Revision 3, November 2004.

15.2 "Standard Methods for the Examination of Water and Wastewater, online Edition; Clesceri, L.S.; Greenberg, A.E.; Eaton, A.D.; Editors; American Public Health Association, American Water Works Association, and Water Environment Federation.

15.2.1 Method 4500-H+ B-2000 or Method 4500--H+ B-2011

16.0 Method Modifications:

Item	Method	Modification
1	9040B 9040C SM 4500 H+ B	Temperature is not reported with the pH result. Sample pH is reported as pH adj. to 25°C.
2	9040B 9040C SM 4500 H+ B	Methods 9040B and 9040C specify use of glass beakers while SM recommends polyethylene. This procedure uses disposable plastic beakers.
3	SM 4500 H+ B	The Standard Methods method states buffer solutions be replaced every 4 weeks. This procedure uses commercially available buffer solutions and uses the manufacturer's expiration date.
4	9040B 9040C SM 4500 H+ B	The source documents do not require preservation of samples for pH as it is intended as a field measurement. The laboratory typically does refrigerate samples during transit and prior to analysis.

17.0 Attachments

Attachment 1: Example pH Calibration and Maintenance Log

Attachment 2: Example Autotitrator pH Calibration Report

Attachment 3: Example Benchsheet

18.0 Revision History

- Revision 16, dated 30 June 2019
  - Annual review
  - Updated copyright information

- Revision 15, dated 30 June 2018
  - Annual review
  - Added statement to section 10.3.10 Note: concerning 9040C temperatures.
  - Corrected Section 10.3.4.6 slope limits

- Revision 14, dated 30 June 2017
  - Annual review
  - Minor formatting changes throughout
  - Removed Section 10.3.13 due to redundancy
  - Changed title of Section 10.4 for clarity
  - Added new Section 10.4.6 to clarify procedure
  - Removed Section 10.5 due to mislabeling, renumbered subsequent sections

- Revision 13, dated 08 July 2016
  - Revised Sections 10.3.4.1 – 10.3.4.6 for clarity and to match the new logbook format with regard to the calibration procedure

- Revision 12, dated 21 April 2016
  - Annual review
  - Changed LIMS to TALS throughout
  - Minor grammar and formatting changes throughout
  - Added language to Section 6.1.1.1 referencing the master list for pH meter and electrode types
  - Added new Section 7.6 defining reagent water
  - Added language to Section 8.0, note 2 specifying that samples analyzed in the lab are flagged as out of hold
  - Corrected language in Section 9.2.2, requiring one duplicate per batch, rather than sample
  - Added language to Section 9.3.2 to clarify source of CCV
  - Reworded and added new subsections to Section 10.3.4 to clarify calibration procedure
  - Added slope requirement to Section 10.3.4.5
  - Added note to Section 10.3.5.2 regarding I.S. standard calibration
  - Restructured Section 10.6 and created new Section 10.7 to add maintenance information

- Revision 11, dated 13 October 2015
  - Added Section 3.2, reference to glossary in the QAM and DV-QA-003P
  - Updated Sections 7.2 and 7.3 to reflect daily replenishment of standards
  - Updated Section 9.1 to be consistent verbiage across SOPs as applicable
  - Added requirement to record pH result of calibration to Section 10.3.4.1
  - Updated Attachment 1 to reflect current pH logbook

- Revision 10, dated 14 January 2015

- Added procedure sections on use of autotitrator for pH determination and all related information regarding instrument throughout
- Added requirement to document calibration slope for manual pH
- Added example autotitration calibration report as new attachment 2
- Revised Section 11.1 to address both manual data and autotitrator data
- Renumbered attachments

- Revision 9, dated 31 July 2014
  - Updated table in Section 8 to include both SW846 and 40 CFR Part 136.3 and to reflect the requirements in each of these programs. Added footnote to table in Section 8 regarding preservation of samples.
  - Revised section 10.3.1.1 to remove requirement to record the calibration slope in the logbook. The assessment of the slope is not a method requirement and no acceptance criteria are provided in the methods.
  - Added Sections 10.5 and 10.6 for Maintenance and Troubleshooting.
  - Revised Section 11.1 to note that results are entered directly into TALS with no transcription.
  - Updated reference section to reflect requirements of 40 CFR Part 136.3.
  - Added method modification item 4 to address refrigeration of samples.
  - Updated logbook to remove Calibration Slope entry per Section 10.3.

- Revision 8, dated 31 January 2014
  - Removed section 9.4 and added 2012 MUR QC requirements to the appropriate sections
  - Added note to section 10.3 that IS is not appropriate for this method
  - Added section 10.4.10 to describe cleaning procedure for stir bars
  - Added statement to section 10.4.8 that data are entered directly into the LIMS
  - Added section 10.5 Maintenance
  - Added information to Sections 1.3 and 12.1 about MDL and RL used for reporting purposes

- Revision 7, dated 04 January 2013
  - Added section 9.4 for 2012 MUR QC requirements
- Revision 6, dated 27 July 2012
  - Revised Section 6 to identify replacement pH meter
  - Updated Section 7 to include second source ICV buffer at pH 7.0
  - Removed HCl and cleaning procedure for probe.
  - Revised Section 8 and added footnote
  - Moved procedural note in Section 4 to Section 10.4.5
  - Updated Sections 9.1, 10.1 and 10.2 to reflect current practice
  - Revised calibration procedure (Section 10)
  - Removed ASTM method reference and added SM 4500-H+ B-2000
  - Added Section 11.2 and removed Attachment
  - Updated Section 16
  - Added Attachments 1 and 2.
  - Source method review
  - Formatting and editorial changes throughout
- Revision 5.5, dated 30 December 2011
  - Annual Technical Review

- Revision 5.4, dated 17 January 2011
  - Annual Technical Review
  - Deleted Attachment 1
  - Updated Attachment 2 (now 1)
  - Added section 6.3, Computer Software and Hardware

Earlier revision histories have been archived and are available upon request.

Attachment 1

Example pH Calibration and Maintenance Log

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Denver

Calibration and Maintenance Log

Wet Chemistry / pH Probe

Daily Maintenance	Day	Sat	Sun	Mon	Tue	Wed	Thu	Fri
No maintenance required when instrument is not in use.	Date/Time:							
	Analyst:							
1) Inspect the probe for scratches or cracks.								
2) Probe solution refilled.								
3) Store probe in storage solution.								
4) Wipe off apparatus and clean up any spills.								
Calibration Standards:	2.0 Buffer Lot #: Expiration Date:							
	2.0 Buffer Calibration Reading							
	4.0 Buffer Lot #: Expiration Date:							
	4.0 Buffer Calibration Reading							
	7.0 Buffer Lot #: Expiration Date:							
	7.0 Buffer Calibration Reading							
	10.0 Buffer Lot #: Expiration Date:							
	10.0 Buffer Calibration Reading							
	12.0 Buffer Lot #: Expiration Date:							
	12.0 Buffer Calibration Reading							
Calibration Slope:								
Instrument Removed From Service: Y / N	**Initials:**	**Date:**	**Return to Service with Passing Calibration**				**Initials:**	**Date:**

Additional Maintenance/Comments: _____

_____

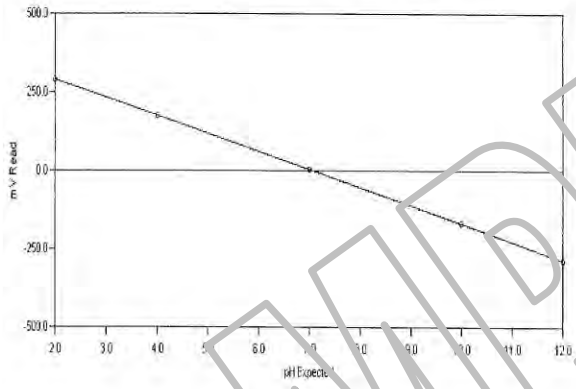
Attachment 2
Example Autotitrator pH Calibration Report

Report Date: 01/06/2015 ; 10:10 AM

PC-TitratION PLUS

Calibration Report

Calibration Record # 749



Calibration Settings

Calibration ID	PH	Date	01/06/2015
Channel	1	Time	10:07 AM
Probe Type	pH	Temperature	293.74 K 20.59 C
Probe ID	PH ELECTRODE	Analysis Type	Single Line Fit

Calibration Results

Slope	-57.810	Corr Coef.	1.0000
Intercept	3.074	Equation:	Y = (-57.810) X + (3.074)

Calibration Validity True Operator

	Result	Minimum	Maximum
Slope	57.810	-65.00	-53.00
Intercept	3.074	-100.00	100.00
Correlation Coefficient	1.0000	0.99	1.00

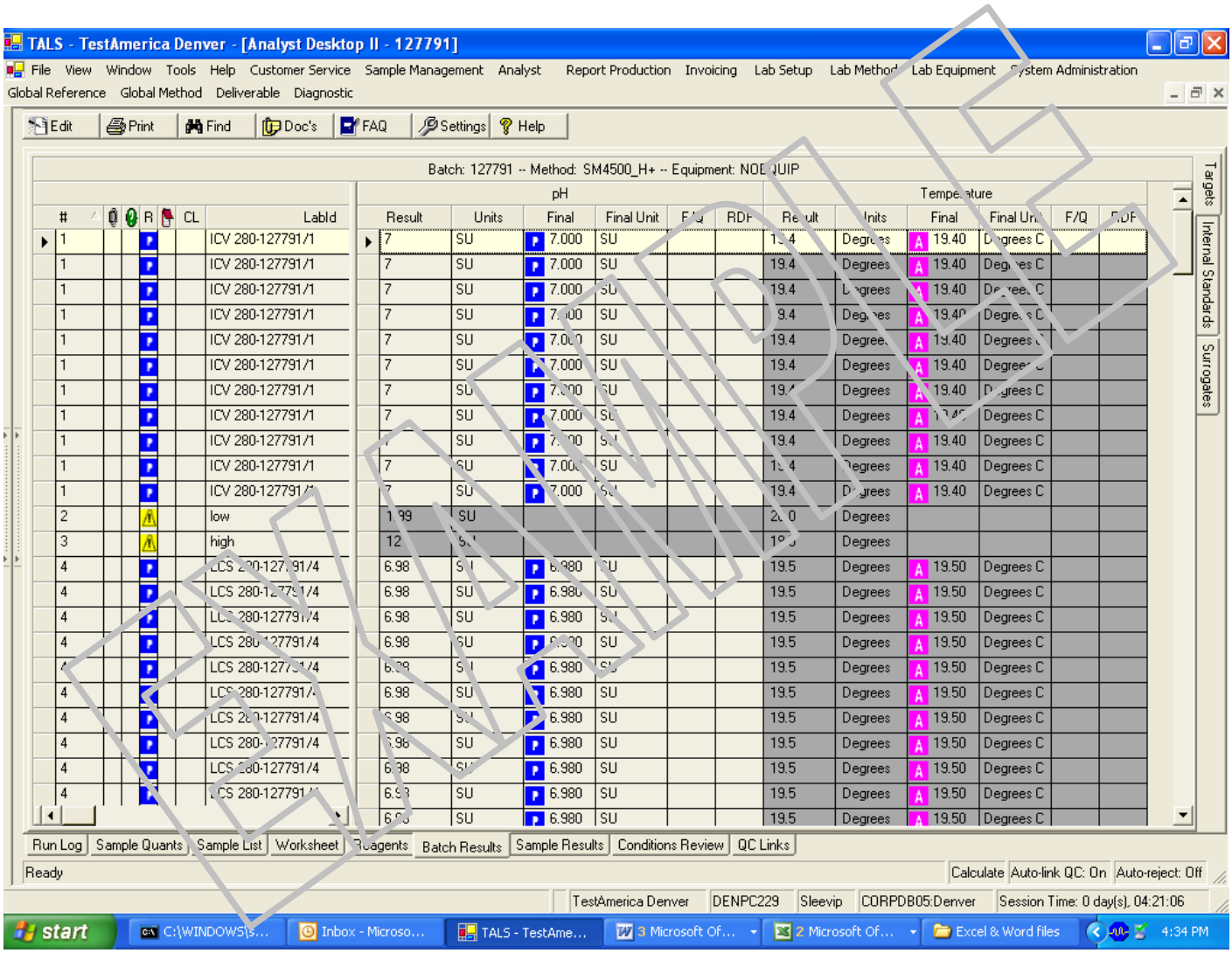
Note: "True" means the calibration was within the specified ranges
"False" means the calibration was NOT within the specified ranges

Calibration Data	Standard	Reading
	2.00	292.23
	4.00	175.65
	7.00	3.17
	10.00	-167.72
	12.00	-287.96

Page: 1 of 1
PC-TitratION PLUS by Man-Tech Associates, Inc.

CALRPRT.SRW

Attachment 3
Example Benchsheet



eurofins | Environment Testing TestAmerica

Eurofins TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

Eurofins TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Carbon in Soil (TOC, TC, TIC) [SW846 9060, 9060A]

Approvals:

Lauren Brousseau — 4/27/2020 — Date
Technical Specialist

Doug Skinner for RP — 4/27/20 — Date
Reed Pottruff
Health & Safety Manager / Coordinator

Roxanne Sullivan — Date
Quality Assurance Manager

Scott Hall — 4/27/20 — Date
Laboratory Director

Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This procedure describes the determination of total organic carbon or total carbon in soils, sludge, and sediments using the Shimadzu TOC-V TOC analyzer. The instrument uses 0.2 gram quantities of sample so results are less prone to precision problems that are typical of the trace TOC instruments that use sample aliquots in the 10-100 mg range. The methods referenced for this procedure are EPA Methods 9060 and 9060A.

1.2 The reporting limit (RL) is 0.4% carbon or 4,000 mg/kg, reported as Total Organic Carbon, Total Carbon or Total Inorganic Carbon.

2.0 Summary of Method

2.1 The sample is treated with 10% sulfuric acid to drive off inorganic carbonates and then dried at 105°C (+/- 2°C) to remove moisture and acid. Organic carbon in the sample is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed is measured by a non dispersive infrared detector (NDIR). The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

2.2 If Total Carbon is to be determined, the sample is not treated with sulfuric acid and the amount of carbon in the sample is determined on the dried sample.

2.3 If Total Inorganic Carbon is to be determined the sample is analyzed for both TOC and TC. The TIC result is the difference of the two concentrations.

3.0 Definitions

3.1 **Total Organic Carbon (TOC):** The carbon measured as a result of oxidation of the sample after the removal of inorganic carbon.

3.2 **Total Carbon (TC):** The carbon measured as a result of oxidation of the dried sample.

3.3 **Total Inorganic Carbon (TIC):** The carbon calculated as the difference in the TC and TOC results.

3.4 **Reagent Water:** Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

4.0 Interferences

Oily samples will cause erratic results. This is minimized by homogenization of the sample.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Spent crucibles must be allowed to cool to room temperature prior to disposal.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive	1 Mg/M3 (TWA)	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Vanadium Pentoxide	Health Hazard	0.05 mg/m3 (TWA)	Very hazardous in case of ingestion or inhalation, Skin irritant.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit. TWA – Time Weighted Average			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Shimadzu TOC-V analyzer, consisting of Control V software, ver 2.00

6.1.2 Shimadzu Solid Sample Module (SSM-5000A), consisting of

6.1.2.1 Platinum/cobalt catalyst

6.1.2.2 Combustion tube

6.1.2.3 Sulfide scrubber, “mistcatcher”

6.1.3 Drying oven, capable of maintaining a temperature of 105 ^{o}C.

6.2 Supplies

6.2.1 Porcelain Combustion Boats (residue is removed with brush before reuse)

NOTE: Porcelain Combustion Boats are a radioactive material.

6.2.2 Weighing tins

6.2.3 Spoons or spatulas

6.2.4 Ceramic fiber.

6.2.5 Pipettes

6.2.6 Analytical balance capable of weighing to 0.0001 g

6.2.7 Muffle oven set at 550-600^{o}C.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software, and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 The reagents listed in this section must be reagent grade or higher quality.

7.2 Concentrated sulfuric acid

7.3 10% sulfuric acid

Add 100 mL of concentrated sulfuric acid to 1000 mL of reagent water. Allow to cool before use.

7.4 TOC Calibration Standard Intermediates: Used for initial calibration, continuing calibration verification, matrix spikes, and matrix spike duplicates. Stability is for 1 year.

7.4.1 10% Dextrose solution – 5 g dextrose and bring to a final volume of 50 mL with DI water

7.4.2 5% Dextrose solution – 2.5 g dextrose and bring to a final volume of 50 mL with DI water

7.4.3 1% Dextrose solution – 5 mL of the 10% dextrose intermediate to a final volume of 50 mL.

7.4.4 0.1% Dextrose solution – 5 mL of the 1% dextrose solution to a final volume of 50 mL.

7.4.5 Calibration curve

mg/kg C (per 100mg)	µg C (Abs C)	Volume (uL)	Dextrose %	Carbon %
0	0	0	0	0
100	10	25	0.1	0.04
1000	100	25	1	0.4
10000	1000	25	10	4

7.5 **TOC Initial Calibration Verification:** This standard is from a different source than the TOC Calibration Standard described in Section 7.4.

7.5.1 Using the secondary dextrose standard, weigh 0.5g dextrose and bring to a final volume of 50mL with DI water. Stability is 1 year.

7.5.1.1 Using the 1% dextrose standard, pipette 25uL into the porcelain boat, yielding 1000 mg/Kg C.

7.6 **TOC LCS Standard (STOC LCS Std)**

This standard is purchased from an outside vendor. The true value will be dependent on the lot received.

7.7 **Total Carbon Standards**

Determination of Total Carbon uses the same standards as TOC, described in Sections 7.4-7.6.

7.8 Hengar Boiling stones (for use as solid matrix)

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	4 oz	Cool, ≤ 6 °C	28 days	SW-846

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the

TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

A batch is a group of no greater than 20 samples excluding QC samples (LCS, MS, MSD, Method Blank), which are processed similarly, with respect to the procedure. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.3 Method Blank (MB)

9.3.1 One method blank (MB) must be processed with each batch. The method blank consists of a solid blank matrix (boiling stones) carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system or process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.3.2 The method blank is prepared by weighing approximately 0.10 g of the blank solid matrix (Section 7.8). Due to the uneven nature of the Hengar boiling stones used for the blank matrix, the mass used in the blank may vary considerably.

Acceptance Criteria: The method blank should not contain any analyte of interest at or above the reporting limit. Some programs, e.g., DoD, require the concentration of the analyte in the method blank to be less than one-half the reporting limit.

Corrective Action: If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be taken in consultation with the client and must be addressed in the project narrative.

If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the project narrative.

If all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the elevated blank value.

9.4 Laboratory Control Sample (LCS)

9.4.1 One LCS must be analyzed with each batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.4.2 The soil LCS is performed by analyzing approximately 0.10 g of a purchased standard that has gone through the sample preparation described in Section 10.4.

Acceptance Criteria: Control limits are provided by the vendor and are maintained in TALS.

Corrective Action: If any analyte is outside established control limits, the system is out of control, and corrective action must occur. Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Samples

9.5.1 One MS/MSD pair must be processed for every 10 samples. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) that is prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to an MS/MSD pair. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked.

9.5.2 The MS and MSD are prepared by placing 0.10 g of the dried soil sample to be spiked into a porcelain boat and adding 25uL of the 14% dextrose solution onto a small piece of prebaked ceramic fiber.

Acceptance Criteria: The recovery of the analyte in the MS and MSD must fall within established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference between the MS and MSD must be no greater than the established RPD limit, which is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM 5 limits apply to projects performed under this program.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.

- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client’s needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.6 Initial Calibration Verification (ICV)

9.6.1 The ICV standard is analyzed immediately following the ICAL.

9.6.2 The ICV is a second-source 1% dextrose solution., 25uL into a small amount of ceramic fiber. TV 1000 mg/Kg

Acceptance Criteria: The analyte recovery must fall within the 90-110% range.

Corrective Action: If the analyte recovery is outside the acceptance limits, check the equipment and standards, correct any problems, and then recalibrate.

9.7 Continuing Calibration Verification (CCV)

9.7.1 The calibration is checked at the beginning of an analytical sequence (ICV), after every ten samples (CCV), and at the end of the sequence (CCV) by measuring a CCV standard.

9.7.2 The CCV is the same as the ICV (9.6.2)

Acceptance Criteria: The CCV recovery must be within the 90-110% range.

Corrective Action: If the analyte recovery is outside the acceptance limits, check the equipment and standards, correct any problems, recalibrate, and rerun all samples analyzed since the last successful CCV.

9.8 Initial and Continuing Calibration Blank (ICB and CCB)

9.8.1 System cleanliness is checked at the beginning of an analytical sequence (ICB), after every ten samples (CCB), and at the end of the sequence (CCB).

9.8.2 The ICB/CCB is 25 ul DI water added to the ceramic fiber.

Acceptance Criteria: Results must be less than the reporting limit.

Corrective Action: If the blank result is greater than the reporting limit, check for carry-over from high level samples, clean the system, recalibrate, and rerun all samples analyzed since the last successful CCB.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Boat Preparation

10.3.1 Put dirty boats in a wide mouth 250 mL poly container or another suitable container.

10.3.2 Fill container with DI water, leaving room to add approximately 40 mL of concentrated H_2SO_4.

10.3.3 Fill remaining space with DI water.

10.3.4 Secure the lid and sonicate for at least 1 hr.

10.3.5 Pour out the water into an acid waste container and put boats in a tub.

10.3.6 Scrub each boat with a small piece of heavy duty scouring pad, until all solids have been removed.

10.3.7 Rinse boats first with tap water and then with DI water.

10.3.8 Place boats in muffle at 550^{o}C for at least 1 hr.

10.3.9 Cool in dessicator and then transfer to an airtight plastic container.

10.4 Sample Preparation – Total Organic Carbon

10.4.1 Samples should be homogenized, and ground to uniform consistency if necessary. Leave out any extraneous artifacts, i.e., glass shards, large twigs, leaves, etc. See SOP DV-QA-0023 for more information on proper subsampling procedures.

10.4.2 In an aluminum drying dish, aliquot approximately 2 - 3 g of sample. Dry the sample at least 6 hrs.

10.4.3 Weigh 0.20 g (+/-0.02 g) sample into a porcelain TOC boat that has been baked in a muffle. Less may be used in the analyst deems necessary, based on historical information or physical observance of the sample.

10.4.4 Add 10% H_2SO_4 to the sample drop wise until the sample is completely moistened and the effervescence stops.

10.4.5 Dry the samples in a 105°C oven for 1 hr or until dry again.

10.4.6 Sprinkle a small amount of vanadium pentoxide onto each sample to prevent buildup in the combustion tube.

10.5 Sample Preparation – Total Carbon

10.5.1 Samples should be homogenized, and ground to uniform consistency if necessary. Leave out any extraneous artifacts, i.e., glass shards, large twigs, leaves, etc. See SOP DV-QA-0023 for more information on proper subsampling procedures.

10.5.2 In an aluminum drying dish, aliquot approximately 2 - 3 g of sample. Dry the sample at least 6 hrs.

10.5.3 Weigh 0.10 g (+/-0.01 g) sample into a porcelain TOC boat that has been baked in a muffle. Less may be used in the analyst deems necessary, based on historical information or physical observance of the sample.

10.5.4 Sprinkle a small amount of vanadium pentoxide onto each sample to prevent buildup in the combustion tube.

10.6 Sample Preparation – Total Inorganic Carbon

The sample must be analyzed for both Total Carbon and Total Organic Carbon. Total Inorganic Carbon is calculated from these results. See Section 11.8.

10.7 Sample Analysis

10.7.1 Power on both the TOC-V and SSM units, and turn on both the compressed air and oxygen tanks.

10.7.2 Select the TOC-Control V icon to open the data acquisition program. Select 'New', and 'Test America SSM' and then press 'Connect' to initialize the system.

10.7.3 The oven will take about 40-50 min to reach 900°C.

10.7.4 Display the Calibration Curve tab in the file viewer, select the proper file and 'drag' it over to the Sample Editor window.

10.7.5 Click on 'Insert' and click on 'Insert-Sample', click 'next' until you can change the units to ppm, and then click 'Finish'.

10.7.6 Prepare the appropriate QC samples (LCS, MB, MS/MSD, CCV and CCB). For TOC determination, the method blank and the LCS are treated with H_2SO_4 along with all the samples in the batch. The standards are not prepared. For TC no acid treatment is applied to the QC samples.

10.7.7 Run a CCV/CCB pair every 10 samples. A typical analysis sequence is:

Initial calibration curve (minimum 5 standards)

ICV
ICB
LCS
LCSD
MB
10 or fewer samples (including MS/MSD)
CCV
CCB
10 or fewer samples (including MS/MSD)
CCV
CCB

10.7.8 Once the instrument is ready, click 'Start' and type the weight (for the calibration standards only) of the sample in the dialog box and click 'Start' again.

10.7.9 If reduced aliquots are used, a sample weight of 0.10 g must be entered and the sample ID should contain the dilution factor (i.e. 12345-a-1 @10) in order the LIMS to properly adjust the reporting limit. An NCM must be generated to explain this.

10.7.10 Open the sample port cover and place the sample boar and sample on the sample boat transfer plate.

10.7.11 Liquid standards must be injected onto a small amount of prebaked ceramic fiber. This includes the matrix spikes.

10.7.12 Close the sample port cover and wait at least 90 seconds to remove any atmospheric carbon from the system.

10.7.13 Once the SSM Measurement window is displayed, push the sample boat into the furnace to the measuring position.

10.7.14 When the measurement is complete, the SSM Measurement window will pop up again and indicate that the sample boat can be pulled back to the cooling position.

10.7.15 The TOC Measurement dialog box will pop up. Click 'Repeat' to perform another injection of the same sample, 'Next' to accept the data and move on to the next sample, 'Stop' to end the measurement process.

10.8 Instrument Shutdown

10.8.1 Click 'Shutdown'

10.8.2 Select 'Shut down instrument' and click 'OK'

10.8.3 Open the sample port cover during the cool down process to prevent water from condensing into the sample line.

10.8.4 Approximately 30 min after beginning the shutdown procedure, the SSM-5000A can be turned off. The TOC-V will shut off automatically.

10.8.5 Shut off both the compressed air and oxygen tanks.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 Data Upload and Processing

11.2.1 TOC

Send the instrument data file to TALS via the Z:\ directory shortcut accessible through file explorer.

11.2.2 TC

The TC results are obtained through direct analysis of a duplicate sample aliquot that has not been acidified prior to analysis (see Section 10.4). The TOC and TC analyses are typically analyzed in the same sample sequence. All TC results are differentiated from the TOC results by entering the abbreviation “TC” into the instrument sequence table in the “Description” column (see Attachment 3).

11.2.3 TIC

The TIC result is calculated offline by entering the TC result as percent carbon from the analytical run and the TOC results from TALS into the spreadsheet found at R:\\QA\Edit\Forms\Wet Chemistry\9060_TC_TIC Calculation Spreadsheet_Rev 1 (see Attachment 4). The spreadsheet will output the TIC result in g/kg, which must then be entered into the Results column of a calculation batch in TALS (under method 9060_TC_TIC) in order to report the final result. See WI-DV-0092 for more information.

11.3 Calculating C from Dextrose ($C_6H_{12}O_6$)

% C in 1 ml Dextrose

C-> 12 g/mol x 6 = 72 g/mol
H-> 1 g/mol x 12 = 12 g/mol
O-> 16 g/mol x6 = 96 g/mol
$C_6H_{12}O_6$ = 180 g/mol
C = 40% $C_6H_{12}O_6$

11.4 Concentration of C in calibration standard intermediates

0.1% $C_6H_{12}O_6$ x 40% C (per $C_6H_{12}O_6$) = 0.04% C x 10,000 ppm (per%) = 400 ppm C

1.0% $C_6H_{12}O_6$ x 40% C (per $C_6H_{12}O_6$) = 0.4% C x 10,000 ppm (per%) = 4000 ppm C

10% $C_6H_{12}O_6$ x 40% C (per $C_6H_{12}O_6$) = 4% C x 10,000 ppm (per%) = 40,000 ppm C

11.5 Absolute value of C in calibration standards

0.025 mL x 400 ug/mL = 10 ug C
0.025 mL x 4000 ug/mL = 100 ug C
0.025 mL x 40,000 ug/mL = 1000 ug C

* 0.025 mL is the amount spiked of the intermediates

11.6 Concentration of C in calibration standards

10 ug C / 100 mg = 0.1 ug/mg = 100 mg/Kg
100 ug C / 100 mg = 1 ug/mg = 1000 mg/Kg
1000 ug C / 100 mg = 10 ug/mg = 10,000 mg/Kg

*100 mg is the nominal weight of samples

11.7 Sample concentration of TOC or TC in either mg/kg or g/kg is calculated using Equation 1.

$$Sample\ Concentration = C \times F \qquad \textit{Equation 1}$$

Where:

C = percent carbon in sample (determined by instrument)
F = conversion factor for concentration
F = 10,000 for conversion to mg/kg
F = 10 for conversion to g/kg

NOTE: Since sample weights are determined on the dried sample, there is no dry-weight correction for TOC or TC in soil.

NOTE: Determination of TC or TOC is dependent upon the sample preparation. See Sections 10.4 and 10.5.

11.8 Sample concentration of TIC is calculated using Equation 2.

$$TIC\ Sample\ Concentration = TC\ concentration - TOC\ concentration \qquad \textit{Equation 2}$$

11.9 Accuracy

11.9.1 Percent recovery for the ICV, CCV or LCS is calculated using Equation 3.

$$\%\ Recovery = \frac{observed\ concentration}{known\ concentration} \times 100 \qquad \textit{Equation 3}$$

11.9.2 Matrix spike and Matrix spike duplicate recoveries are calculated using Equation 4.

$$MS\ or\ MSD\ \%\ Recovery = \left(\frac{SSR - SR}{SA}\right) \times 100\% \qquad \textit{Equation 4}$$

Where:

SSR = observed concentration in spiked sample
SR = observed concentration in unspiked sample
SA = concentration of spike added to sample

11.10 Precision (RPD)

$$RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2} \times 100 \qquad \textit{Equation 5}$$

Where:

R_1 = Measured concentration for first sample
R_2 = Measured concentration for second sample

11.11 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Acidic waste – Waste Stream F

14.2.3 Porcelain Combustion Boats - Contact Radioactive Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 9060A, Total Organic Carbon, Revision 1, November 2004.

15.1.2 Method 9060, Total Organic Carbon, Revision 0, September 1986.

15.2 Shimadzu Manual, TOC-V CPH/CPN Total Organic Carbon Analyzer (For TOC-Control V ver. 2)

15.3 Shimadzu Manual, TOC-V Series, SSM-5000A

16.0 Method Modifications:

Item	Method	Modification
1	SW 9060 SW 9060A	Methods 9060 and 9060A are designed for water samples, and require quadruplicate analysis to overcome potential precision problems. This procedure is exclusively for soil samples, and the LECO instrument is designed for soil analysis. The sample aliquots are 10-100 times larger than are practical with most other

Item	Method	Modification
		non-dispersive IR instruments, and so the precision is acceptable with single analyses.
2	SW 9060 SW 9060A	Methods 9060 and 9060A require the use of a blender to homogenize samples. Since this procedure is for soils, the samples are ground to a uniform consistency prior to treatment with acid.
3	SW 9060 SW 9060A	Methods 9060 and 9060A are adapted to the determination of Total Organic Carbon in solid samples. The removal of inorganic carbon from the solid sample is accomplished by the addition of 6 N HCl to an aliquot of the sample that exceeds the amount needed for the determinative step by at least 10-fold.
4	SW 9060 SW 9060A	Solid samples are only preserved by refrigeration prior to analysis.
5	SW 9060 SW 9060A	The source methods use a carbonate-bicarbonate standard. This method uses a high purity calcium carbonate standard.

17.0 Attachments

Attachment 1: Example Bench Sheet

18.0 Revision History

- Revision 10, dated 4 May, 2020
  - Added/removed the information necessary for the change in instruments.
- Revision 9, dated 31 May 2017
  - Annual Review
  - Minor formatting changes throughout
  - Added new Section 3.4 defining reagent water
  - Corrected language in Section 6.1.2 for clarity
  - Added new Sections 10.6.5.3 – 10.6.5.8 to describe ICAL spreadsheet
  - Added section 10.7.2 to require proper labeling of TC and TOC in raw data
  - Added new Section 11.2 and subsections to explain TOC/TC/TIC data handling
  - Updated Attachment 1 to reflect new calibration curve spreadsheet
  - Added new Attachment 2 example calibration raw data sheet
  - Added new Attachment 3 raw data sheet
  - Added new Attachment 4 example TIC calculation spreadsheet
- Revision 8, dated 31 May 2016
  - Annual Review
  - Minor formatting and language corrections throughout
  - Changed MSDS to SDS in Section 5.4
  - Reworded Sections 7.4 and 7.5 for clarity
  - Changed weight requirement for blank in Section 9.3.2, added explanation for higher weight
  - Added weight range to MS/MSD prep in Section 9.5.2
  - Added corrective actions to Section 9.5.2 to reflect current policy
  - Adding reference to subsampling SOP in Sections 10.3.1 and 10.4.1
  - Change sample aliquot weight to a weight range in Section 10.7.1
  - Added information to Section 11.6 regarding data review
  - Added new analysts training requirement to Section 12.3

- Revision 7, dated 31 May 2015
  - Annual Technical Review
  - Added Antimony to section 6.2.4.2
  - Updated section 9.1 to include DoD criteria and standardized SOP verbiage
  - Added maintenance information to section 10.8
  - Archived all revision histories prior to 2011

- Revision 6, dated 31 May 2014
  - Added section 10.8 Troubleshooting

- Revision 5, dated 14 May 2013
  - Added detail on determination as Total Carbon and Total Inorganic Carbon, throughout
  - Added use of hotplate overnight to dry samples (Sections 10.3.3 and 10.4.2)
  - Changed HT reference to SW-846

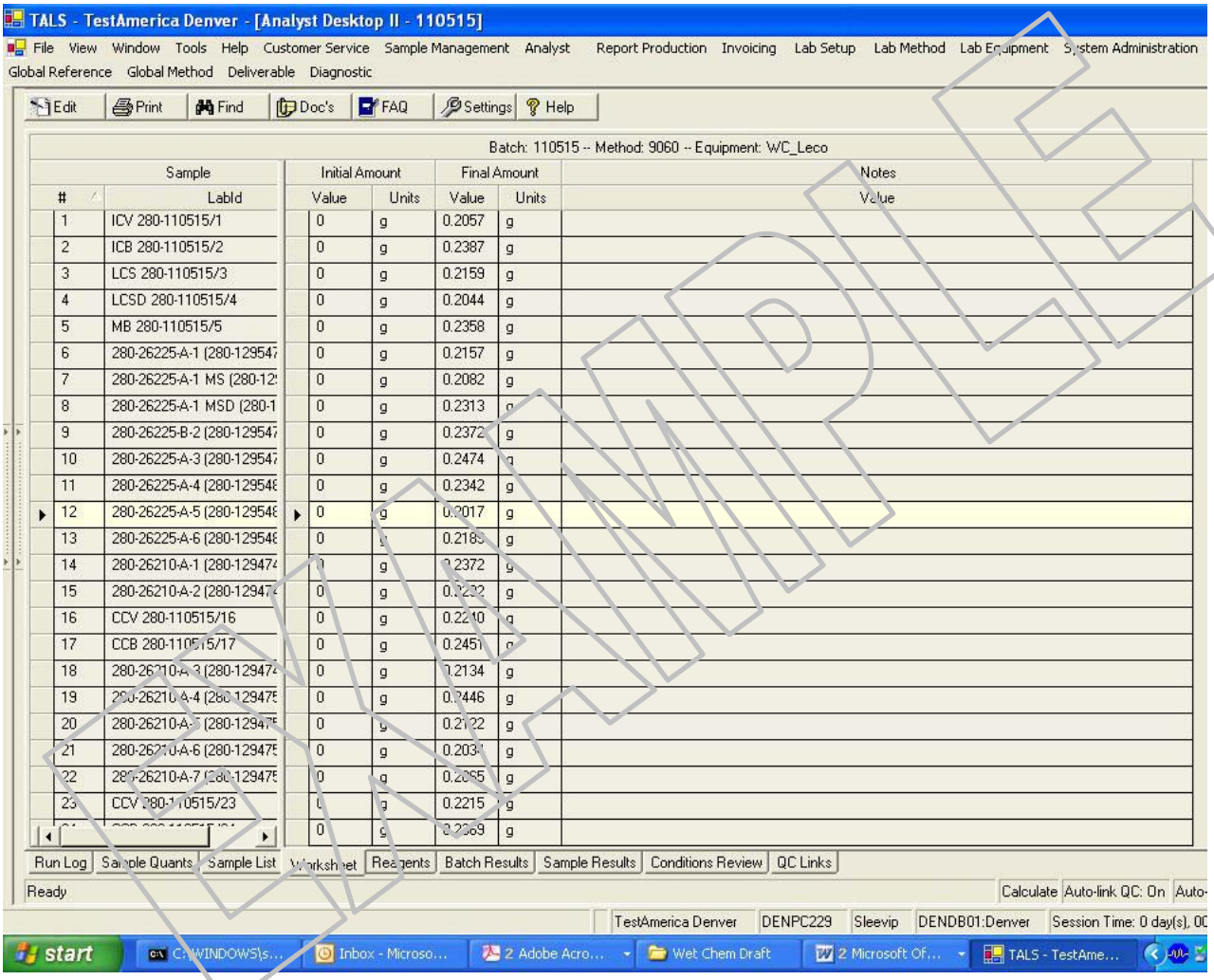
- Revision 4, dated 13 July 2012
  - Updated reporting limit and method references in section 1
  - Clarified frequency of MS/MSD required by method (section 9.5)
  - Added example analysis sequence in Section 10.5
  - Corrected equation for calculation of TOC concentration in soil samples and revised note about dry weight since samples are dried before weighing for analysis. (Section 11)
  - Removed data review checklist and added reference to data review SOP in Section 11.11
  - Removed reference to control limits stored in LIMS in Section 11. Already specified in Section 9.
  - Removed paragraph on when to report ND; covered by RL SOP.
  - Removed redundant reference to corporate calibration SOP in section 11.
  - Revised MDL discussion in Section 12 to general description including statement that MDLs are updated at least annually unless method or program requirements require greater frequency.
  - Deleted second paragraph under section 12.3. IDOC already described in section 12.2.
  - Revised Section 16 including addition of items 3-5.
  - Added example spreadsheet for calibration and bench sheet from TALS as attachments.
  - Source method review
  - Formatting and grammatical changes throughout

- Revision 3.2, dated 31 May 2011
  - Annual Review
  - Updated Attachment 1

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Example Bench Sheet



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Flash Point by Automatic Pensky-Martens Closed Cup Apparatus

[SW 1010, SW 1010A, ASTM D93]

Approvals (Signature/Date):

Connie Jewell 11/8/18 Technical Specialist Date	Doug Gomer 11/8/18 Health & Safety Manager / Coordinator Date
Roxanne Sullivan 11/8/18 Quality Assurance Manager Date	Richard Clinkscales 11/8/18 Laboratory Director Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure is used for the determination of the flash point of liquid wastes using an automatic Pensky-Martens closed-cup tester, which complies with SW-846 Methods 1010 and 1010A and ASTM D93-80. The flash point is used in shipping and safety regulations to define "flammable" and "combustible" materials, and to classify wastes in accordance with hazardous waste regulations (40 CFR Part 261.21).

1.2 This procedure is applicable to fuel oils, lube oils, liquid wastes, suspensions of fine-grained solids in a liquid, and liquids that tend to form a surface film under test conditions. This procedure may be applied to other liquid wastes that meet the physical requirements specified in ASTM D93. In all cases, the liquid must be easily stirred by the testing apparatus and must not contain large solid particles that could interfere with the stirring mechanism. Per 40 CFR Part 261.21 definitions, the ignitability of solid samples is not determined by this procedure.

1.3 The tester currently used in the laboratory is not equipped with cooling capability, which theoretically limits the applicability of the test to samples with flash points above 100 °F. Correcting for the air pressure at the altitude of the laboratory, the lowest flash point that can be accurately determined in compliance with ASTM D93 is approximately 107 °F. Samples are typically heated to a maximum of 160 °F. Samples that do not flash below this upper limit are reported as having a flash point greater than 160 °F.

1.4 The approximate analytical time is between 30 and 60 minutes per sample, including preparation and clean up.

2.0 Summary of Method

2.1 Flash point measures the tendency of a sample to form a flammable mixture with air under controlled laboratory conditions. Flash point measurement is an empirical method that relies on strict adherence to the standard method and specified apparatus. The test cup is filled with sample to a marked level. The sample is heated at a slow, constant rate with continual stirring. A small flame or electric igniter is dipped into the cup just above the liquid level at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature of the liquid at which application of the test flame causes the vapor above the sample to ignite.

2.2 For this particular SOP, an automatic flash point analyzer is used. A flash point determination is accomplished by filling the test cup with sample material, placing it in the heating block, and initiating the preprogrammed analysis. The heating of the sample is controlled at the specified rate and the igniter is activated and dipped at the correct temperature and frequency to comply with Methods 1010 and 1010A (ASTM D93-80). The accuracy of the analyzer is verified by testing a reference standard and a water blank.

3.0 Definitions

3.1 **Flash Point** - The lowest temperature corrected to a barometric pressure of 760 mm Hg, at which application of a test flame or heated coil causes the vapor of a specimen to ignite under specified conditions of test. The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample.

3.2 **Ignitability** - One of the characteristics defined in the Federal hazardous waste regulations (40 CFR Part 261.21). A liquid waste, other than an aqueous solution containing less than 24 percent alcohol by volume, that has a flash point less than 60 °C (140 °F) as determined by this method is classified as a hazardous waste, a D001 listed waste.

3.3 **Flammability** - In contrast to ignitability, flammability is defined by Federal regulations as applying to a liquid with a flash point under 100 °F.

3.4 Refer to the Glossary of the TestAmerica Denver *Quality Assurance Manual* (QAM), and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample. Occasionally, the application of the test flame will cause a blue or green halo or an enlarged flame. This generally occurs near the actual flash point but in some cases, especially with halogenated hydrocarbons and admixtures, can occur at any temperature. These phenomena are not to be considered true flash points.

4.2 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material from samples. Containers should not be opened unnecessarily or for long periods of time unless the temperature of the sample is at least the equivalent of 18 °F below the expected flash point. In addition, volatile materials can be lost from leaky containers or may diffuse through plastic containers.

4.3 ASTM D93 requires that samples are continually stirred during the test. Samples that contain large particles that interfere with the stirring mechanism or that can lodge between the mixer blades and the temperature probe and possibly break the probe should not be subjected to this procedure. Likewise, viscous liquids that impede the stirring mechanism should not be subjected to this procedure.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual, and this SOP.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses nitrile gloves, lab coats and closed-toe nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1 (as per the Environmental Health and Safety Manual), laboratory coat, and nitrile gloves must be worn when handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately. VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be worn when other solvents are handled.

NOTE: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.

5.3.1 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers must be kept closed unless transfers are being made.

5.3.2 The flashpoint standard is highly flammable and harmful if inhaled. Keep away from heat, sparks, and open flame. Use in a hood only. Store in a cabinet designed for flammable solvents.

5.3.3 The operator must take particular care during the initial application of the test flame, since samples containing low-flash materials can react vigorously when the test flame is first applied.

5.3.4 Operation of the flash point tester without the temperature probe in place in the cup cover is a safety hazard. It could result in ignition of vapors outside of the instrument and a possible flame or explosion.

5.4 Primary Materials Used

The following is a list of the materials used in this method that have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
PMCC D-93	Flammable Irritant	100 ppm – TWA	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears, and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns, and eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Pensky-Martens closed-cup tester with stirrer and stirring motor that complies with ASTM D93A or ASTM D93B, e.g., Herzog Automatic Flash Point Tester Optiflash. The flash point tester should be located and operated in a properly vented hood.

6.2 Thermometer made for the Pensky-Martens tester, operable in the 60 – 160 °F range. (See SOP DV-QA-0001 for calibration verification requirements for thermometers.)

6.3 USB flash drive to upload data form instrument.

6.4 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 PMCC D-93 reference standard, minimum 95% purity.

7.3 Reagent Water: Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples are to be collected in glass jars with a minimum of headspace and stored at 0 - 6 °C. Samples collected in plastic containers are not valid due to possible diffusion of volatile materials through the container walls.

8.2 A minimum sample volume of 70 mL is needed to fill the test cup to the correct level. If a sample duplicate is analyzed, then a total volume of at least 140 mL is required. To provide sufficient volume for contingencies and reruns, a sample volume of at least 280 mL (i.e., four aliquots) is strongly recommended.

8.3 Samples are not chemically preserved.

8.4 There is no holding time requirement.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Instrument QC

9.2.1 The automatic flash point tester is set up and operated in accordance with the OptiFlash user manual. The user manual can be found in G:\Wetchem\ OptiFlash User Manual PM_V1_0D_EN. Any problems with the instrument should be recorded in the instrument maintenance logbook, including any action taken to correct the problem. The tester must be configured so that the igniter is dipped over the entire working temperature range, i.e., approximately room temperature to 160 °F, unless the sample flashes before 160 °F is reached or the expected flash point is known. In the former case, the heating program may be terminated after the sample has flashed. In the latter case, the tester may be operated over a narrower range by setting the expected flash point temperature and starting the igniter dipping at 20 °F below the expected flashpoint.

9.2.2 Turn on the instrument, and follow the operation instructions in the user manual. ASTM D93 requires that all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument must be followed.

9.2.3 The heating block, cup, and sample temperature should not be higher than 20 degrees below the expected flash point at the beginning of the analysis. For example, the expected flash point of the PMCC D-93 reference standard is 140 °F at sea level and approximately 134 °F at the laboratory location. Therefore, the heating block and test cup temperature should be at or below 80 °F before starting the test. The lowest achievable temperature is dictated by the ambient room temperature, which is typically 68 to 85 °F. Between tests, the heating block is cooled to a temperature of approximately 80 °F, further limiting the test to samples with flash points above 100 °F. In actual practice, the laboratory is able to determine the flash point of PMCC D-93 within limits prescribed in ASTM D93 by immediately starting the igniter dipping. Historical data for the PMCC D-93 reference standard exhibit a 1.5 % positive bias (i.e, 1.2 °F).

9.3 Reference Standard Analysis (LCS)

Prior to using the tester for each day's sample testing, determine the flash point of the PMCC D-93 reference standard following the instructions in Section 10.4. Repeat this determination after testing all samples. This sample serves as the laboratory control sample (LCS).

Acceptance Criteria: When the tester is operating properly, a value of 140 ± 2 °F should be obtained. The limits are entered as percent recovery (%R) in TALS, i.e., 98 – 102 %R.

Corrective Action: If the flash point is not within limits, consult the user manual for troubleshooting instructions. An automatic tester with a computer interface should include programmed diagnostic checks to help identify instrument problems. In general, the following components of the tester should be evaluated for proper operation:

- Igniter filament coil should not be crushed or show signs of mechanical or physical damage.
- Electrical connections for the igniter and detector should be tight and secure.
- Shutter and stirring motor should operate properly.
- Sample cup, cover, and stirring mechanism should be clean.

If the flash point is still not acceptable, stop analysis, and consult with the user manual for further troubleshooting.

9.4 Blank Analysis

Prior to using the tester for each day's sample testing, test a sample of reagent water or equivalent following the instructions in Section 10.4. Repeat this determination after testing all samples. If two successive samples in a row exhibit a flash point within the test range, then perform the blank analysis and repeat the analysis of the second sample to confirm the flash point.

Acceptance Criteria: The water sample should not flash.

Corrective Action: If a flash is observed, ensure that the test cup is clean and free of any residual flammable solvent. Repeat the analysis after cleaning the test cup. If a flash is still recorded for the blank, consult the user manual for diagnostic testing. No samples may be analyzed without a passing blank result.

9.5 Sample Duplicate Analysis

Sample duplicates are performed at a minimum frequency of one per batch of twenty or fewer samples. The sample selected for the duplicate must produce a detectable flash in the working temperature range. If there is insufficient sample volume to perform a duplicate or none of the samples in the batch flashes, a duplicate is not tested. Whenever a duplicate is not tested the reason must be documented in an NCM.

Acceptance Criteria: If the sample flashes within the working range, successive results obtained by the same operator should agree within 4 °F (per ASTM D 93, repeatability data).

Corrective Action: If this criterion is not met, follow the corrective actions listed in Section 9.3 and repeat the test once more. If it still fails, note the QC failure in a nonconformance memo (NCM). If there is insufficient sample remaining for the reanalysis, document in an NCM.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described

10.3 Sample Preparation

10.3.1 Examine the sample container. If it is plastic or shows evidence of leaking, record this information on the TALS worksheet and prepare an NCM.

10.3.2 Record visual observations about the nature of the sample on the worksheet. (e.g., liquid, viscous oil, slurry, etc.).

10.3.3 If the sample contains large particles that can interfere with the stirring mechanism or if the sample is too viscous to be easily stirred, it should not be tested using this method. Inform the PM that the sample should not be tested for flash point, but could be tested for ignitability instead.

10.4 Sample Testing

NOTE: Instrument-specific instructions in this section are based on the user manual for the Herzog OptiFlash Automatic Flash Point Analyzer. For more detailed instructions, including instructions for responding to instrument error messages, consult the user manual.

10.4.1 Turn on the instrument and initiate the analysis.

10.4.2 Press the corresponding field on the touchscreen and enter the required test parameters:

- Operator identification
- Sample ID
- Product name (ASTM D93 pre-programmed test)
- expected FP (Flash Point) temperature

10.1.1 Select the desired pre-programmed test and adjust settings as needed for specific samples according to the user manual. Instrument parameters include start and end points defined as a temperature interval relative to

a detected flash point, heating rates, stirring speeds, igniter dipping intervals, and ignition power levels.

10.1.1.1 Specific to the Herzog model OptiFlash, most samples are analyzed as unknowns using Program ASTM D93. This program has been configured to dip the igniter, and thus test for the flash point, from approximately room temperature to 160 °F, while maintaining ASTM D93 specified heating rates, dipping intervals, and stirring speeds.

10.1.1.2 Record the appropriate information in the instrument maintenance log on the day samples are analyzed. Any problems with the instrument should be recorded in the instrument maintenance logbook, including the action taken to correct the problem.

10.1.2 Inspect all parts of the test cup and accessories to be sure that they are clean and dry.

10.1.2.1 The test cup must be thoroughly cleaned if there are visible stains or discoloration on the surfaces of the cup and cover. If there are stains on the cup, the cup should be cleaned with an abrasive pad to remove the discoloration.

10.1.2.2 In between tests, at a minimum, the cup is emptied, rinsed with reagent water, and wiped dry. If noticeable discoloration remains, then an abrasive pad is used to further clean the cup.

10.1.2.3 A solvent may be used to remove oily residue, but the cup must be allowed to dry completely so that there is no trace of the solvent when the cup is used for testing.

10.1.3 Gently shake the sample bottle to ensure that the sample is thoroughly mixed.

10.1.4 Carefully pour the sample into the test cup to the mark (approximately 70 mL). Prevent wetting of the test cup any higher than the sample mark.

10.1.5 Place the cup containing the sample into the heating block and place the cover on the cup. Ensure that the cover is completely seated and secure.

10.1.6 Carefully inspect the igniter coil for cleanliness and mechanical and physical damage. Do not proceed with the test if there is any indication that the igniter is damaged.

WARNING: If the igniter is damaged and does not function, the tester may give an incorrect flash point or no flash point at all. No flash point detection may lead the analyst to believe that the flash point is higher than it actually is and cause the sample

to be heated above its flash point, which could lead to auto- of the sample and result in a fire.

10.1.7 Follow the instructions in the user manual to make the proper connections for the stirrer, igniter, test cup cover, and thermometer.

10.1.8 Start the test according to the instructions in the user manual.

10.1.9 Observe the trial check of the zero point drive of the cup cover mechanism and the trial dip of the ignition system to make sure that they are operating properly.

10.1.10 The analyzer will indicate whether a flash point was detected, and if so, at what temperature. The test may also end due to an analysis error, in which case the analyzer will display an error message and error number. Errors and corrective actions are explained in the user manual. For each test, one of the following results is possible:

10.1.10.1 A flash point is detected, with no errors, within the working temperature range. If the flash point temperature is less than 80 °F, write an NCM to note that the actual flash point may be overstated due to limitations of the tester used, which does not have the capability to cool the sample.

10.1.10.2 No flash is detected over the working temperature range and there are no instrument errors. The result is reported as “flash point > 160 °F.”

10.1.10.3 A flash may or may not have been detected, but an error occurred during the test. Consult the instrument user manual to determine the nature of the error and whether any corrective action is needed. Perform the corrective action, if indicated, and repeat the test on a second aliquot of sample. If there is insufficient sample for the repeat test, write an NCM to explain.

NOTE: In most cases, errors are caused by detection of a flash between igniter dipping cycles. In this case, test another aliquot of the sample while carefully observing the test to visually verify the occurrence of a flash. If the same error is repeated, record the temperature at which the flash was observed. Write an NCM to explain the error and document the supporting visual verification.

10.1.11 All data is automatically saved internally to the instrument and uploaded to the USB flash drive after analysis.

10.2 Maintenance

The sample test cup, cover and stirring mechanism must be clean before sample introduction.

10.3 Troubleshooting

10.3.1 Examine the filament coil for signs of mechanical or physical damage if the flash point result is suspect.

10.3.2 Electrical connections for the igniter and detector must be tight and secure.

10.3.3 Shutter and stirring motor must function properly.

10.3.4 Error codes are an indication of apparatus failure and the associated conditions are in the instrument manual.

11.0 Calculations / Data Reduction

11.1 The observed flash point is corrected for air pressure using the following equation:

$$Corrected\,FlashPoint(^{o}F) = F + 0.06 \times (760\ \text{mm Hg} - \text{ACTUAL mm Hg})$$

Equation 1

Where:

F	=	Observed flash point, °F
ACTUAL mm Hg	=	Ambient barometric pressure at laboratory altitude

11.2 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limits

Determination of a method detection limit is not appropriate for this method.

12.2 Initial Demonstration of Capability

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.8 Four aliquots of the LCS are analyzed using the same procedure used to analyze samples, including sample preparation.

12.2.9 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.10 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test

need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.11 Detailed guidance for demonstrations of proficiency is provided in SOP DV-QA-0024.

12.3 Training and Qualification

The group leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are provided in SOP DV-QA-0024.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

13.2 Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Solvent waste and samples exhibiting a flash: Flammable Solvent – Waste Stream C

14.2.2 Oily samples: Waste Stream O

14.2.3 Expired Chemicals/Reagents – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste.

15.0 References / Cross-References

15.1 ASTM D93-80, *Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester*, American Society for Testing and Materials, 1980.

15.2 SW-846 Manual, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Update IIIB, USEPA Office of Solid Waste.

15.2.1 Method 1010, Revision 0, September 1986, *Pensky-Martens Closed Cup Method for Determining Ignitability*.

15.2.2 Method 1010A, Revision 1, November 2004, *Test Methods for Flash Point by Pensky-Martens Closed Cup Tester*.

15.3 User Manual for the Herzog Automatic Flash Point Analyzer, OptiFlash

16.0 Method Modifications:

Item	Method	Modification
1	SW 1010 SW 1010A	Methods 1010 and 1010A specify using ASTM D93-79 or ASTM D93-80 for the determination of flash point using a Pensky-Martens closed tester. The ASTM standard describes a manual method, but acknowledges in a note that an automatic tester may be used as long as all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument are followed. This SOP refers the analyst to the automatic flash point tester user's manual for specific instructions for proper operation of the tester.
2	ASTM D93	ASTM D93 specifies that the material to be tested must be brought to a temperature of 60 ± 10 °F or 20 °F lower than the estimated flash point, whichever is lower. The tester currently in use does not have cooling capability. Therefore, the lowest achievable temperature is room temperature, which typically ranges from 68 to 85 °F. This theoretically limits the test to samples that flash at a temperature grater than 88 to 92 °F at the laboratory altitude. This translates to approximately 95 to 99 °F at sea level. However, by configuring the tester to start dipping the igniter immediately after starting the heating cycle, the laboratory has successfully determined (within ± 2 °F) the flash point of the reference standard PMCC D-93, which has a flash point of 140 °F at sea level, or approximately 133 °F at the laboratory altitude. By applying the same testing protocol to unknown samples, the demonstrated practical lower limit of the test is 81 °F (or approximately 74 °F at the laboratory altitude).

17.0 Attachments

Attachment 1: Example Flash Point TALS Batchsheet.

18.0 Revision History

- Revision 8, dated 9 November 2018
  - Updated throughout to change equipment form Herzog MP-329 to OptiFlash.
  - Removed section 10.3.4 referencing external barometric pressure. Instrument is equipped with internal ambient pressure reading and auto correction.
  - Removed attachment 2, all data is recorded and corrected from instrument reading.
  - Removed Attachment 1 referencing model MP-329
  - Removed all references to p-Xylene, an alternate standard, PMCC D-93 that flashes greater than room temperature is used.

- Revision 7, dated 01 February 2018
  - Annual Review
- Revision 6, dated 31 January 2017
  - Annual Review
  - Updated sections 2.2, 10.4.13, 10.1.14.1, 10.1.14.2, 10.4.14.3 and 11.2 to remove printed result. The data are manually entered into an excel spreadsheet at the time of analysis.
  - Changed Elga Water to Reagent Water in Section 7.3 and throughout
- Revision 5, dated 29 February 2016
  - Annual Review
  - Minor formatting and grammar changes throughout
  - Removed unnecessary language in Section 9.1
  - Folded note into Section 9.2.3
  - Removed note from Section 9.3 due to redundancy
  - Reworded Section 9.5 for clarity
  - Reworded Section 10.4.3.1
  - Condensed subsections under 10.4.14 for clarity
  - Removed older revision histories
- Revision 4, dated 23 February 2015
  - Updated section 10.4.2.2 to include setting for LCS/LCSD
  - Updated section 9 to include DoD criteria references
  - Added section 13.1
  - Annual review
- Revision 3, dated 28 February 2013
  - Added maintenance and troubleshooting to Section 10 (Sections 10.5 and 10.6)
  - Annual review
- Revision 2, dated 28 February 2013
  - Annual technical review
  - Added section 3.4
  - Revised sections 5.1, 6.3, 9.1, 9.2, 10.1, 10.2, and 11.4 to reflect current practice
  - Clarified section 9.3 to indicate the Reference Standard Analysis serves as the LCS
  - Added requirement to write NCM if no duplicate can be analyzed due to no measureable flashpoints in the batch (Section 9.5)
  - Revised section 10.4.2 for clarity and to add that any adjustments made to instrument be documented in the maintenance log.
  - Revised Attachment 2 to reflect current practice
  - Formatting and editorial changes throughout
- Revision 1.4, dated 28 February 2012
  - Source method review.
  - Inserted references to SW-846 Method 1010 as it is still requested by some states and/or clients. It is identical in process to Method 1010A.
  - Removed paragraph 8.5 which was redundant to previous paragraphs in Section 8.
  - Inserted paragraph 9.1 (and subparagraphs) to describe general QC requirements and references to QA SOPs.

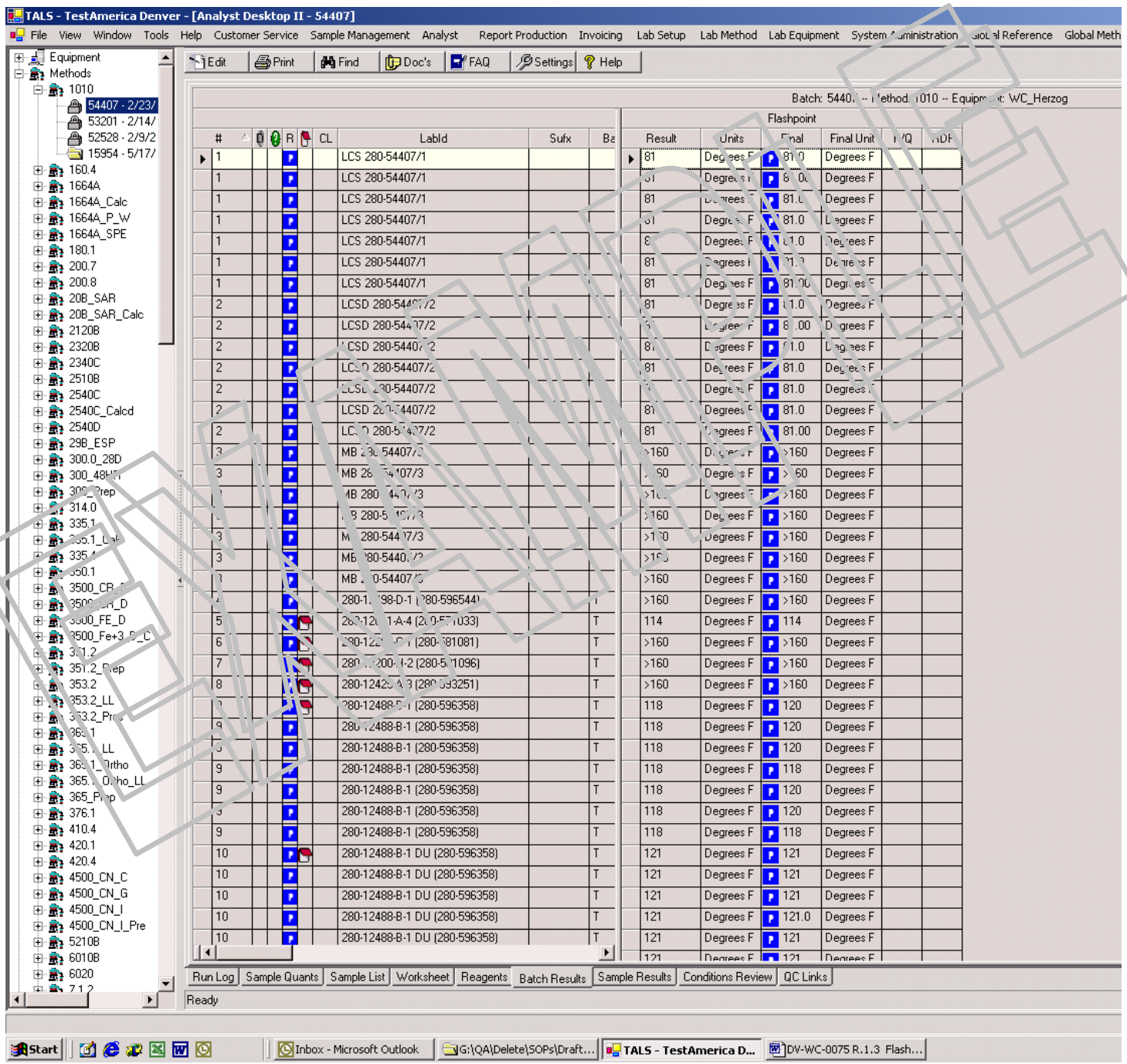
- Included references to use of Excel spreadsheet in procedure.
- Inserted new Attachment 2: Excel spreadsheet used to calculate barometric pressure and corrected flash point temperature
- Renumbered Attachment 2 in rev 1.3 to Attachment 3

- Revision 1.3, dated 04 March 2011
  - Annual Technical Review
  - Corrected various spelling and formatting areas
  - Updated Attachment 2

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Example Flash Point TALS Batchsheet



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Total and Amenable Cyanide by SM 4500-CN⁻ B, 4500-CN⁻ C, 4500-CN⁻ E, 4500-CN⁻ G, SW 9012A and SW 9012B Weak Acid Dissociable Cyanide by 4500-CN⁻ I

Approvals (Signature/Date):

Connie Jewell — Date 6/27/19
Department Supervisor

Doug Gomer — Date 6/27/19
Health & Safety Manager / Coordinator

Roxanne Sullivan — Date 6/27/19
Quality Assurance Manager

Charles Newton — Date 6/27/19
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 Analytes, Matrices, and Reporting Limits

1.1.1 This procedure is for the determination of:

Analyte	CAS Number	Reporting Limit Water (mg/L)	Reporting Limit Soil (mg/kg)
Total Cyanide	57-12-5	0.01	0.50
Amenable Cyanide	STL00015	0.01	0.50
Weak Acid Dissociable Cyanide	STL00195	0.01	0.50
Free Cyanide	STL00131	0.01	0.50

1.1.2 This procedure is applicable to drinking, ground, surface and saline waters, solids and wastes.

1.1.3 This procedure detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine total cyanide, amenable cyanide and weak acid dissociable cyanide as described in Standard Methods 4500-CN^- B, 4500-CN^- C, 4500-CN^- E, 4500-CN^- G and 4500-CN^- I and total and amenable cyanide by SW-846 Methods 9012A and 9012B.

1.1.4 Cyanide amenable to chlorination and weak acid dissociable cyanide appear to be identical, however, in some industrial effluents the cyanide amenable to chlorination yields negative values and the measure of weak acid dissociable is a better alternative.

1.1.5 This procedure describes the reduced volume version of the methods and uses the same reagents and molar ratio to meet the quality control and performance requirement stated in the method.

1.2 Dynamic Range

The approximate working range extends from 0.01 mg/L to 0.4 mg/L for water samples and 0.5 mg/kg to 20 mg/kg for soil samples. Samples with higher concentrations are analyzed at a dilution. Current method detection limits are maintained in the TestAmerica LIMS (TALS).

2.0 Summary of Method

2.1 Total Cyanide

Cyanide, as hydrocyanic acid (HCN) is released from samples by means of reflux-distillation under acidic condition and absorbed in a scrubber containing sodium

hydroxide (NaOH) solution. The cyanide concentration in the scrubber solution is determined using an automated analyzer. The cyanide is converted to cyanogen chloride by reactions with Chloramine-T that subsequently reacts with pyridine and barbituric acid to give a red-colored complex. The color intensity which is proportionate to the cyanide concentration is measured at 570 nm. The concentration of NaOH must be the same in the standards, the scrubber solutions and any dilution of the original scrubber solution.

2.2 Amenable Cyanide

Cyanide amenable to chlorination is determined by using two sample aliquots. The first aliquot is distilled for total cyanide and the second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination. Cyanide amenable to chlorination is the difference in these two values.

2.3 Weak Acid Dissociable Cyanide

Weak acid dissociable cyanide is determined by distillation of the sample to which has been added acetate buffer and zinc acetate solution and sufficient acetic acid to obtain a pH of approximately 4.5 - 6 as determined by methyl red indicator. The cyanide concentration collected under these conditions is determined as described in Section 10.6.

3.0 Definitions

3.1 Cyanide: The term “cyanide” refers to all of the CN groups in cyanide compounds that can be determined as the cyanide ion, CN^- by various chemical methods. These compounds include both simple and complex cyanides.

3.2 Total Cyanide: All cyanides including nondissociable cyanides and cyanide bound in complexes that are readily dissociable or of intermediate stability

3.3 Cyanide Amenable to Chlorination: Free cyanide and complex cyanides that are potentially dissociable, wholly or in large degree.

3.4 Weak Acid Dissociable Cyanide: Cyanide that is available in a slightly acidified solution, including free cyanide.

3.5 Free Cyanide: Cyanide that is not complexed in the sample. Free cyanide is determined as weak acid dissociable cyanide.

4.0 Interferences

4.1 Oxidizing agents such as chlorine will destroy cyanide. Ascorbic acid is used to remove chlorine interferences.

4.2 Some unidentified organic compounds may oxidize or form decomposition products during chlorination, giving higher results for cyanide after chlorination than before chlorination; this gives a negative value for cyanide amenable to chlorination. Examples include samples from petroleum refineries, the steel

industry, and pulp from paper processing. The weak acid dissociable method should be used for these samples. See SOP DV-WC-0083.

4.3 Samples that contain sulfide compounds may produce hydrogen sulfide during the distillation and interfere with color development. This is treated by adding cadmium chloride (Standard Methods) or bismuth nitrate (SW-846 methods) to the sample prior to distillation, which removes sulfur by precipitation as cadmium sulfide or bismuth sulfide.

NOTE: Requirements for sample and standard processing are different for Standard Methods or SW-846 Methods. See Section 10.4.2.

4.4 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide.

4.5 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide. Sulfamic acid is added to remove the nitrate and/or nitrite interference.

4.6 Samples containing surfactants may foam excessively during distillation.

4.7 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation.

4.8 Aldehydes, glucose, and other sugars may convert cyanide to cyanohydrin during distillation. If the client has indicated the possible presence of aldehydes, add 3.5% ethylenediamine as described in Section 10.4.3.

4.9 Amino acids may distill with the cyanide and interfere with the analysis.

4.10 Fatty acids may interfere by forming soaps in the absorption solution.

4.11 Thiocyanate greater than 10 mg/L may interfere.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, latex or nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Potassium cyanide and sodium cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, and dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium Hydroxide	Corrosive Poison Reactive	2 mg/m^3 - ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness and swelling. Greater exposures cause severe burns with possible blindness.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive Poison	5 ppm - ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract and in severe cases, pulmonary edema, circulatory failure and death. Can cause redness, pain and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric acid	Corrosive Poison Irritant, Carcinogen	1 mg/m^3 TWA	Inhalation symptoms may include irritation of the nose and throat, and labored breathing. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, and diarrhea. Skin contact can cause redness, pain, and severe burn. Eye contact can cause blurred vision, redness, pain and severe tissue burns.
Calcium hypochlorite	Strong oxidizer	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Glacial acetic acid	Corrosive Poison Flammable Irritant	10 ppm TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Swallowing can cause severe injury leading to death. Skin contact may include redness, pain, and skin burns. Eye contact may cause severe eye damage followed by loss of sight.
Sulfamic acid	Corrosive Irritant	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Chloramine-T	Irritant	None listed	May cause irritation to the mucous membranes and upper respiratory tract, skin and eyes.
Barbituric acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as a potential health hazard; do not ingest.
Bismuth nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m^3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns, and eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 Build-up of pressure in the distillation apparatus will cause the hot, acidic solution to spray out of the thistle tube. In case vacuum is lost, the condensers must be opened to prevent build-up of pressure. If the solution overflows onto the heating block, turn it off.

5.6 All distillations are to be performed with adequate ventilation.

5.7 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of

mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.8 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. For cyanide amenable to chlorination, the chlorination step will also be performed in a fume hood.

5.9 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Denver associate. The situation must be reported immediately to a laboratory supervisor and the Health and Safety Officer.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Alpkem Automated Segmented Flow Analyzer

- Autosampler
- Proportioning pump
- Injection module
- Colorimeter with 570 nm filter and 10 mm flow cell
- WinFlow Software
- Debubblers
- Miscellaneous tubing and reaction coils.

6.1.2 Midi-distillation apparatus consisting of 100 mL distillation tubes, cold-finger condensers, absorption tubes, and associated apparatus. See Attachment 2.

NOTE: It is important to make sure the joints in the apparatus are well greased to prevent freezing and to prevent leaks. Inspect the joints and re-apply grease as needed, (typically weekly under normal work-loads).

6.1.3 Vacuum pump.

6.1.4 Recirculating chiller.

6.2 Supplies

6.2.1 Disposable auto-sampler vials or culture tubes for samples.

6.2.2 Syringe filters with 0.45 µm filter.

6.2.3 Eppendorf pipettes, various sizes.

6.2.4 Volumetric flasks, class A, various sizes.

6.2.5 Volumetric pipettes, class A, various sizes.

6.2.6 Miscellaneous laboratory apparatus (e.g., magnetic stirrer) and glassware.

6.2.7 pH test strips.

6.2.8 Lead acetate test paper.

6.2.9 Potassium iodide-starch test paper.

6.2.10 Boiling chips.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software, and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

NOTE: TALS IDs for standards and reagents are given in parentheses.

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 One of the calibration standards (ICAL or ICV) must be obtained from a calibration laboratory accredited to ISO/IEC 17025 (ISO Guide 34) by a recognized Accreditation Body. The other may be obtained from other acceptable sources.

7.3 Cyanide Calibration Stock Standard, 1,000 mg/L (CN CAL STD)

7.3.1 This standard is purchased commercially.

7.3.1.1 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the certified concentration from the vendor.

7.3.2 *Alternatively,* the calibration stock standard can be made as described here.

7.3.2.1 Dissolve 2.51 g of dried (103 °C) potassium cyanide and 2.0 g potassium hydroxide in water and dilute to 1,000 mL.

7.3.2.2 This stock solution (prepared by the lab) will be standardized initially and weekly thereafter as described in Section 7.3.

7.3.2.3 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the results of the monthly standardization.

7.4 Standardization of the Stock Cyanide Solution

7.4.1 Reagents

7.4.1.1 Indicator: Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 mL acetone. This standard is purchased commercially.

7.4.1.2 Silver nitrate ($AgNO_3$) titrant; 0.0192 N: Obtain a commercially prepared certified standard.

7.4.2 Cyanide Standardization (required weekly)

7.4.2.1 Add 10 mL of the 1,000 mg/L Cyanide Stock Standard (Section 7.3.2) solution to a 500 mL Erlenmeyer flask. Dilute to 250 mL with reagent water.

7.4.2.2 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution.

7.4.2.3 Titrate with standard silver nitrate, $AgNO_3$, to the first change in color from a canary yellow to a salmon hue.

7.4.2.4 Prepare a blank in a similar fashion. Add 250 mL of deionized water to a 500 mL Erlenmeyer flask.

7.4.2.5 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution. Titrate with standard $AgNO_3$ to the first change in color from a canary yellow to a salmon hue. The standardization should be done in duplicate.

7.4.2.6 Calculate the true stock cyanide concentration as follows:

$$mg/L\ Stock Cyanide = \frac{[(A-B)\times 1000]}{mL\ of\ sample} \qquad \textit{Equation 1}$$

Where:

A = Volume of $AgNO_3$ for titration of sample, mL
B = Volume of $AgNO_3$ for titration of blank, mL

7.4.2.7 If the verification result is within 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard the TALS Reagent module.

7.4.2.8 If the verification result is less than 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard in the TALS Reagent module.

7.4.2.8.1 Create a “new” standard in the TALS Reagent Module for this standard using the new concentration from the verification process.

7.4.2.8.2 Prepare a label for the stock solution with the name of the analyst, date of verification, expiration date (one month beyond the date of re-standardization), lot number, 1% (0.25 N) NaOH, and the actual stock cyanide concentration.

7.4.2.9 The true concentration of the stock cyanide solution after standardization with silver nitrate is used to prepare intermediate and working standards. This concentration does not always equal 1,000 mg/L. The dilution factor relative to the stock standard is provided to aid in the calculations to determine the True Working Standard concentration for the standards listed in Section 7.6.2, where:

$$Conc.\ of\ True\ Working\ Std = \frac{True\ Conc.\ of\ Stock}{DF} \qquad \textit{Equation 2}$$

Where:

$$DF = \frac{Vol.\ of\ intermed\ std\ used\ (mL)}{Final Vol\ of\ dilution (mL)} \qquad \textit{Equation 3}$$

7.5 Intermediate Standard I, 10 mg/L (CN 10ppm)

7.5.1 Obtain a clean 100 mL class A volumetric flask.

7.5.2 Add approximately 50 mL of the 1% (0.25 N) sodium hydroxide (1% NaOH) to the flask.

7.5.3 Pipette 1.0 mL of the 1,000 mg/L calibration stock standard (Section 7.1) into the flask.

7.5.4 Dilute to volume with 1% (0.25 N) sodium hydroxide (1% NaOH). Prepare every 7 days from the standardized stock.

7.6 Calibration Standards

7.6.1 Working Standard, 1.0 mg/L (CN CAL 1 ppm)

7.6.1.1 Pipette 10.0 mL of Intermediate Standard I (Section 7.5) into a 100 mL volumetric flask.

7.6.1.2 Dilute to volume with 1% (0.25 N) sodium hydroxide and mix. Prepare daily.

7.6.2 Initial Calibration Standards

Dilute the 1.0 mg/L or 0.1 mg/L cyanide working standard with 1% (0.25 N) sodium hydroxide as follows:

Standard Level	Intermediate	Vol of Std Used (mL)	Final Volume (mL)	Concentration (mg/L)
1	1 mg/L	0	50	0.00 (Blank)
2	1 mg/L	0.5	50	0.01
3	1 mg/L	1.0	50	0.02
4	1 mg/L	2.5	50	0.05
5	1 mg/L	5.0	50	0.10
6	1 mg/L	10.0	50	0.20
7	1 mg/L	10.0	25	0.40

Calculate the exact concentration for each calibration curve standard using Equations 2 and 3 if stock concentration is different from 1.0 mg = 1.0 mL. When using the TALS Reagent Module, the concentration of the calibration standards will be automatically calculated. Prepare daily.

7.7 Continuing Calibration Verification Standard (CCV), 0.2 mg/L:

The Level 6 calibration standard described in Section 7.6.2 is used as the working CCV standard.

7.8 Second-Source Standards

7.8.1 Initial Calibration Verification (ICV) Stock Standard, 1,000 mg/L (CN ICV Std)

The second-source standard is obtained from a different source than the calibration standards. This standard is available commercially. Manufacturer's expiration date is used. It is prepared and standardized as described in Section 7.4 for the primary standard if unavailable commercially.

7.8.2 Intermediate ICV (second-source) Standard, 10 mg/L (CN ICV Int)

Pipette 1.0 mL of the 1,000 mg/L ICV stock (Section 7.8.1) into a 100 mL volumetric flask. Dilute to volume with 1% (0.25 N) sodium hydroxide. Prepare every 7 days.

7.8.3 Working ICV (second-source) Standard, 0.10 mg/L (CN ICV Daily)

Spike 1 mL of the 10 mg/L intermediate ICV standard (Section 7.8.2) into a 100 mL volumetric flask and fill to the mark with 1% (0.25 N) NaOH. Prepare daily.

7.9 Pyridine-Barbituric Acid Solution, per OI Manual (CN PYR/BARB_)

7.9.1 This standard is purchased commercially.

7.9.2 *Alternatively*, the Pyridine-Barbituric acid solution can be made as described here.

7.9.3 In a hood, place 15 g barbituric ($C_4H_4N_2O_3$) acid (Barbitiric) in a 250 mL volumetric flask and add about 100 mL reagent water, rinsing down the sides of the flask.

7.9.4 Place on a magnetic stirrer and add a stir bar.

7.9.5 Add 75 mL pyridine (Pyridine) while mixing.

7.9.6 Carefully add 15 mL concentrated hydrochloric acid (HCL) while mixing.

7.9.7 Add 150 mL of reagent water and stir until the barbituric acid is dissolved.

7.9.8 Dilute to volume with reagent water.

7.9.9 Store in an amber bottle.

7.9.10 Expires 6 months from preparation.

7.10 Phosphate Buffer Solution 1 M (CN BUFFER)

7.10.1 Dissolve 138 g sodium dihydrogen phosphate monohydrate ($NaH_2PO_4 \bullet H_2O$) (Sodium Phos) in reagent water and dilute to 1,000 mL.

7.10.2 Add 4 mL of Brij-35 to the solution and mix gently.

NOTE: Actual volume varies depending upon equipment operation. It may be necessary to add additional Brij-35 for smooth operation of the equipment.

7.10.3 Store at room temperature.

7.10.4 Filter the solution through a glass fiber filter.

7.10.5 This solution expires 3 months from preparation.

7.11 Chloramine-T, per OI Manual (CN CHLOR-T)

7.11.1 Dissolve 2.0 g Chloramine-T in reagent water and dilute to 250 mL.

7.11.2 Prepare fresh daily.

7.12 Sodium Hydroxide, 10 N (10N_NaOH)

Purchased ready to use.

7.13 Sodium Hydroxide, 2% wt/wt (0.5 N) (2% NaOH_)

7.13.1 Place 80 g NaOH in a plastic 4 liter container.

7.13.2 Add 2,000 mL of reagent water to the container.

7.13.3 Add a magnetic stir bar and stir until the NaOH is dissolved.

7.13.4 Add 2,000 mL of reagent water to the flask. Stir to mix.

7.14 Sodium Hydroxide Dilution Solution, 1% wt/wt (0.25 N) (1% NaOH_)

7.14.1 Place 20 g of NaOH in a 2 liter volumetric flask.

7.14.2 Add approximately 1 liter of reagent water.

7.14.3 Add a magnetic stir bar and stir until the NaOH is dissolved.

7.14.4 Allow to cool, remove stir bar, bring to volume with reagent water, and mix well.

7.15 Sulfuric acid, concentrated, reagent grade (H2SO4)

7.16 Sulfuric acid, 0.02 N (0.02 H2SO4)

7.16.1 If commercial solution is not available, this solution can be prepared as follows. In a 2,000 mL volumetric flask, carefully add 1 mL concentrated sulfuric acid to approximately 1,900 mL reagent water.

7.16.2 Dilute to final volume of 2,000 mL with reagent water and mix.

7.17 Calcium hypochlorite solution, 0.35 M, $Ca(OCl)_2$

7.17.1 Combine 5 g of calcium hypochlorite and 100 mL of reagent water. Shake well before using. Replace monthly.

7.17.2 Alternatively, fragrance free commercial liquid bleach (e.g., Clorox Bleach) can be purchased and used in place of the calcium hypochlorite solution. Replace 1 month after opening.

7.18 Magnesium Chloride solution, 2.5 M (CN Mag Chl)

This reagent is purchased though an approved vendor.

7.19 Glacial acetic acid, reagent grade (Acetic acid).

7.20 Acetate buffer (WAD Acetate)

7.20.1 Dissolve 410 g sodium acetate trihydrate (SODIUM ACETAT) in approximately 450 mL reagent water.

7.20.2 Adjust the pH to 4.5 with glacial acetic acid and dilute to final volume of 500 mL with reagent water.

7.20.3 The solution expires 1 year from preparation.

7.21 Zinc Acetate solution (Zinc Buffer)

7.21.1 Dissolve 100 g zinc acetate dihydrate in a 1 liter volumetric flask filled with approximately 500 mL reagent water.

7.21.2 Dilute to final volume of 1,000 mL with deionized water.

7.21.3 Store in a 1 L plastic container.

7.21.4 The solution expires 1 year from preparation.

7.22 Methyl Red Indicator solution (Methyl Red)

7.22.1 Dissolve 0.1 g methyl red in 100 mL reagent water.

7.22.2 Expires 1 year from preparation.

7.23 Acetic acid, 10% (10% Acetic Acid)

7.23.1 In a vent hood, carefully add 100 mL glacial acetic acid to about 500 mL reagent water, mix, and dilute to 1,000 mL.

7.23.2 The solution expires 1 year from preparation.

7.24 Ascorbic acid crystals (Ascorbic Acid)

7.25 Sulfamic Acid (NH_2SO_3H), 10% wt/wt (CN SULFAMIC)

7.25.1 Dissolve 100 g sulfamic acid in 1,000 mL of deionized water. Mix well.

7.25.2 Store in a repipetter container.

7.25.3 Expires 1 year from preparation.

7.26 Cadmium Chloride powder, ($CdCl_2$), (CAD CHL CN)

Used to remove sulfide interference.

7.27 3.5% Ethylenediamine (EDTA) solution

Used to remove client-identified aldehyde interferences

7.28 Bismuth Nitrate ($Bi(NO)_3•5H_2O$), 0.062 M, (CN_BiN3O9)

7.28.1 Obtain a clean, dry 250 mL volumetric flask.

7.28.2 Add approximately 75 mL of reagent water to the flask.

7.28.3 Add 7.5 g bismuth nitrate to the flask and stir to dissolve.

7.28.4 Slowly add 60 mL of glacial acetic acid, swirling frequently.

7.28.5 Stir until completely dissolved.

7.28.6 Bring to volume with reagent water.

7.29 Brij-35 Start-Up Solution

Concentrated Brij-35 is a buffer solution obtained from the equipment vendor. The start-up solution is prepared by diluting 1 mL of the Brij-35 concentrate to 500 mL with reagent water.

7.30 Reagent Water

Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

7.31 Teflon boiling chips for use as solid matrix for LCS and MB. Record lot number in TALS in prep batch information.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water[1]	HDPE, Glass	500 mL	Cool, ≤ 6 °C	48 hours **	40 CFR Part 136.3
Water[1,2]	HDPE, Glass	500 mL	NaOH, pH > 10; Cool, ≤ 6 °C;	14 days	40 CFR Part 136.3
Water[2]	HDPE, Glass	500 mL	NaOH, pH > 12; Cool, ≤ 6 °C	14 days	SW-846
Solid	HDPE, Glass	5 g	Cool, ≤ 6 °C	14 days	SW-846

[1] Add 1.2 g of ascorbic acid per liter of sample if residual chlorine is present.

[2] Preservation to pH > 10 for NPDES compliance samples (Method 335.4, the SM 4500-CN^- methods); pH >12 for Methods 9012A and 9012B.

****NOTE:** If the client sample arrives unpreserved the sample needs to be checked for interferences (Section 10.4). If there are interferences, treat the sample and preserve immediately. If there are no interferences preserve with NaOH (pH noted above) and follow the 14 day holding time. Document the preservation using an observation Nonconformance Memo (see SOP DV-QA-0031).

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 and 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 **Sample QC:** The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	<1/2 Reporting Limit or < 10% of sample concentration
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Total Cyanide: .90-110% LCS/LCSD RPD: ≤ 10% Statistical Limits[4] for WAD
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Total Cyanide: .90-110% LCS/LCSD RPD: ≤ 10% Statistical Limits[4] for WAD
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Total Cyanide: .90-110% MS/MSD RPD: ≤ 10% Statistical Limits[4] for WAD

Quality Controls	Frequency	Control Limit
High Distilled Standard (HLCS) (0.35 mg/L)	1 in 20 or fewer samples	WAD: 75-120%
Low Distilled Standard (LLCS) (0.10 mg/L)	1 in 20 or fewer samples	WAD:75-120% 335.4: 44-167%

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are stored in TALS.

9.3 Method Blank

9.3.1 Preparation

9.3.1.1 Water Samples:

Add 50 mL of 1% (0.25 N) NaOH into a distillation flask immediately prior to distillation.

9.3.1.2 Solid Samples:

Weigh 1.0 g of Teflon chips into a distillation flask and add 50 mL 1% (0.25 N) NaOH.

9.3.2 Acceptance

Acceptance Criteria: Concentrations in the method blank must be less than one-half the reporting limit or less than 10% of the sample concentration.

Corrective Action: The corrective action for method blank failures is redistillation and reanalysis of all samples in the batch. If there is insufficient sample for reanalysis, a Nonconformance Memo (NCM) must be prepared and the client contacted by the laboratory Project Manager.

9.4 LCS / LCSD (Second Source)

9.4.1 Preparation

9.4.1.1 Water Samples

9.4.1.1.1 Measure 50 mL of 1% NaOH in a graduated cylinder and transfer to the distillation flask.

9.4.1.1.2 Spike 0.5 mL of the 10 mg/L second source intermediate standard (Section 7.8.2) into the flask. Swirl to mix.

9.4.1.2 Soil Samples

9.4.1.2.1 Weigh 1.0 g of Teflon chips into a distillation flask.

9.4.1.2.2 Measure 50 mL of 1% NaOH in a graduated cylinder and transfer to the distillation flask.

9.4.1.2.3 Spike with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.8.2) into the flask. Swirl to mix.

9.4.2 Acceptance

Acceptance Criteria: LCS recoveries for Total Cyanide are 90-110%. Recoveries for Weak Acid Dissociable Cyanide (WAD) are compared to the historical limits stored in TALS. See also Section 9.2.

For DoD QSM 5.0, QC limits for LCS are:

Water: 83-116%
Solid: 76-120%

The LCS for Cyanide Amenable to Chlorination should be 0% which demonstrates that the chlorination process has worked effectively. (This is not reported.)

If a LCSD is also analyzed the RPD must be within ± 10% for Total Cyanide and within historical limits stored in TALS not to exceed ± 20% for WAD. See Also Section 9.2.

Corrective Action: If the LCS fails, redistill and reanalyze all samples in the batch. If reanalysis is not possible, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager. See the TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*, for additional guidance.

9.5 MS/MSD

9.5.1 Preparation

9.5.1.1 Water Samples

Measure 50 mL of sample into a class A graduated cylinder. Spike the aliquot with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.8.2) and bring to 50 mL with sample. The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

9.5.1.2 Soil Samples

Weigh 1.0 g of sample into a distillation flask. Spike the sample aliquot with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.8.2), then add 1% (0.25 N) NaOH for a total volume of 50 mL. The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

9.5.2 Acceptance

Acceptance Criteria: MS/MSD recoveries are compared to the limits stored in TALS. The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. See also Section 9.2. For QSM 5.0 the recovery limits are the same as the LCS (see Section 9.4.1.2.3) and the RPD limit is ± 20%

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check the calculations and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);

- Flag the data for any results outside of acceptance limits.

- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.

- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client’s needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are

reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Any samples that have target analytes at such low levels do not provide useful precision data via duplicate analyses. If a sample duplicate is performed for DoD QSM 5.0, the RPD limit between duplicates is ± 20%.

9.6 High and Low Distilled Standards (HLCS & LLCS), 0.4 mg/L and 0.10 mg/L

Standards are distilled to monitor the efficiency of the distillation process and verify the linearity of the curve.

9.6.1 Preparation

9.6.1.1 Water Samples

In each of two graduated cylinders, add 25 mL of 2% NaOH (see Section 7.13).

9.6.1.1.1 For the HLCS, add 1.75 mL of the 10 mg/L cyanide standard (CN 10ppm) and bring to 50 mL with reagent water.

9.6.1.1.2 For the LLCS, add 0.5 mL of the 10 mg/L cyanide standard (CN 10ppm) and bring to 50 mL with reagent water.

9.6.1.2 Soil Samples

Weigh 1 g of Teflon chips into a distillation flask for each standard.

9.6.1.2.1 For the HLCS, add 25 mL of 1% NaOH to the distillation flask and spike 2.0 mL of the 10 mg/L cyanide standard to the flask and add 25 mL of 1% NaOH for a total volume of 50 mL.

9.6.1.2.2 For the LLCS add 25 mL of 1% NaOH to the distillation flask and spike 0.5 mL of the 10 mg/L cyanide standard to the flask and add 25 mL of 1% NaOH for a total volume of 50 mL.

9.6.2 Acceptance

Acceptance Criteria: Recoveries for the HLCS and LLCS standards must be ± 10% of the true value. For DoD QSM 5.0, check method comments to determine if the variance for the LLCS is accepted, in which case the acceptance limits are ± 15% of the true value for LLCS and ± 10% for the HLCS.

Corrective Action: Distilled standard failure results in re-distillation and reanalysis of all associated samples. One possible exception is the situation in which recoveries are greater than 110% and cyanide was not detected in the samples. In that case, a Nonconformance Memo should be prepared and the failure noted in the report together with the sample results without taking other corrective action. For DoD QSM 5.0 check method comments to determine if this is acceptable for the project.

9.7 Cyanide QC

9.7.1 The amenable cyanide method requires three different procedures.

9.7.2 The first is the total cyanide. The QC for total cyanide is performed as described in sections 9.3 -9.6.

9.7.3 The second is the Non-Amenable Cyanide. The QC required is as follows:

9.7.3.1 A treated Method Blank. The Method Blank is prepared the same as described in section 9.3. It than goes through the treatment process as described in section 10.4.

9.7.3.2 A treated LCS (see section 10.5). The treated LCS is prepared the same as described in section 9.4. It than goes through the treatment process as described in section 10.5. The treated LCS should have a 0% recovery to show that the chlorination process is working correctly.

9.7.3.3 An LCS/LCSD that is spiked post-treatment. The LCS/LCSD are treated method blanks that are than spiked after the chlorination treatment but just prior to distillation. The post-treated LCS/LCSD are to show that the distillation procedure is working correctly and there is no excess chlorine.

9.7.3.4 A sample duplicate is performed for demonstration of batch precision.

9.7.4 The third is the Amenable Cyanide. The Amenable Cyanide is a calculation method using the total cyanide and non-amenable cyanide results.

9.8 Instrument QC

9.8.1 Initial Calibration Verification (ICV)/ Initial Calibration Blank (ICB)

Immediately after the initial calibration, the calibration is verified using a second-source ICV standard and an initial calibration blank ICB (1% NaOH).

Acceptance Criteria: The measured result for the ICV must be within 10% of the expected value.

The ICB must be less than the reporting limit.

For DoD QSM 5.0, the ICB must be < LOD.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

9.8.2 Continuing Calibration Verification (CCV) / Continuing Calibration Blank (CCB)

A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.7) are required after every 10 or fewer samples and at the end of the run.

Acceptance Criteria: The standard check (CCV) must be within 10% of the expected value.

Blanks must be less than the reporting limit.

For DoD QSM 5.0, the CCB must be < LOD.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Record identification for all pipettes, balances and other equipment used in the determination of cyanide in the batch record.

10.4 Sample Preparation

10.4.1 The following sections describe separate preparation procedures for the different forms of cyanide.

Section 10.4: Cyanide Amenable To Chlorination
Section 10.5: Total Cyanide
Section 10.6 Weak Acid Dissociable Cyanide

10.4.2 Check aqueous samples for sulfide prior to distillation using lead acetate paper.

10.4.2.1 Moisten the paper with 2 or 3 drops of acetate buffer, and then place 1 drop of sample on the paper. A dark color indicates a positive test for sulfide. Record the result as "positive" or "negative" in the TALS prep batch.

10.4.2.1.1 If the test for sulfide is positive, Method 9012A and Method 9012B require that the samples be treated with bismuth nitrate rather than cadmium chloride and processed in a separate batch. The standards must be treated in the same manner as the samples, including addition of bismuth nitrate and distillation. See Section 10.6.10**.**

10.4.2.1.2 If the samples test positive using lead acetate paper and are to be analyzed using Standard Methods, treat 75 mL of the stabilized sample (pH > 12) with cadmium chloride.

10.4.2.2 Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper.

10.4.2.3 Filter the solution through a dry filter paper and from the filtrate, measure the sample aliquot to be used for analysis.

10.4.2.4 The filter papers are contaminated with cadmium. Use the designated disposal container for these filters.

10.4.3 If the client has specifically indicated that aldehydes may be present, 2 mL of 3.5% ethylenediamine is added per 100 mL of sample upon receipt.

10.5 Cyanide Amenable To Chlorination Sample Preparation

10.5.1 Two sample aliquots are required for the determination of cyanide amenable to chlorination. The first aliquot is distilled for total cyanide (see Section 10.6). The second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination.

NOTE: If the results for Total Cyanide are less than the reporting limit, the cyanide amenable to chlorination is not determined and reported as ND. The non-amenable cyanide is then equal to the total cyanide result. The method blank reported in this case is the Total Cyanide method blank.

NOTE: The chlorination process must be performed in a fume hood.

10.5.2 Measure the sample aliquots to be chlorinated (including a method blank, LCS and sample duplicate) into 100 mL beakers covered with a large plastic tub. Keep beaker covered with wrapped watch glass. Alternative means of protecting samples from light may be used.

10.5.2.1 For water samples, use 50 mL of sample.

10.5.2.2 For soil samples, use 1.0 g of sample and add 50 mL 1% (0.25 N) NaOH.

10.5.2.3 Clearly label the samples with the proper identification and "chlorinated" as appropriate.

10.5.3 Check the pH of samples with pH test strips. Record the results on the bench sheet.

10.5.4 Adjust the pH of the samples in the beakers to between 11 and 12 with the 10 N sodium hydroxide solution.

10.5.5 Test the samples with KI-Starch paper and add bleach solution drop-wise to each sample while mixing (use a magnetic stirrer) until a positive test is obtained. A positive test is indicated by a blue or black color on the paper.

10.5.6 Maintain the excess chlorine level in the sample for 1 hour while keeping the pH of the samples between 11 and 12 with constant mixing (use a magnetic stirrer). Add bleach solution and sodium hydroxide as necessary. Document the PH in Worksheet tab.

10.5.7 After 1 hour, add 0.1 g portions of ascorbic acid crystals until a negative test is obtained with KI-Starch paper.

10.5.8 Add an additional 0.1 g of ascorbic acid crystals to the sample to ensure an excess of the reagent

10.5.9 Transfer the contents of the beakers into distillation flasks quantitatively, rinsing with reagent water.

10.5.10 Proceed to section 10.6.4 for the distillation process.

10.6 Total Cyanide Sample Preparation

10.6.1 Check the pH of aqueous samples with pH test strips. Record the results on the TALS bench sheet. If the sample pH is ≤ 12 for Methods 9012A or 9012B or ≤ 10 for SM 4500 methods, document the improper preservation with a NCM.

10.6.2 Check aqueous samples for oxidizing agents such as chlorine.

10.6.2.1 Place one drop of sample on a strip of potassium iodide (KI)-starch test paper. A blue color indicates the need for treatment.

10.6.2.2 Record the result as "positive" or "negative" on the benchsheet.

10.6.2.3 If a positive test is obtained, add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper.

10.6.2.4 Add an additional 0.1 g of ascorbic acid in excess.

10.6.3 Measure sample aliquots into the distillation flasks as follows:

10.6.3.1 For water samples use 50 mL of sample.

10.6.3.2 For solid samples use 1.0 g of sample and add 50 mL 1% (0.25 N) NaOH.

10.6.3.3 Prepare the batch QC samples as described in Section 9 (MB, LCS, MS/MSD, LLCS, HLCS)

10.6.4 Place 25 mL 2% sodium hydroxide into the absorption tubes.

10.6.5 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

10.6.6 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

NOTE: Batch QC samples must be rotated through all distillation glassware and positions. Do **not** use specific glassware or distillation positions for the MB and LCS/LCSD.

10.6.7 Turn on the vacuum pump and ensure the chiller is on. Also be sure that the slot hood is operating.

10.6.8 Adjust the vacuum to provide a flow rate of approximately 2-3 bubbles per second (i.e., this is approximately 1/8-1/4 inch of foam in the scrubber) in the distillation flask.

10.6.9 Verify that there are no leaks in the system by observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

10.6.10 If the samples are logged for Method 9012A or 9012B and the test for sulfide is positive, all standards (minimum of five standards and blank), QC and samples must be processed in a separate batch.

10.6.10.1 Add 5 mL of 0.062 M bismuth nitrate solution (see Section 7.28) through the thistle tube to every standard, sample and QC sample in the analytical batch.

10.6.10.2 Samples designated for analysis using DoD QSM 5.0 where the project specifically requires MSA are analyzed using the method of standard addition utilizing a 2-point spike and calculating the sample concentration using the MSA data. Consult your Supervisor, a Technical Specialist, or the QA Manager before proceeding.

10.6.11 Add 2 mL of 10% sulfamic acid solution (Section 7.25) through the thistle tube. Allow to mix for 3 minutes.

10.6.12 Slowly and carefully, add 2.5 mL concentrated sulfuric acid through the thistle tube. Rinse the tube with a little reagent water and allow to mix for 3 minutes.

10.6.13 Add 2 mL of magnesium chloride solution (Section 7.17) and mix. If excessive foaming is observed, add additional magnesium chloride.

10.6.14 Turn on the heating mantles and heat the samples to boiling. While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

10.6.15 Allow samples to reflux for 1.5 hours by initiating the timer on the heating mantle.

10.6.16 After 1.5 hours of refluxing, allow the samples to cool for 15 - 30 minutes while air is flowing.

10.6.17 *While the vacuum is still on*, remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with reagent water.

Note: It is important to keep the vacuum on to prevent the distillate from being trapped inside the bubbler.

10.6.18 Remove the flasks and dispose of the contents as directed in Section 14.0. Residue not removed by this method must be scrubbed out.

10.6.19 Dilute the sodium hydroxide in the absorption tube to 50 mL with deionized water using a class A graduated cylinder and store in labeled plastic vials.

10.6.20 Place each batch of distillates in a box and store the samples at 4 °C in the sample refrigerator until they are analyzed.

10.6.21 At the end of the day, turn off the vacuum.

10.6.22 Proceed to Section 10.8 for colorimetric analysis of the distillates.

10.7 Weak Acid Dissociable Cyanide in Water – Sample Preparation (SM 4500-CN I)

10.7.1 Measure the pH of water samples with a pH test strip. Record the results on the bench sheet. If the sample pH is less than 12, document the improper preservation with a NCM.

10.7.2 Measure 50 mL or an aliquot diluted to 50 mL into a distillation flask for each sample plus matrix spike and matrix spike duplicate. (See Section 9.5).

10.7.3 Prepare the batch QC samples as described in Section 9.0 (MB, LCS, LLCS, and HLCS)

10.7.4 Record the sample volume on the bench sheet.

10.7.5 Place 25 mL 2% (0.5 N) sodium hydroxide into the absorption tubes.

10.7.6 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

10.7.7 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

10.7.8 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

10.7.9 Adjust the vacuum to provide a flow rate of about 4 bubbles per second in the distillation flask, verifying that there are no leaks in the system by observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

10.7.10 Add through the air inlet tube: 2 mL acetate buffer (Section 7.20), 2 mL zinc acetate solution (Section 7.21), and 2 or 3 drops of methyl red indicator. Rinse tube with about 2 mL reagent water and allow to mix.

10.7.11 If the solution is not pink, add 10% acetic acid drop-wise until a pink color persists, rinsing in with reagent water.

10.7.12 Turn on the controller and heat the samples to boiling. While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

10.7.13 Allow samples to reflux for 1.5 hours. Then allow the samples to cool for 15 minutes while air is flowing. Record start and end times for the distillation in the batch record.

10.7.14 *While the vacuum is still on*, remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with reagent water.

Note: It is important to keep the vacuum on to prevent the distillate from being trapped inside the bubbler.

10.7.15 Dilute the sodium hydroxide in the absorption tube to 50 mL with reagent water using a class A graduated cylinder and store in labeled plastic vials.

10.7.16 Place each batch of distillates in a box and store the labeled distillates at 4 °C until they are analyzed.

10.7.17 Raise the cold-finger condensers and rinse into the distillation flasks with de-ionized water. Dispose of the contents as directed in Section 14.0. Residue not removed by this method must be scrubbed out.

10.7.18 Rinse absorber tubes with reagent water.

10.7.19 At the end of the day, turn off the vacuum.

10.7.20 Proceed to Section 10.8 for colorimetric analysis of the distillates.

10.8 Instrument Set-Up

10.8.1 Verify that the 570 nm filter is installed.

10.8.2 Instrument Stabilization (per Alpkem manual)

10.8.2.1 Connect the reagent pump tubes to a reagent bottle containing the start-up solution (Section 7.29).

10.8.2.2 Start the pump, allowing the start-up solution to flow through the entire system.

10.8.2.3 Make sure that the flow cell of the detector is purged of all bubbles and the flow is stable and free from surging.

10.8.2.4 Once a stable flow is achieved, connect the reagent pump tubes to their respective reagent bottles, as shown in the schematic in Attachment 3.

10.8.2.5 Allow the reagents to flow through the entire system, then, once again, verify that the flow cell of the detector is purged of all bubbles.

10.9 Initial Calibration

10.9.1 Calibration is performed daily or each time the instrument is set up using the standards shown in Section 7.6 and the external standard method.

NOTE: The use of internal standards is not applicable for this spectrophotometric method.

10.9.2 A minimum of five standards and a blank are required for the calibration. The high standard in Section 7.6 may be dropped if needed and sample dilutions performed appropriately.

NOTE: If sulfide was detected during the sample preparation step and the samples are logged for 9012A or 9012B, then bismuth nitrate must be used to precipitate the sulfide. The method of standard additions spike must be prepared, and all calibration standards must be treated and distilled in the same manner as the samples, including the addition of bismuth nitrate. A minimum of five standards and a blank shall be distilled. Use the same calibration levels as shown in the table in Section 7.6.

10.9.3 The calibration function is calculated by least-squares linear regression. See Section 11.2

Acceptance Criteria: The correlation coefficient, r, must be $\geq$ 0.995 ($r^2 \geq 0.99$) and the absolute value of the intercept must be lower than one-half the response for the reporting limit.

Corrective Action: If the correlation coefficient is < 0.995 or the absolute value of the intercept is too large, locate and correct the problem and re-calibrate the instrument.

10.9.4 Assess the peak height of the synchronization (sync) standard.

Acceptance Criteria: The peak height of the sync standard should be ± 10% of pervious sync.

Corrective Action: If the peak height is < 150,000, the flow cell of the instrument must be cleaned (consult manufacturer's instructions), and then the instrument must be recalibrated

10.9.5 Initial Calibration Checks

Immediately after the initial calibration, the calibration is verified using a second-source, initial calibration verification (ICV, see preparation in Section 7.8.3) standard and an initial calibration blank (ICB, 1% NaOH).

Acceptance Criteria: The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

10.9.6 Continuing Calibration Checks

A standard check (CCV; see preparation in Section 7.7) and a blank (CCB made up of 1% NaOH) and are required after every 10 or fewer samples and at the end of the run.

Acceptance Criteria: The measured result for the CCV must be within 10% of the expected value, and the CCB must be less than the reporting limit.

Corrective Action: If either or both the CCV and CCB fail, all samples since the last successful calibration check must be reanalyzed.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≥0.995 correlation coefficient
Initial Cal. Verification (ICV)	Immediately after the calibration	± 10% of the expected value
Initial Cal. Blank (ICB)	Immediately after the calibration	Less than the reporting limit
Continuing Cal. Verif. (CCV)	Prior to / after every 10 injections	± 10% of the expected value
Continuing Cal. Blank (CCB)	Prior to / after every 10 injections	Less than the reporting limit

10.10 Sample Analysis

10.10.1 Following instrument set up and calibration, the sample distillates are analyzed in exactly the same manner as the calibration standards. The routine run sequence is as follows:

Sync
Carryover
Baseline
Cal 0.00 ppm
Cal 0.02 ppm
Cal 0.05 ppm
Cal 0.10 ppm
Cal 0.20 ppm
Cal 0.40 ppm
Blank
Baseline
Second-source ICV
ICB
Baseline
High concentration distilled standard (HLCS)
Low concentration distilled standard (LLCS)
LCS
LCSD

Method blank
5 samples (may include MS/SD)
Blank
Baseline
CCV
CCB
Baseline
10 samples (may include MS/SD)
Blank
Baseline
CCV
CCB
Baseline
Additional cycles of 10 samples with CCV/CCB
Blank
Baseline
Closing CCV
Closing CCB
Baseline

10.11 Instrument Shut-Down

10.11.1 Disconnect the reagent lines and put all of them into the reagent water, except the buffer line.

10.11.2 The buffer line is put into the bridge water.

10.11.3 Rinse all tubes for at least 10 minutes

10.11.4 Switch the reagent lines to Kleenflow Base and rinse for 10 minutes

10.11.5 Switch the reagent lines to reagent water and rinse for 10 min.

10.11.6 Turn off instrument and pump.

10.11.7 Raise platens.

10.11.8 Empty Waste.

10.12 Troubleshooting / Maintenance

10.12.1 Ensure vacuum system is free of leaks by *lightly* applying stopcock grease to all ground glass joints. DO NOT over-grease.

10.12.2 Ensure chiller is on and functioning.

10.12.3 Ensure glassware is free of etching and chips.

10.12.4 Ensure standard solutions are at pH ≥12.

10.12.5 Verify instrument reagent flows are proper and that pump tubing is in good condition.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the TestAmerica Policy, CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 Total Cyanide

11.2.1 A linear calibration model is used to relate the cyanide concentration to the absorbance as follows:

$$y = mx + b \qquad \textit{Equation 4}$$

Where:

y	=	Absorbance of cyanide standard at 570 nm.
x	=	Cyanide concentration of standard, mg/L.
m	=	Slope of the fitted straight line.
b	=	y-intercept of the fitted straight line.

11.2.2 The cyanide concentration in an unknown aqueous sample extract is calculated by solving the calibration equation (Equation 1) for concentration (x) and using the measured absorbance of the sample, as follows:

$$x = \frac{y - b}{m} \qquad \textit{Equation 5}$$

Where:

x	=	cyanide concentration in sample (mg/L)
y	=	absorbance of the distillate at 570 nm
m	=	slope of the calibration line
b	=	y-intercept of the calibration line

11.2.3 If an aqueous sample was diluted, use Equation 5 to calculate a final result.

$$C_s = x \times DF \qquad \textit{Equation 6}$$

Where:

C_s	=	Cyanide concentration in the original sample (mg/L).
x	=	Cyanide concentration in sample distillate (mg/L).
DF	=	Dilution factor, if applicable.

11.2.4 The cyanide concentration in an unknown solid sample extract is calculated using the following equation:

$$C_s = \frac{x \times V_t}{W \times D} \times DF \qquad \textit{Equation 7}$$

Where:

C_s = Cyanide concentration in the original sample (mg/L)
x = Extract analyte concentration, mg/L
V_t = Volume of distillate, L (nominal 0.050 L)
W = Weight of sample, kg (nominal 0.001 kg)
D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis
DF = Dilution factor, if applicable.

NOTE: All routine calculations for total cyanide are performed by the instrument data system and TALS, provided dilutions and other information have been correctly entered.

11.3 Cyanide Amenable to Chlorination:

Amenable Cyanide = Total CN Result – Treated Result *Equation 8*

11.3.1 The "Total CN Result" is the cyanide concentration for the sample aliquot that was distilled without treatment with chlorine. The treated result is the sample portion that was chlorinated and then distilled. The treated result is reported as Nonamenable Cyanide (the cyanide remaining after chlorination) and is calculated in the same manner as Total Cyanide. See Section 11.2 for detailed descriptions of the calculations.

11.3.2 If the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, a corrective action and/or a discussion in the final report is required. Iron-cyanides can cause this to occur. Weak acid dissociable cyanide would be a better method for these types of samples.

11.4 Weak Acid Dissociable Cyanide

The concentration of cyanide measured after the procedure in Section 10.7 is the weak acid dissociable cyanide. See Section 11.2 for detailed descriptions of the calculations.

11.5 Accuracy

ICV, CCV, LCS, HLCS, LLCS:

$$\%\,\mathrm{Recovery} = \frac{observed\,concentration}{known\,concentration} \times 100\% \qquad \textit{Equation 9}$$

MS/MSD

$$\%\,\mathrm{Recovery} = \frac{(spiked\,sample\,conc.) - (unspiked\,sample\,conc.)}{spike\,concentration} \times 100\% \qquad \textit{Equation 10}$$

11.6 Precision (RPD)

$$\%\,\mathrm{RPD} = \frac{|\,orig.\,sample\,value - dup.\,sample\,value|}{[(orig.\,sample\,value + dup.\,sample\,value)/2]} \times 100\% \qquad \textit{Equation 11}$$

11.7 Data Upload

Refer to Work Instruction WI-DV-0068, Cyanide Upload, for upload instructions into TALS.

11.8 Reporting

11.8.1 Reporting units are mg/L for water samples and mg/kg for solids samples.

11.8.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.8.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.8.4 All associated data are entered or uploaded into TALS as required. For soil samples, the total cyanide concentration is calculated by the LIMS using equation 7 (section 11.2.4). If the non-amenable cyanide data are uploaded, the soil concentration is also calculated by TALS. If the non-amenable cyanide data are manually entered, the analyst must first calculate the soil concentration from the instrument result using equation 7 (section 11.2.4). In either case, the amenable cyanide result is calculated by TALS using equation 8 (section 11.3). If dilutions are performed, the amenable cyanide result in TALS does not reflect an adjusted RL due to the dilution.

Note: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.9 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. CA-Q-

S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements indicate a greater frequency.

12.1.2 The current MDL value is maintained in TALS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1 Four aliquots of the QC check sample or LCS (independent source from the calibration) and a method blank are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample (or LCS) should be equivalent to a mid-level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. The method blank must be less than ½ the RL. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 For Amenable Cyanide, a purchased QC or PT sample is used in place of LCS aliquots.

12.2.5 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.6 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 In general, the quantity of chemicals purchased by TestAmerica Denver is based on expected usage during its shelf life. The volume of reagents and standards prepared for this procedure reflects anticipated usage.

13.2 Source reduction is achieved through the use of midi-distillation followed by an automated colorimetric determination.

13.3 The volume of hazardous waste is minimized through proper segregation and management of the various waste streams generated by this procedure.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Environmental Health and Safety Manual, *Waste Management and Pollution Prevention*.

14.2 The following waste streams have been identified for this method:

14.2.1 Cyanide standardization waste – Aqueous Alkaline (E)

14.2.2 Distilled sample – Aqueous Acidic (F)

14.2.3 Distillate – Aqueous Alkaline (E)

14.2.4 Alpkem process waste – Aqueous Alkaline, contains pyridine (E)

14.2.5 Contents of sampler cups – Aqueous Alkaline (E)

14.2.6 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Standard Methods for the Examination of Water and Wastewater, On-line Edition.

15.1.1 4500-CN^- A-1999. Introduction

15.1.2 4500-CN^- B-1999. Preliminary Treatment of Samples

15.1.3 4500-CN^- C-1999. Total Cyanide after Distillation

15.1.4 4500-CN^- E-1999. Colorimetric Method

15.1.5 4500-CN^- G-1999. Cyanides Amenable to Chlorination after Distillation

15.1.6 4500-CN^- I-1999. Weak Acid Dissociable Cyanide

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.2.1 Method 9012A, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 1, December 1996.

15.2.2 Method 9012B, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 2, August 2002.

15.3 OI Manual (available on instrument computer)

15.4 Department of Defense Quality Systems Manual for Environmental Laboratories, DoD QSM Version 5.0, July 2013.

16.0 Method Modifications:

Item	Method[1]	Modification
1	SM 4500-CN^- SW 9012A SW 9012B	This SOP substitutes bleach for calcium hypochlorite solution.
2	SM 4500-CN^-	There are differences among the referenced methods concerning the sodium hydroxide concentration in working standards: Standard Method 4500-CN E states in Section 4a that working standards are made using a solution containing 1.6 grams per liter of water, which is equal to 0.04 N. Methods 9012A and 9012B state in Section 7.4.1 that calibration standards are prepared using 50 mL of 1.25 N sodium hydroxide and diluting to 250 mL, which produces a 0.25 N sodium hydroxide concentration. This procedure uses 0.25 N NaOH to ensure stability of standards. This is the same concentration used in EPA Method 335.4 and SW-846 Method 9012.
3	SM 4500-CN^- SW 9012A SW 9012B	The stock standard is verified if no certificate of analysis is available. Monthly verifications are sufficient to monitor the concentration due to the rate of use of the standard.
4	SM 4500-CN^- SW 9012A SW 9012B	The reflux time for Cyanide Amenable to Chlorination and Total Cyanide has been changed to 1.5 hours versus 1.0 hours to accommodate the reflux time for samples requiring distillation under the Clean Water Act (EPA Method 335.4).
5	SM 4500-CN^-	When the sulfide test is positive for analysis by SM 4500-CN^-, the samples will be treated with cadmium chloride rather than the lead(IV) carbonate listed in SM 4500-CN^- B or lead(II)carbonate listed in SM 4500-CN^- C. This change reduces environmental pollution and provides a standardized sulfide treatment across all cyanide methods.

Item	Method[1]	Modification
6	SM 4500-CN⁻ SW 9012A SW 9012B	SM 4500-CN⁻ B-1999 states that use of ascorbic acid be used for preservation when residual chlorine is suspected. 40 CFR Part 136.3, Table II states use of reducing agent with none specified. This laboratory uses the ascorbic acid rather than sodium arsenite as sodium arsenite is a hazardous material and the regulation is not explicit regarding which reducing agent to use.
7	SW 9012A SW 9012B	Methods 9012A and 9012B state the amenable cyanide test must be performed under amber light. In this procedure the beakers and watch glasses are wrapped with foil or kept in the dark by alternate means as described in SM 4500-CN⁻.
8	SW 9012A SW 9012B	Calibration is verified with an independently prepared check standard (ICV) with every analytical run and a CCV is run after every 10 samples, instead of for every 15 samples. Acceptance criteria used is ± 10% rather than ± 15%.
9	SW 9012A SW 9012B	Methods 9012A and 9012B state the use of a 15 mm flow cell. The equipment used in this laboratory utilizes a 10 mm flow cell, which is that specified in SM 4500-CN⁻.
10	SW 9012A SW 9012B	Methods 9012A and 9012B utilize the method of standard additions if MS/MSD recoveries exceed acceptance limits. Because MS/MSD results may not have a direct bearing on other samples in the batch, matrix effect is assumed if MS/MSD recoveries exceed acceptance limits when LCS recoveries are acceptable.
11	SM 4500-CN⁻ SW 9012A SW 9012B	Chloramine-T solution is prepared by adding 2.0 g Chloramine-T to deionized water and diluting to 250 mL. Methods 9012A/B use a proportion of 1.0 g to 250 mL and SM 4500 CN uses 1 g to 100 mL. This laboratory has determined that the more concentrated solution, consistent with the SM 4500 CN provides more consistent coloration of samples.

[1] SM 4500-CN⁻ refers to approved version 1999 or 2011.

17.0 Attachments

Attachment 1: Example Cyanide Preparation Bench Sheet

Attachment 2: Cyanide Midi-Distillation Apparatus

Attachment 3: Alpkem Manifold Schematic

18.0 Revision History

- Revision 11, dated 30 June 2019
  - Annual Review
  - Updated copyright information

- Revision 10, dated 30 June 2018
  - Updated section 10.5.6 Required PH documentation.

- Revision 9, dated 9 March 2018
  - Updated section 7.9 to reflect source method procedure.
  - Updated Section 12.1 with new corporate SOP#.

- Revision 8, dated 14 July 2017
  - Annual Review

- Updated Section 9.2 High Distillation Standard (HDS) from 0.4 to 0.35 mg/L
- Updated Section 9.6 to perform the High Distillation Standard (HDS) from a 0.4 to a 0.35 mg/L concentration
- Updated instructions in Sections 9.6.1.1.1 and 9.6.1.1.2 to better reflect current procedure

- Revision 7, dated 31 August 2016
  - Annual Review
  - Minor language changes and reformatting throughout
  - Reformatted Section 2.0 for consistency with other SOPs
  - Added MS/MSD information to Section 9.5

- Revision 6, dated 31 August 2015
  - Added current section 7.2 – Reference to ISO Guide 34 for standards
  - Removed previous section 7.3.1.1 not requiring standardization of stock standard
  - Updated section 7.3.2 to require stock standards to be standardized weekly
  - Updated section 7.3.2.2 to require stock standards to be standardized weekly
  - Added reference to the use of standardized stock standards to section 7.5.4
  - Removed the reference for not requiring standardization of stock standard to section 7.8.1
  - Added section 7.17 calcium hypochlorite solution recipe in addition to bleach reference
  - Changed section 7.16 from 6 month expiration to 1 month expiration for calcium hypocholrite solution.
  - Updated sections 12.2.5 and 12.3 regarding documentation of trainer/

- Revision 5, dated 28 February 2015
  - Updated section 7.6 to level 6 calibration
  - Updated section 7.16 from 1 month to 6 months expiration
  - Updated section 9.2 to reflect current TALS values
  - Corrected section 9.5.1 from 25ml to 50ml.
  - Corrected order of section 10.5
  - Corrected section 10.5.19 and 10.6.15 for the use of class A graduated cylinder.
  - Added note to section 10.5.6 regarding the rotation of distillation glassware.
  - Updated run sequence in 10.9.1
  - Increased rinse time in section 10.10.3 from 5 to 10min.
  - Updated section 11.1 to reflect the current corporate calibration SOP.
  - Updated section 12.2 to include Amenable DOC requirements

- Revision 4, dated 9 April 2014
  - Indented sections 7.3.2.9 and 7.3.2.10 as 7.3.2.8.1 and 7.3.2.8.2 to clarify that a new standard is created ONLY if the re-standardized value is <97%
  - Revised section 7.4 to clarify preparation of intermediate standard
  - Revised Section 7.5 to reflect current practice (2 mL changed to 10 mL)
  - Revised table of calibration standards to reflect current practice and match those in SOP DV-WC-0091.
  - Revised Section 7.8.6 to remove filtering of barbituric acid
  - Revised Section 7.9.3 to state solution stored at room temperature
  - Revised Section 7.9.4 to specify solution filtered through glass fiber filter
  - Revised Section 7.12 to reflect current practice
  - Revised Section 7.13 to reflect current practice

- Added use of plastic container for storage in Section 7.20.3
- Section 7.24.1: Changed amount and final volume of sulfamic acid prep to match practice. Added storage requirements.
- Specified aldehyde interferences must be client identified for treatment at lab in Section 7.26.1.
- Revised Section 7.27 to reflect current practice
- Changed use of Ottawa sand to Teflon chips for solid matrix for QC samples throughout.
- Revised Section 9.4.1 to reflect current practice
- Removed Note from Section 9.4.1 since second source standard is always used for LCS spike.
- Change acceptance criteria to reflect only HLCS in Section 9.6.2.1.2.
- Revised Section 10.3.2.1 to record sulfide test results in TALS prep batch
- Revised Section 10.4.2 from covering the beakers with aluminum to covering with a large plastic tub
- Dropped reference to Method 335.4 from sample preservation; covered in a separate SOP.
- Revised Section 10.5.9.2 to use MSA only when specified for a project under DoD QSM 5.0.
- Revised Section 10.5.14 & 10.5.15 to reflect use of automatic timer.
- Revised Section 10.5.16 wording for clarity and added note for emphasis.
- Revised Section 10.5.19 to store distillates in sample fridge to distinguish from standard fridge
- Added Trouble Shooting and Maintenance to Section 10
- Revised Method Modification 6 to clarify use of ascorbic acid for all methods in place of sodium arsenite.
- Added criteria for DoD QSM 5.0 throughout.
- Removed reference to SM 4500 CN^- - 1997.

- Revision 3, dated 31 August 2013
  - Added TALS reagent module IDs for reagents and standards in section 7.0
  - Revised concentration of Intermediate Standard I to reflect current practice
  - Added note to section 7.9.2 to address potential need for addition of more Brij-35 solution for optimal instrument performance
  - Increased amount of Chloramine-T in section 7.10 to 2.0 g for improved performance
  - Revised sample preservation pH to comply with EPA 2012 MUR for NPDES compliance samples (Methods 334.1 and SM 4500 CN-)
  - Updated MB acceptance criteria to reflect current practice , Sections 9.2 & 9.3
  - Corrected units in section 11.2.4
  - Added new section 11.7 on data upload instructions
  - Revised Section 11.8.4 for clarification in handling data required for measuring the amenable cyanide concentration
  - Added item 11 to Method Modification table
  - Added requirements of 12 QC elements specified in EPA 2012 MUR to 40 CFR Part 136 throughout

- Revision 2, dated 31 August 2012
  - Combined SOP DV-WC-0082 with this SOP and retired SOP DV-WC-0082 highlighting details that are specific to the respective source methods
  - Added Sections 2.3 and 2.4

  - Renumbered and reordered ICAL standards table to be consistent with numbering used at the bench (Section 7.5.2)
  - Updated Section 9 and 10 to clarify which batch QC samples and their frequency are performed for each type of cyanide determined.
  - Revised order in Section 11, added equation for calculation of concentration in solid samples
  - Removed reference to Method 9013
  - Added references for SM 4500-CN^- - 1999 (per 2012 MUR)
  - Updated method modifications table for Methods 9012A and 9012B (#1,3,4) and added modifications 6-10
  - Source method review

- Revision 1.1, dated 18 June 2012
  - Updated section 9.1 for consistency with other SOPs.
  - Updated volumes for spiking LCS/MS/MSD/HLCS and LLCS and concentration of standard used to reduce volumes used.
  - Added section 10.1 and 10.2 for consistency with other SOPs.
  - Revised statement regarding frequency of standard verification in Section 16 consistent with procedure.
  - Corrected typographical errors and section references throughout.

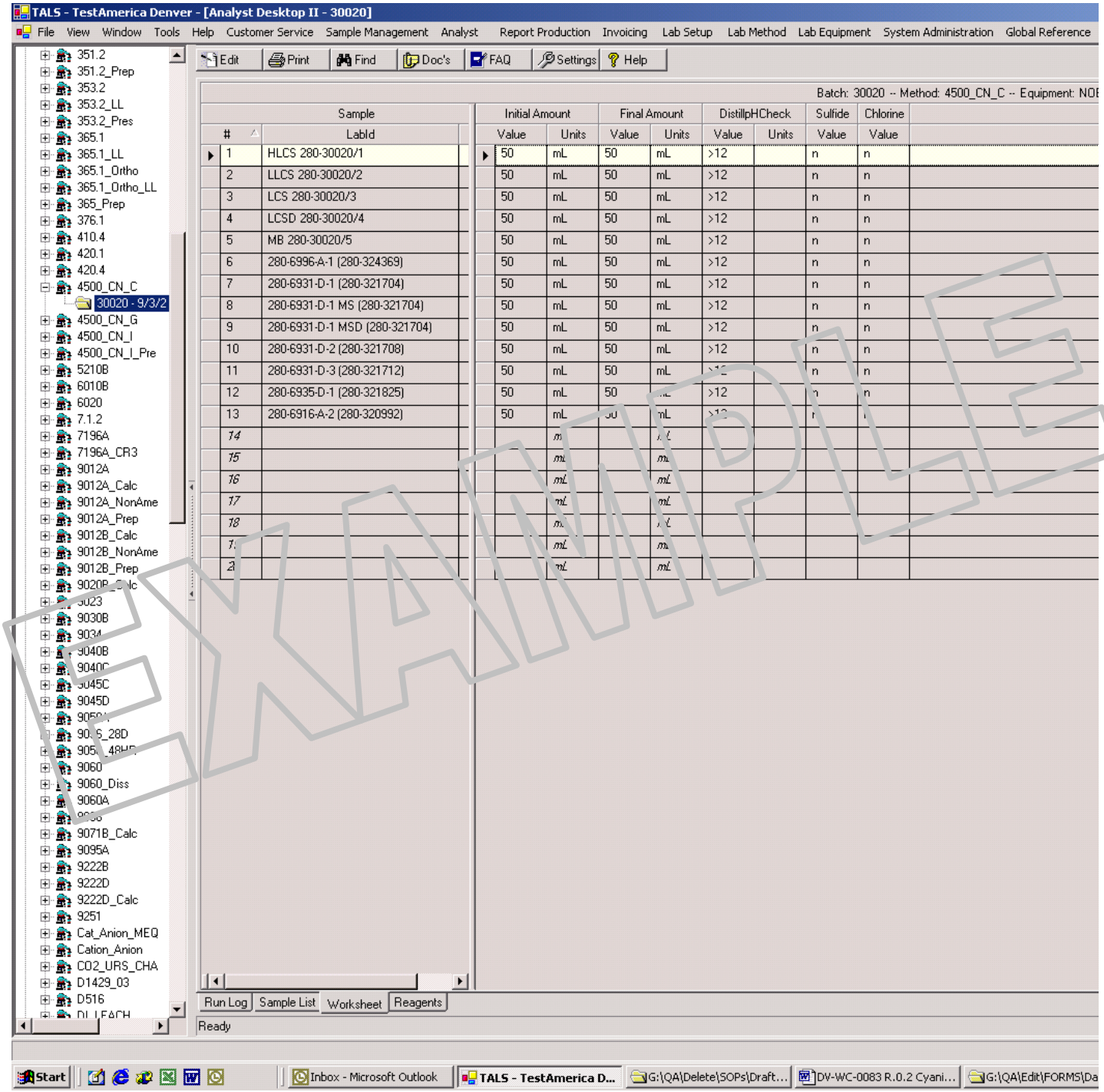
- Revision 1, dated 04 April 2012
  - Source method review
  - Expanded Section 1 to include discussion of differences between Cyanide Amenable to Chlorination and Weak Acid Dissociable Cyanide (WAD). Added WAD and Free Cyanide to list of analytes and listed RLs.
  - Added definitions of the forms of cyanide (Section 3)
  - Clarified when standardization of stock standard is required.
  - Changed “Standards Log” to “Reagent Module” in the LIMS to describe current practice.
  - Added procedural steps for preparation of soil samples and associated QC samples (Section 9 and 10).
  - Added SM requirement to spike LCS and MS/MSD with second source standard
  - Added requirement to protect samples from light during chlorination step
  - Added equation for linear regression and use of that equation for calculation of sample concentration (Section 10 and 11).
  - Reordered Section 11.
  - Added method blank requirement from Standard Methods to IDOC procedure (Section 12.3)
  - Replaced Attachment 1 with example of worksheet in TALS
  - Removed Attachment 2 and renumbered existing attachments
  - Replaced diagram of distillation apparatus with midi-distillation apparatus more comparable to equipment in use.
  - Formatting and grammatical changes throughout

- Revision 0.2, dated 31 March 2011
  - Annual Technical Review
  - Removed the Note from Section 10.6.2 – distillation of curve
  - Corrected grammar and formatting errors
  - Updated Attachment 1 & 2
  - Added Section 11.1 referencing corporate SOP CA-Q-S005, “Calibration Curves”.

Earlier revision histories have been archived and are available upon request.

Attachment 1.

Example Cyanide Preparation Bench Sheet



Attachment 2.

Cyanide Distillation Apparatus

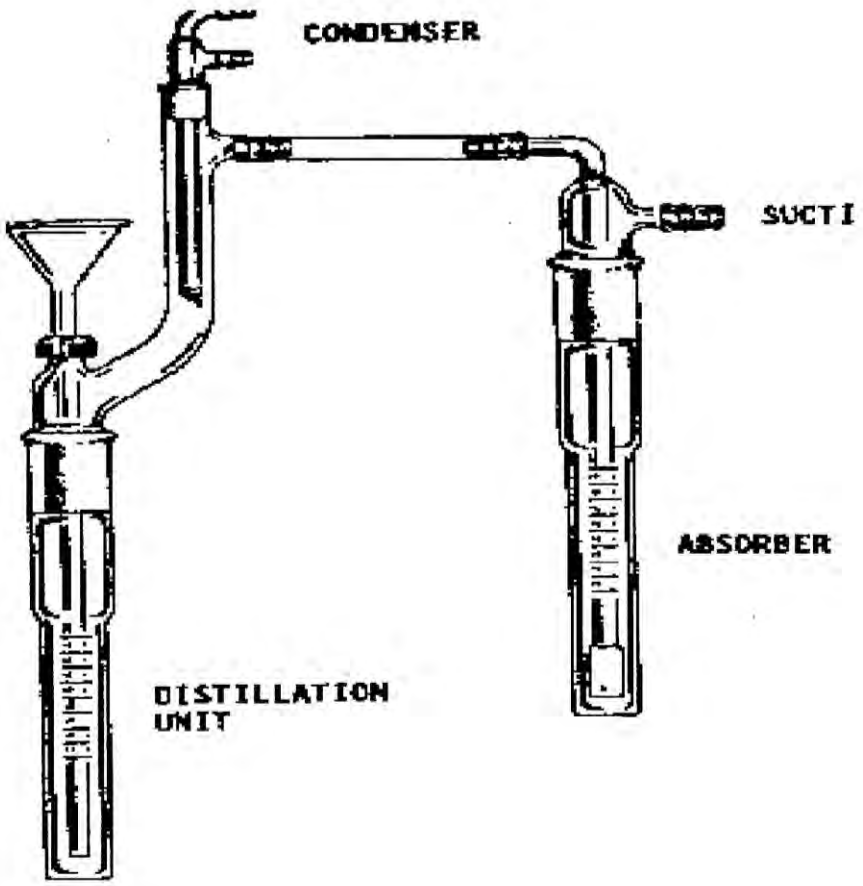
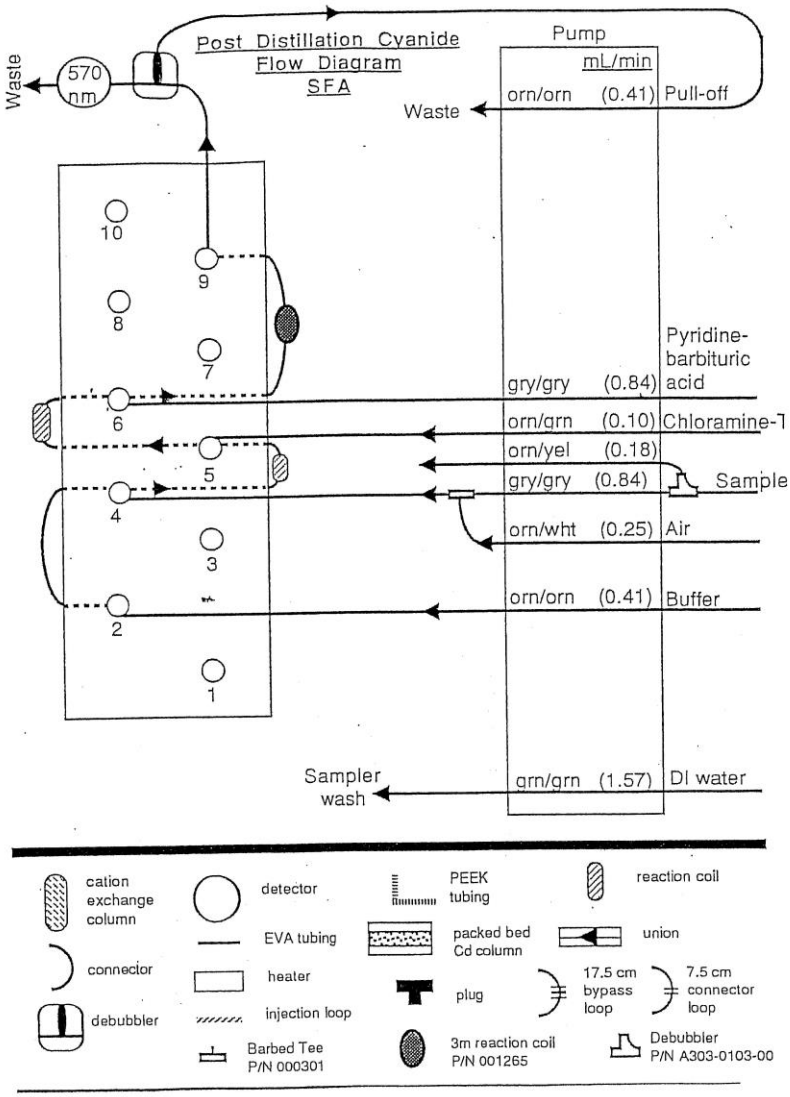


FIGURE 1. MIDI DISTILLATION APPARATUS

335.4-15

Attachment 3.

Alpkem Manifold Schematic



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Acid-Soluble and Acid-Insoluble Sulfides: Distillation and Titration [SW 9030B/ SW9034]

Approvals (Signature/Date):

Connie Jewell — 3/12/19
Department Supervisor — Date

Doug Gomer — 3/15/19
Health & Safety Manager / Coordinator — Date

Roxanne Sullivan — 3/15/19
Quality Assurance Manager — Date

Richard Clinkscales — 3/15/19
Laboratory Director — Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This SOP covers both the sample pretreatment procedure designed to remove interfering substances prior to analysis and the determination of the concentration of acid-soluble sulfide. It is based on SW-846 Methods 9030B and 9034.

1.2 This method does not measure acid-insoluble sulfides.

1.3 This method is applicable to the measurement of total acid-soluble sulfides in drinking water, surface and saline waters, domestic and industrial wastes and solid waste materials.

1.4 The reporting limit for water samples is 4 mg/L and for soil samples is 10 mg/kg. Samples that contain more than 50 ppm sulfide should be diluted.

2.0 Summary of Method

2.1 The sulfide method is a two part method involving the distillation of the sample using a micro-distillation apparatus followed by titration of the collected scrubber solution.

2.2 The separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample in a closed system. The sample is then heated (for total sulfide) to 70 °C. The H_2S which is formed is carried by a nitrogen stream into a zinc acetate/formaldehyde gas scrubbing bottle. Under these conditions it is precipitated out as ZnS.

2.3 The titration is accomplished by the addition of excess iodine to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3.0 Definitions

Please refer to the Glossary of the TestAmerica Denver Quality Assurance Manual and SOP DV-QA-003P for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Aqueous samples should be taken with a minimum of aeration to avoid the oxidation or volatilization of sulfide.

4.2 Reduced sulfur compounds, such as sulfite and bisulfite decompose in acid to form SO_2. The SO_2, if carried over into the scrubbing solution, may yield false positives. The addition of formaldehyde in the scrubber solution removes this interference. This method shows no sensitivity to sulfite or bisulfite at concentrations up to 10 ppm.

4.3 Many metals (e.g., Hg, Cd, Cu) form insoluble sulfides and give low recoveries.

4.4 The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds, both solid and dissolved. These compounds are common in some sample types, such as refinery waste and boiler feed water.

4.5 High levels of color and turbidity may interfere.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.3.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.4 Hydrogen sulfide (H_2S) gas is generated by the addition of sulfuric acid. Inhalation of H_2S gas can cause headache, dizziness, nausea, and unconsciousness and potentially death.

5.3.5 Acid should be added dropwise until the sample reactivity is observed, particularly for soil samples and discolored water samples. If a strong reaction is observed, increase stirring rate and continue to add slowly.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Formaldehyde	Toxic	0.75 ppm (TWA)	Inhalation may result in spasm, inflammation, and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Iodine	Poison Corrosive Oxidizer	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Sulfide	Corrosive	10 ppm (TWA) 15 ppm (STEL)	Will form hydrogen sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
Sodium Hydroxide	Corrosive	2 mg/m^3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Nitrogen gas.

6.2 Boiling Tubes

6.3 Dropping funnels, 50 mL

6.4 Scrubber bottle, 50 mL

6.5 Tygon tubing

6.6 Glass tubing

6.7 Smart-Dist capable of maintaining 70 °C ± 5 °C

6.8 5 mL autopipettor

6.9 Burette, 25 mL, Class A.

6.10 Bubbler Vessel, 50 mL

6.11 Assorted analytical glassware, as needed.

6.12 pH strips

6.13 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 All reagents listed herein shall be ACS Reagent Grade, unless specified otherwise.

7.2 Reagent Water**:** Water that is free of the substances that interfere with the analytical method. Water with a resistivity of 1 megohm-cm or greater. The TestAmerica Denver house deionized water meets this requirement with a minimum resistivity of 10 megohm-cm.

7.3 Reagent sand, such as Ottawa sand for use as blank solid matrix.

7.4 Sulfuric acid, concentrated. (H_2SO_4).

7.5 Hydrochloric acid, concentrated (HCl).

7.6 Sodium Sulfide nonahydrate, $Na_2S \bullet 9H_2O$, crystals (SFD ICV STK or SFD CAL STK)

7.7 Sulfide Standard Solution (Na_2S), approximately 1,000 mg/L (SFD CAL INT or SFD ICV INT)

Select larger pieces of sodium sulfide nonahydrate (SFD CAL STK) rather than small pieces that are more likely to have been oxidized. Spray the crystals with reagent water to remove the cloudy film covering it. Dry with a Kimwipe® as quickly as possible to limit the amount of crystal dissolved then weigh approximately 4 g of the crystals. Record the exact weight of sodium sulfide used. Dissolve the crystals in reagent water and add to a 500 mL volumetric flask to which 2 mL of 50% sodium hydroxide has been added. Dilute to volume with reagent water. The target concentration should be 1,100 – 1,200 mg/L. Store the solution in a glass amber bottle and keep refrigerated when not in use. Replace the standard when the daily standardization concentration is reduced to 800 mg/L.

7.8 Zinc Acetate Dihydrate, (ZINC ACETATE)

Zinc acetate dihydrate, 99.9%, solid, purchased directly from vendor. .

7.9 Zinc Acetate Solution, 0.5 M (znac)

Dissolve 110 g of zinc acetate dihydrate (Section 7.8) in 200 mL of reagent water. Add 1 mL of concentrated HCl and dilute to 1 liter.

7.10 Formaldehyde, 37% (formalin)

Obtain solution from a commercial vendor.

7.11 Hydrochloric Acid, 6 N (1:1) (HCL Sol)

Very carefully and slowly, and with constant mixing, add 250 mL of concentrated hydrochloric acid (HCl) to 250 mL of reagent water and mix. Allow to cool before use.

7.12 Starch Indicator (Starch Ind)

7.12.1 The starch indicator solution is purchased commercially.

7.12.2 Alternatively, to prepare the indicator solution in the lab, dissolve 2 g of laboratory-grade soluble starch and 2.0 g of salicylic acid, as a preservative, in 100 mL of hot reagent water. Mix, cool, and store in a labeled poly bottle.

7.13 Sodium Thiosulfate, 0.0250 N (Na thio)

This solution is purchased as a standardized 0.0250 N solution.

7.14 Iodine Solution, 0.0250 N (Iod)

This solution is purchased as a standardized 0.0250 N solution.

7.15 Sodium Hydroxide (NaOH), 50%

7.15.1 This solution is usually purchased from commercial sources.

7.15.2 If prepared in the lab, very carefully and slowly, with constant stirring, add 250 g of NaOH to 200 mL of reagent water in a beaker.

CAUTION: This solution gets very hot. Add the NaOH to the water slowly to allow the heat to dissipate.

7.15.3 Allow to cool, then transfer the solution to a 500 mL volumetric flask and dilute to volume with reagent water.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE	250 mL	NaOH/Zn Acetate pH>9 Cool, ≤ 6 °C	7 Days	SW-846
Soil	Glass	4 oz. jar	Cool, ≤ 6 °C	7 Days	SW-846

NOTE: Samples should be collected with a minimum of aeration. **The sample bottle should be filled completely, excluding all head space, and sealed.** Analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins.

8.1 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

8.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

8.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0

and 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

8.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via method comments in TALS and in the Quality Assurance Summaries (QAS) available in the public folders.

8.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

8.2 Sample QC - The following quality control samples are prepared with each batch of samples.

8.2.1 Method Blank

A minimum of one method blank must be included in each QC batch of 20 or fewer field samples. The method blank consists of reagent water for batches of aqueous samples or Ottawa sand for batches of solid samples. Prepare and analyze the blank in the same manner as samples.

Acceptance criteria: The result for the method blank must be less than one-half the reporting limit for the analyte of interest or less than 10% of the lowest analyte concentration found in the associated samples, whichever is higher. In the latter case, an NCM must be generated explaining the analyte detection in the blank

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed after first checking all reagents and glassware for sources of contamination, checking the condition and cleanliness of the burette delivering the titrant, and correcting any problems found.

If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

If there is insufficient sample for reanalysis, an NCM must be prepared and the client contacted by the laboratory Project Manager.

8.2.2 Laboratory Control Sample (LCS)

A minimum of one LCS must be included in each QC batch of 20 or fewer field samples. The LCS is prepared by spiking 50 mL of reagent water or 10 g of Ottawa sand with 1 mL of the Sulfide Standard Solution (Section 7.7). The nominal spike concentration for the water LCS is 20 mg/L and for the solid LCS is 100 mg/Kg. The LCS is analyzed in the same manner as the sample distillates. A duplicate LCS (LCSD) may be prepared and analyzed to provide a measure of analytical precision if required by the client or project and in cases where there is insufficient sample to prepare an MS and MSD.

Acceptance criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean and must be no wider than the limits specified in the reference methods.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed. If reanalysis is not possible, an NCM must be prepared and the client contacted by the laboratory Project Manager.

8.2.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair is required with each batch of 20 or fewer samples for total sulfide. The MS and MSD are prepared by adding 1 mL of the Sulfide Standard Solution to the sample aliquot required (Section 7.7). If insufficient sample volume is available for the preparation of an MS/MSD or a sample duplicate, a duplicate LCS (or sample duplicate) must be analyzed.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD, or between the sample and sample duplicate, must be less than

the established RPD limit, which is set at 3 standard deviations above the historical mean.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Record verification of volumetric pipettes and the repipettors used in this procedure in the designated spreadsheets.

10.4 Sample Preparation

10.4.1 Prepare the boiling tube by adding 47 mL of reagent water to a 50 mL bottle. Add 2 mL of zinc acetate solution and 1 mL of 37% formaldehyde (formalin).

10.4.2 Prepare reaction vessels, as follows:

10.4.2.1 Solids and Sludges

10.4.2.1.1 Weigh 10 g of soil or sludge into a boiling flask. Add 50 mL of reagent water and stopper immediately. The sample weight can be reduced to 5 g if the matrix absorbs water or does not stir efficiently.

10.4.2.2 Liquids

10.4.2.2.1 Check the pH of the sample with wide-range pH paper and record on the benchsheet. Shake the sample container to suspend any solids and then quickly transfer 50 mL of sample with a graduated cylinder into a boiling flask.

10.4.3 Assemble the apparatus as shown in Attachment 1. Connect the addition funnel to the reaction flask. Connect the nitrogen stream to the flask as shown in Attachment 1. Be sure all fittings and joints are air-tight.

10.4.4 Adjust the nitrogen flow to about 5 bubbles per second in the scrubber bottle.

10.4.5 Once the gas flow has stabilized, purge the system for 15 minutes.

10.4.6 Add sulfide standard solution by pipette to the LCS and MS/MSD (if MS/MSD are analyzed). Keep the tip of the pipette beneath the water surface and seal the vessel up tightly after addition. Spiking is normally done with 1 mL of sulfide standard solution (Section 7.7).

NOTE: Be sure the location of the MB and LCS are rotated amongst all distillation positions over time to demonstrate cleanliness of all glassware and proper functioning of hotplates. Make a note in either the Batch Comments or in the Worksheet comments regarding the positions used in each batch.

10.4.7 Add 5mL of concentrated sulfuric acid into the top of the addition funnel. Open the addition funnel so that the concentrated sulfuric acid drips slowly into the sample at a rate of approximately 5mL/min. Some samples may react violently with the acid, in which case the addition rate of acid may be decreased.

10.4.8 Allow the reaction to proceed for 90 minutes, checking the temperature frequently. Record the start and end times of the distillation in the batch record.

10.4.9 Once the distillation is complete, use low level pH paper to check the pH of the spent solution in the reaction vessel. If the pH is not less than or equal to 1, reprepare the sample using more acid.

10.4.10 If the pH is less than or equal to 1, close the scrubber bottles and proceed to the analysis outlined in Section 10.6 for total sulfide determination by titration by Method 9034.

10.5 Calibration

10.5.1 Standardization of Sulfide Standard Solution

10.5.1.1 Pipette 20.0 mL of iodine solution (Section 7.14) into a 250 mL Erlenmeyer flask.

10.5.1.2 Add 2 mL of 6 N hydrochloric acid (Section 7.11).

10.5.1.3 Pipette 5.0 mL of the Sulfide Standard Solution (Section 7.7) into the flask, making sure that the delivery tip of the pipette is below the surface of the solution. Dilute to approximately 100 mL with reagent water.

10.5.1.4 Titrate with 0.0250 N sodium thiosulfate solution (Section 7.13) to a pale yellow straw color.

10.5.1.5 Add ~1 mL of starch indicator solution (Section 7.12) and swirl until a homogenous blue color develops.

10.5.1.6 Continue the titration until the blue color disappears. Record the volume of the sodium thiosulfate titrant used in the Sulfide by Titration Bench sheet (Attachment 3).

10.5.1.7 Calculate the sulfide concentration using the Sulfide by Titration Bench sheet (Attachment 3).

10.5.1.8 Perform the standardization titrations in duplicate. Use the average of the two results as the final sulfide concentration of the Sulfide Standard Solution.

10.6 Sample Analysis

10.6.1 Determination of Total Sulfide (Acid-Soluble Sulfides)

10.6.1.1 Add 0.4 mL of 6 N HCl (Section 7.11) to the distilled sample in the Erlenmeyer flask and swirl.

10.6.1.2 Using a calibrated pipette, dispense a known amount of 0.025 N iodine solution (Section 7.14) into the flask. The amount added needs to be in excess of the amount needed to oxidize the sulfide. This is typically 1 mL for "ND" samples and 5 mL for samples with sulfide in the range of the LCS samples. Add enough iodine solution to turn the liquid in the flask a deep amber color. Record the volume of iodine used.

NOTE: There must be enough iodine to oxidize the entire amount of sulfide present as zinc sulfide precipitate in the scrubber solution. Use about 1.5 mL of the 0.0250 N iodine solution for every mg of sulfide estimated to be in the sample. The amount of iodine necessary can be estimated by the amount of precipitate present in the scrubber solution. After addition of the iodine, the solution should be a deep amber color. If, in the analyst's opinion, the amount of iodine needed to oxidize the sulfide present in the sample will take more than 25 mL, a smaller sample aliquot may be taken for analysis. This volume must be measured and recorded.

10.6.1.3 Add a stir bar and place the flask on a magnetic stirrer.

10.6.1.4 Titrate the solution with 0.0250 N sodium thiosulfate solution (Section 7.13) to a pale yellow color.

10.6.1.5 Add sufficient starch indicator solution (Section 7.12) to achieve a deep and homogeneous blue color. Typically this volume is approximately 1 mL.

10.6.1.6 Continue the titration until the blue color just disappears. Allow the sample to equilibrate to ensure the endpoint is not missed.

10.6.1.7 Record the volume of sodium thiosulfate solution used in TALS worksheet.

10.7 Troubleshooting and Maintenance (Distillation)

10.7.1 Ensure the Nitrogen inlet tube is below the sample surface and is not clogged.

10.7.2 Check the ground glass joints for light grease and not excessive grease.

10.7.3 Check the tubing for cracks or signs of frailty or excessive wear.

10.7.4 Ensure the inlet and outlet tubes for the nitrogen are not chipped.

11.0 Calculations / Data Reduction

11.1 Data for the standardization of sulfide standard solution are entered into the designated spreadsheet. Data for titration of samples and QC are entered directly into TALS.

11.2 All calculations are performed in the sulfide titration benchsheet or in TALS. For manual verification use the following calculations.

11.3 One mL of 0.0250 N iodine solution reacts with 0.4 mg of sulfide present in the titration flask. Use the following equation to calculate sulfide concentration:

$$S = \frac{[(A \times B) - (C \times D)] \times 16000}{V_s} \qquad \textit{Equation 1}$$

Where:

S = Concentration of sulfide in sample (mg/kg or mg/L)
A = Volume of iodine solution added (mL)
B = Normality of iodine solution
C = Volume of sodium thiosulfate solution (mL)
D = Normality of sodium thiosulfate solution
V_s = Volume (mL) or weight (g) of original sample aliquot

11.4 If the normality of the iodine solution is exactly the same as that of the sodium thiosulfate solution, i.e., 0.0250 N, the following calculation may be used instead of the equation in section 11.3:

$$S = \frac{(A - C) \times 400}{V_s} \qquad \textit{Equation 2}$$

Where:

S = Concentration of sulfide in sample (mg/kg or mg/L)
A = Volume of iodine solution added (mL)
C = Volume of sodium thiosulfate solution (mL)
V_s = Volume (mL) or weight (g) of original sample aliquot

11.5 LCS Percent Recovery

Use the following equation to calculate the percent recovery of sulfide in the LCS:

$$\text{LCS \%Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\% \qquad \textit{Equation 3}$$

11.6 MS and MSD Percent Recovery

Use the following equation to calculate the percent recovery of the added sulfide in the MS and MSD samples:

$$\text{MS \%Recovery} = \frac{SSR - SR}{SA}$$ *Equation 4*

Where:

SSR = Spiked sample result (concentration of sulfide in the MS or MSD).

SR = Sample result (concentration of sulfide in the parent sample).

SA = Concentration of added spike (concentration of sulfide in the sample in addition to any native sulfide as the result of adding the spiking solution).

11.7 Relative Percent Difference (RPD)

Use the following equation to calculate the relative percent difference between two analytical results (e.g., MS and MSD, LCS and LCSD, sample and sample duplicate).

$$RPD = \frac{\left|(R_1 - R_2)\right|}{1/2(R_1 + R_2)} \times 100\%$$ *Equation 5*

Where:

R_1 = Result for first sample (MS, LCS, or sample).

R_2 = Result for duplicate sample (MSD, LCSD, or sample duplicate).

11.8 All data undergo first and second level review using a checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no true MDL for this method. For the purposes of method detection the smallest increment of titrated volume is calculated and used as the MDL. Whenever a new burette is used the volume is rechecked and put through the calculation. The current MDL value is maintained in TALS.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.2 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.2.3 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.4 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Scrubber waste – Aqueous Acidic (Reactivity) - Waste Stream F

14.2.3 Contents of reaction vessel – Aqueous Acidic (Reactivity) - Waste Stream F

14.2.4 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 9030B: Acid-Soluble and Acid-Insoluble Sulfides: Distillation, Revision 2, December 1996.

15.1.2 Method 9034, Titrimetric Procedure for Acid-Soluble and Acid Insoluble Sulfides, Revision 0, December, 1996.

16.0 Method Modifications

Item	Method	Modification
1	SW 9030B	In this SOP the distillation apparatus uses only one scrubber.
2	SW 9030B	For total sulfide, 5 mL of concentrated sulfuric acid is added to the system without first titrating the sample. After the 90 minute reaction period, the spent sample solution pH is measured. If the pH is not ≤ 1.0, the sample is re-distilled using additional sulfuric acid.
3	SW 9034	Rinse solution of standardized 0.0250 N iodine, 1 mL of 6 N HCl, and reagent water is not used in the above procedure. As samples are preserved with 1% sodium hydroxide and zinc acetate, and the total sulfides are preserved with formaldehyde and zinc acetate, rinsing the bottles with reagent water introduces very minimal loss of sulfide due to oxidation.
4	SW 9034	The sodium thiosulfate and iodine solutions are purchased standardized from a vendor and therefore are not standardized in the laboratory.
5	SW 9034	The Method 9034 procedure is to add the iodine solution into a 500 mL flask, bring to 100 mL with reagent water, add acid, and pipette the scrubber solutions from method 9030 under the iodine solution. The scrubber solutions are approximately 200 mL. Due to the large volume of scrubber solution, the laboratory transfers the scrubber solutions into a flask and then adds the acid and iodine solution.
6	SW 9030B	Soil samples are refrigerated upon receipt and are not preserved with zinc acetate. The laboratory uses the 7 day holding time stated in SW-846. Since sample composition and moisture content vary so widely, it is difficult to preserve the sample in the field as prescribed. It is recommended that the sample be taken with no headspace.
7	SW 9030B	Aqueous samples are measured volumetrically in graduated cylinders, not by weight as indicated in the source method. Generally the liquid samples received by the lab are water rather than non-aqueous wastes.

17.0 Attachments

Attachment 1: Distillation Apparatus
Attachment 2: Example Sample Preparation Benchsheet - TALS
Attachment 3: Example Titration Benchsheet – Excel Spreadsheet
Attachment 4: Example Titration Benchsheet - TALS

18.0 Revision History

- Revision 3, dated 15 March 2019
  - Annual review
  - Updated copyright information.

- Revision 2, dated 01 February 2018
  - Annual review
  - Updated section 10.4 to reflect current procedure

- Revision 1, dated 31 January 2017
  - Updated Section 7.7 to properly reflect current procedure
  - Added new Section 7.8 for solid Zinc Acetate
  - Removed note in section 10.4.1 regarding pipette verification because solutions used are non-quantitative.
  - Updated attachment 3 to exclude pipette verification

- Revision 0, dated 20 December 2016

  This SOP, using a microdistillation apparatus, replaces DV-WC-0042 which used a macrodistillation. The following lists changes from the macrodistillation SOP to convert the method to microdistillation:

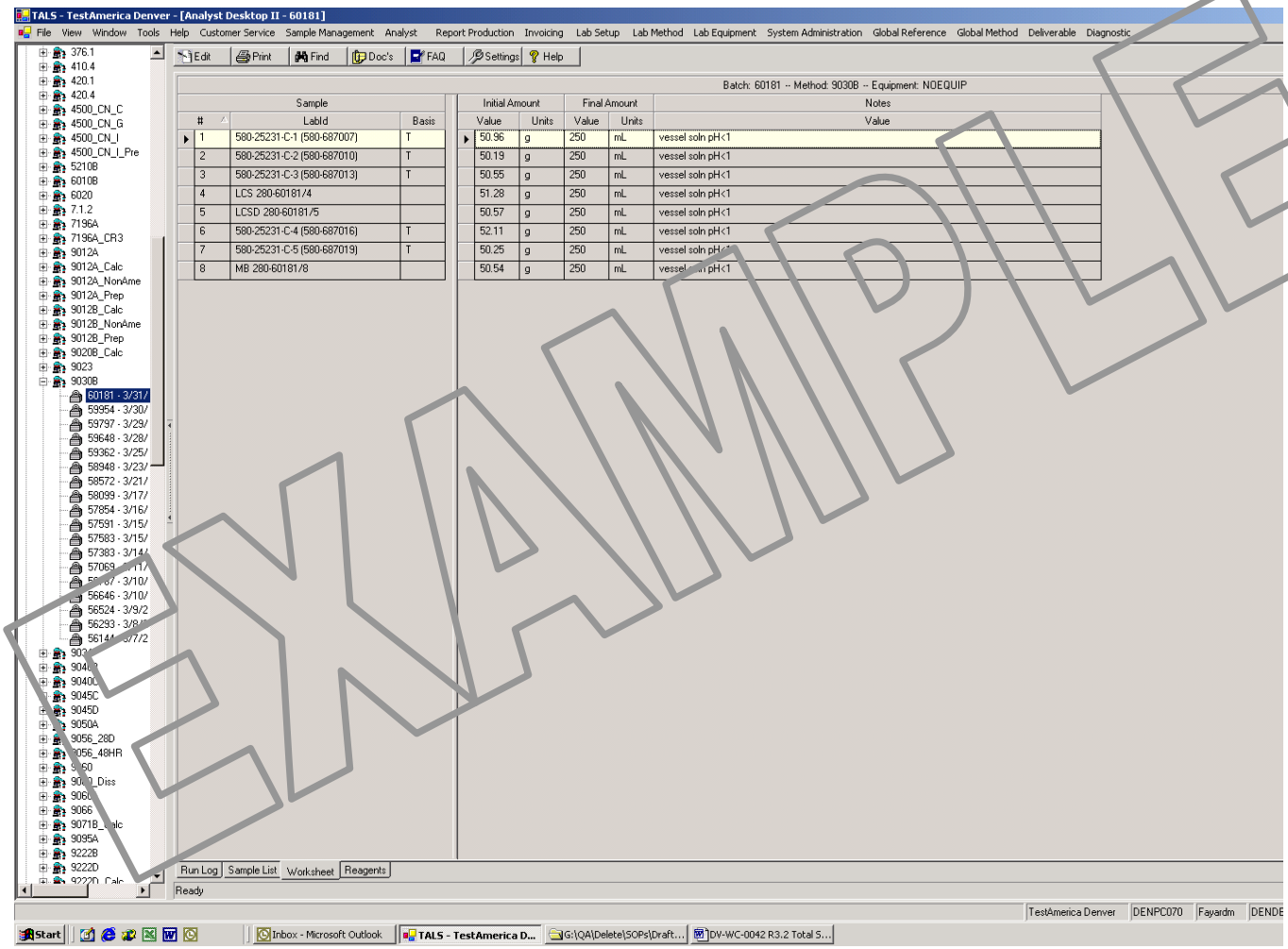
  - Changed LIMS to TALS throughout
  - Corrected section references throughout
  - All volumes used for prep and processing reduced by 5 times from DV-WC-0042.
  - Changed RL for soil to 10 mg/Kg
  - Changed iodide to iodine in Section 2.3
  - Removed comment from Section 4.4 concerning washing precipitate as it is not part of the normal procedure
  - Removed Section 4.6 regarding unpreserved samples
  - Updated Section 6.0 to include micro distillation supplies
  - Added MS/MSD corrective action information to Section 9.2.3 to conform with current procedure
  - Updated Section 10.4 to lower volume used for micro distillation
  - Added note to Section 10.4.1 regarding the calibration of pipette(s)
  - Added new analysts training requirement to Section 12.3
  - Updated Attachment 1 to micro distillation apparatus

Attachment 1.

Distillation Apparatus

Attachment 2.

Example Sample Preparation Benchsheet - TALS



Attachment 3.
Example Titration Benchsheet – Excel Worksheet

Laboratory Bench Sheet — TestAmerica-Denver

Sulfide by Titration

Analyst:		**SOP Information:**	
Date:		Number:	
Titration Solutions		**Calibration Information**	
Solution 1:	Iodine	Source/Ver-Lot#:	
TALS ID		Prep Date:	
Normality:	0.025	Made By:	
Solution 2:	sodium thiosulfate	Concentration:	#DIV/0!
TALS ID		Expiration Date:	
Normality:	0.025		
	Starch Indicator		
TALS ID			

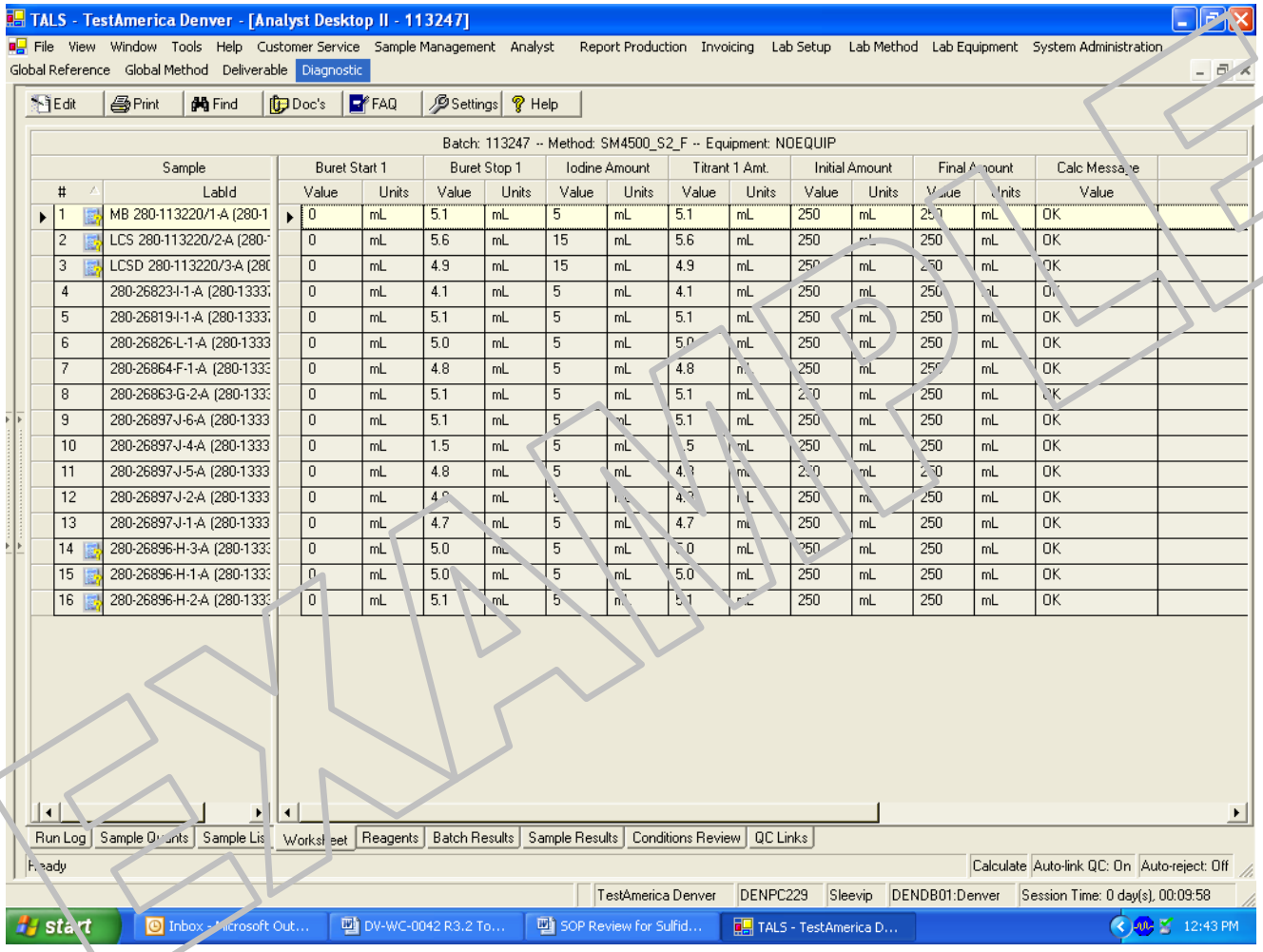
	CAL Volume	Buret Start	Buret End	m Iodin	Final mL	Conc mg/L
CAL					.00	#DIV/0!
CAL					0.00	#DIV/0!

R:\QA\Edit\FORMS\Wet Chemistry\Sulfide\Sulfide by Titration_Master.xls
Revision 6 - 1/31/2017

Page 1 of 1

Attachment 4.

Example Titration Benchsheet - TALS



Eurofins Test America SOPs

Savannah

SAMPLE RECEIPT PROCEDURES

Approvals (Signature/Date):

08/23/2019

Bernard Kirkland — Date
Laboratory Director

08/23/2019

Kimberly Chamberlain — Date
Quality Assurance Manager
Environmental Health & Safety Coordinator

Facility Distribution No. ____1_____ **Distributed To:_TALS File System Shares**

1.0 Purpose

The purpose of this SOP is to describe the routine procedures used for receiving samples into the laboratory, checking the integrity of samples, verifying pH of samples, assigning of a sequential laboratory identification number to the samples, and distributing the samples to the appropriate laboratory department.

This SOP also describes the internal chain-of-custody (ICOC) procedures employed by the laboratory.

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Scope

2.1 The laboratory has a written Sample Acceptance Policy that clearly outlines the circumstances under which samples shall be accepted or rejected. The required elements include:

- All samples must be received in good condition.
- The chain-of-custody (COC) must be filled out completely.
- All samples must be properly labeled.
- Proper sample containers with adequate volume for the analysis and necessary QC must be used.
- All samples must be preserved according to the requirements of the requested analytical method.
- All sample holding times must be met.

Data from samples which do not meet these criteria are qualified using the TALS NonConformance Module and/or the Sample Receipt Checklist.

2.2 Upon arrival in the laboratory, a Triage Checklist is initiated for each shipping container (i.e., cooler) arriving at the laboratory, and coolers are opened and inspected. The Uncorrected and Corrected temperature of each cooler is measured and recorded on the Triage Checklist.

If the integrity of the shipping containers or the sample containers has been compromised, if the cooler temperature is out of range, or if there is a discrepancy between the COC and samples, a notation is made in the Sample Receipt Checklist housed within the TALS.

During Login, the contents of the coolers are checked against the COC. TALS assigns a sequential laboratory identification number (i.e., Job number), which is then recorded on the COC. A sample barcode label containing the Job number and sample designation are affixed to each container to aid in maintaining internal chain-of-custody.

Sample preservation, if applicable, is verified by Sample Receiving Department.

The samples are distributed to the appropriate laboratory department where samples are

relinquished to the department and scanned in using barcode readers. The samples are then properly stored until preparation and/or analysis.

3.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

3.1 Specific Safety Concerns or Requirements

The samples received at the laboratory are unknowns, and the toxicity or carcinogenicity of these samples cannot be precisely defined. Each sample must be treated as a potential health hazard, and exposure to samples must be minimized.

All coolers must initially be opened under a hood or in another well-ventilated area (e.g., outside on the loading dock). If there is no breakage, or no other indications of a potential hazard (such as staining, a foul odor, material safety data sheets enclosed, etc.), then the cooler may be moved inside for processing. If there is sample container breakage and the indication of a potential hazard (e.g., odor, fumes, MSDS/SDS, or annotations on the COC), move the cooler to a hood for processing. Notify the PM, and contact the client, if necessary, to characterize the potential hazard posed by the samples.

Note: Cut resistant gloves must be worn when inspecting coolers upon receipt and when cleaning out coolers. Latex gloves do not provide protection against cuts.

If a material safety data sheet/Safety Data Sheet (MSDS/SDS) is received with the samples, the information in the MSDS/SDS must be reviewed before the samples are processed. A copy of the MSDS/SDS must accompany the samples into the laboratory areas.

Proper lifting techniques must be used when lifting coolers. Note: A fully loaded cooler may require more than one employee to safely lift onto a counter.

3.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on TANet Oasis, and through TALS File System Shares.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Nitric Acid	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2mg/m^3 Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

3.3 Sample Preservative Color Code System

Special sampling containers are designated for each analysis group in the laboratory. The laboratory uses a preservative color-code system as received from vendor (typically, ESS) for laboratory-supplied vials to alert sampling teams, sample receiving personnel, and analysts handling the containers as to which preservative was added to stabilize the sample during shipment and storage.

Note: In some cases, sample containers may be procured from an alternate vendor with a different preservation color system.

4.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

Chain-of Custody (COC) – The COC form (Attachment 1) is used as evidence of documentation of sample collection, shipment, and laboratory receipt. The term "chain-of-custody" can also be used to describe the process of documenting sample location and possession.

A sample is considered "in custody" if it is:
- in actual possession of the sampler or transferee
- in view after being in physical possession of the sampler or transferee
- sealed to maintain sample integrity while in possession of the sampler or transferee
- in a secured area restricted to authorized personnel

The procedures used by Eurofins TestAmerica Savannah are in accordance with this definition of custody. In addition, field samples are tracked in the laboratory using the Sample Internal Custody Log and are tracked in and out of storage areas by use of a barcode system.

Internal Chain-of-Custody (ICOC) – The process used by the laboratory to document a sample's history within the laboratory. ICOC is accomplished by using sample barcode readers and internal COC forms.

Temperature Blank – This bottle (usually a 100mL plastic bottle filled with water) is included in each out-going cooler and is used to verify the temperature of samples upon receipt.

5.0 Procedure

Eurofins TestAmerica Savannah's chain-of-custody procedures allow "cradle to grave"

documentation of a sample's history. The chain-of-custody forms are commonly used as evidentiary documents in legal proceedings

5.1 Sample Handling

Samples must be processed and distributed to the appropriate department as soon as possible after receipt. Every effort should be made to reduce the amount of time samples are in transition (e.g., on the counter, on carts, etc.) to minimize exposure to room temperature. Parameters with short holding times (Attachment 2) and samples with RUSH status should be given priority for distribution.

5.2 Apparatus and Materials

Infra-Red Thermometer ("IR gun") – verified quarterly against a NIST-certified thermometer and verified daily against a digital thermometer in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Kevlar or Blue MAPA gloves

Note: Care should be taken if Sharpie pens are used in the receiving area. These pens contain volatiles, which may contaminate samples. A "Peel-off China Marker" or equivalent "grease" pen may be used for labeling purposes.

5.3 Receipt, Inspection, and Acceptance of Sample Containers

Refer to Attachment 4 for the laboratory's Sample Acceptance Policy.

The process of receiving samples into the laboratory and taking custody of the samples is a dynamic process. Some procedures listed in this section may be performed concurrently, although listed in different subheadings.

Coolers may arrive in the lab via common courier (Federal Express, UPS, etc.), client delivery, courier, or Eurofins TestAmerica field crew.

Foreign soils must be handled in accordance with the Restricted Foreign and Domestic Soil section of the Savannah Addendum to the Environmental Health and Safety Manual (EHSM). Each foreign soil container must have a fluorescent green sticker affixed to the outside that reads **"FOREIGN SOIL - STERILIZE BEFORE DISPOSAL".** Foreign soils must be segregated from other samples during storage and are stored in coolers or bins marked as foreign soils.

As coolers are received, a Triage Checklist form is completed (Attachment 6) for each set of coolers for the same Project/Login.

5.3.1 Cooler and Sample Inspection

Inspect the general condition of the coolers upon receipt. Cut-proof gloves (Kevlar or blue MAPA) must be worn when performing the initial inspection of the cooler contents.

Coolers with custody seals must be checked to ensure that the seal is in place. This verification should be documented on the TALS Sample Receipt Checklist. All coolers must be inspected for signs of tampering and damage. If the custody seal has been compromised or if signs of damage or tampering are evident, the Project Manager must be notified immediately and the situation noted in the TALS Sample Receipt Checklist.

Note: Coolers that are known to contain RUSH status samples and/or samples with short holding times (Attachment 2) must be given priority. Labels may be affixed to the coolers that indicate short holds or rush samples are included.

Open the coolers and inspect the general condition of the contents.

Upon receipt, complete the triage checklist and check the coolers for:

- sample container temperature
- the presence of a chain-of-custody form
- method of receipt

5.3.2 Temperature Verification

Cooler temperature is verified as follows:

Select a container from the cooler at random to verify cooler temperature. If this container's temperature is within limits, the cooler is considered to be acceptable. If this container's temperature is not within limits, and there is evidence of cooling, carefully remove the remaining containers from the cooler to attempt to locate the temperature blank bottle. If provided, the temperature blank bottle is then used to verify temperature.

Note: Try to avoid labels, lids, or other shiny surfaces, as the IR gun reading may be adversely affected.

Record the cooler temperature on the COC, triage sheet and in the Login Screen within the TALS.

Note: All thermometers are assigned a Correction Factor. Both the Uncorrected then the Corrected Temperature must be recorded.

If the temperature is outside criteria, record this information on the Sample Receipt Checklist. An NCM must be generated to denote this situation.

Email the PM daily with a listing of all coolers received outside the acceptable temperature limits. The PM will notify the client to determine if the laboratory should proceed.

Note: If a "temperature bottle" is received frozen, the temperature is measured using a client sample. This situation must be documented on the Sample Receipt Checklist.

Note: The acceptance temperature for coolers is less than 6°C with no frozen samples. This criterion is used as the default criteria. Some states, programs, methods, and clients may specify other acceptance criteria.

Note: WVDEP does not allow the use of a temperature blank as a proxy for samples when verifying thermal preservation. Temperature of all sample containers for compliance samples originating from West Virginia must be verified immediately upon receipt. This information must be recorded on the West Virginia Sample Temperature Form (Attachment 7), and this form must be scanned and attached to the TALS login. Following temperature verification, WV compliance samples must be transferred to a refrigerator in the Custody Department, where they will remain until retrieved by the appropriate laboratory department personnel.

5.3.2.1 Evidence of Cooling for Same Day Sampling Events

For samples that are received into the lab immediately or soon after sampling, the cooler and sample temperature will most likely not be less than 6°C. This situation must be documented on the Sample Receipt Checklist, and an NCM must be generated to denote the situation.

5.3.3 Chain-of-Custody Reconciliation

Compare the contents of the cooler to the COC. If there are discrepancies between the COC and samples, the sample receipt personnel must notify the assigned Project Manager by initiating a TALS NCM, and recording this information in the Sample Receipt Checklist.

- If no COC has been included in the cooler or has not been sent with the project samples, the sample receipt personnel must notify the Project Manager by initiating a TALS NCM. This deficiency must be noted in the Sample Receipt Checklist.

- If sample containers are missing or have been broken or damaged beyond use, the sample receipt personnel must notify the Project Manager by initiating a TALS NCM. This deficiency must be noted in the Sample Receipt Checklist.

- If the sample containers are not labeled or if the sample identification cannot be determined, the sample receipt personnel must notify the Project Manager by initiating a TALS NCM. This deficiency must be noted in the Sample Receipt Checklist.

- If the sample container label does not match the COC, the sample receipt personnel must notify the Project Manager by initiating a TALS NCM. This deficiency must be noted in the Login Comments and the Sample Receipt Checklist.

- Note: WVDEP requires verification of field filtration for dissolved hexavalent chromium samples. COCs must be evaluated for state of origin, and if hexavalent chromium samples are received from WV, COCs must be further evaluated for evidence of field filtration. If field filtration for these samples is not evident, an NCM must be initiated to

denote this situation.

5.4 Job Number Assignment and Initiation of Login

TALS assigns a laboratory identification number (Job number) to each group of samples. Record this Job number on the COC. The Job number will be affixed to each sample container via a barcode sticker.

TALS assigns Job numbers sequentially with the first three digits equivalent to the laboratory's location code. Eurofins TestAmerica Savannah's job numbers will all have a prefix of 680-. This "680" is commonly truncated when referring to current Job numbers. Refer to Attachment 3 for an example of laboratory codes.

Once the Job number is assigned, each sample is sequentially numbered with a sample number. This sample number (e.g., 680-12345-1) is referred to as the lab sample ID.

Each container associated with a sample number is assigned a sequential alphabet character id. This letter (e.g., 680-12345-A-1) allows the laboratory to determine which bottle was used to perform an analysis.

5.5 Barcode Label Generation

TALS barcode labels for all samples in the Project are printed and affixed to the outside of each container. The barcode label contains the following information:

- the project Job number (e.g., 680-39376)
- the sample number (e.g., 680-39376-1)
- the sample designation for each container for a particular field sample (e.g., 680-39376-A-1, 680-39376-B-1, 680-39376-C-1, etc.)
- a barcode relating this information to the TALS login
- unique bottle number (680-1212681)
- client sample id (e.g., DISCHARGE POND)
- original sample location (e.g., SAV Sample Custody Area, SAV GE/ME Cart, SAV EX Cart, SAV % Solids Lab)
- bottle type (e.g., Plastic 500mL – unpreserved)

5.6 Sample Preservation Checks

Samples collected in preserved containers are verified upon receipt, using pH paper

strips, to ensure the pH is within acceptable range. Samples/methods requiring acid preservation (i.e., sulfuric acid, nitric acid, or hydrochloric acid) typically require pH<2. Samples/methods requiring basic preservation (i.e., sodium hydroxide) typically require pH>9.

Sample pH must be recorded in TALS in a PRESERV_CHK batch. Any pH excursions must be noted on the Sample Receipt Checklist in TALS, and a distinctive new sticker is added to the container to notify the laboratory of the preservation discrepancy, so that the analyst can make any necessary adjustments upon receipt to the department. Currently the laboratory uses a black dot sticker appended to the lid of the container to denote samples that did not meet pH requirements.

Note: Due to the method-specific requirements and/or the volatile nature of the analytes being tested, pH checks for the parameters listed below are not performed by the Sample Receiving Department. Preservation checks for these parameters are performed by the associated laboratory departments:

- Volatiles via GC/MS or GC that are considered zero headspace methods
- AOX and TOX
- Oil and Grease
- Sulfide
- TOC
- Drinking Water Methods
- Bottles logged in at other Eurofns TestAmerica laboratories and Workshared to Savannah location.

5.7 Distribution of Samples to the Laboratory and Internal Chain-of-Custody (ICOC)

After the samples have been inspected and accepted by the Sample Receiving Department, they are distributed to the appropriate laboratory department. The laboratory department must scan the containers into their specific locations using the TALS Internal Chain of Custody Program.

Due to storage requirements, samples are picked up from the custody department during the day/shift and transported to the appropriate departments and moved to refrigerated storage.

To help ensure holding times are met, a courtesy email must be sent to the affected laboratory departments to notify them that samples are available for distribution.

5.7.1 Sample Receipt and Distribution to the Laboratory Departments

The analytical SOPs contain information for the receipt of samples into each of the laboratory departments. These instructions include information on preservation and residual chlorine checks, as applicable.

5.7.1.1 If there is a discrepancy in the number and/or types of samples received, contact the Project Manager or Sample Receiving Department via TALS NCM to resolve the discrepancy immediately.

5.7.2 Barcode Tracking

Internal COC for client samples is accomplished using the bar code readers and the TALS Internal Chain of Custody Program.

5.7.2.1 Scan the samples into the department storage area using the barcode reader. Track the location of each sample using the barcode reader when:

- the sample is removed from the storage area
- the sample is returned to the storage area after use
- the sample container is empty or consumed
- the sample is removed from the storage area for disposal

5.7.2.2 The barcode entries are tabulated by the TALS and can be used to generate a report documenting the sample's history in the laboratory.

Note: The barcode system is used to track original sample containers only – it is not used to track sample extracts. Refer to Section 5.6.3 for procedures to be used to track extract internal COC.

5.7.3 Samples Which Require a Higher Level of Internal COC (extract/digestate ICOC)

Currently the laboratory only maintains a formal ICOC program for original client sample containers. Sample extracts/digestates cannot currently be scanned using the TALS ICOC program, and ICOC for these types of containers is not performed unless specifically requested to do so by the client in the pre-project planning stages (client QAPP, etc.). For those samples that require a higher level of internal chain-of-custody documentation (i.e., extract ICOC), a worksheet note, in the form of a Login Comment, must be used to communicate this requirement to the laboratory.

5.8 Project Management Process

Copies of COCs and receipt information are routed to the Project Management staff. COCs are scanned into the system using a TALS generated label, and loaded into the Document Manger section of TALS. Any sample receipt issues must be addressed and brought to the attention of the client, as applicable.

Project Managers and Project Manager Assistants review samples that have been logged into the TALS in accordance with SOP SA-PM-001: *Project Management.*

6.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based

on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

7.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

7.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Samples are disposed of in the laboratory areas.
- Broken samples must be disposed of in the same manner as other samples.

8.0 Responsibilities

8.1 The Sample Receiving Department Supervisor is responsible for:

- training and supervision of custody technicians/Receiving Department
- maintenance of chain-of-custody records
- identification, documentation, and project management notification of all non-conformances associated with sample receipt
- identification and implementation of all non-routine custody procedures

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

8.2 Sample Receiving Department personnel are responsible for:

- inspection of cooler condition and sample integrity
- unpacking field samples
- documentation of sample receipt into the laboratory
- distributing field samples to the appropriate laboratory department
- logging samples into the Laboratory Information Management System (TALS)
- verifying sample preservation (i.e., pH) is acceptable.

8.3 Project Management personnel are responsible for:

- notifying the client of non-conformances associated with samples
- notifying laboratory personnel of client-specific or state/program-specific requirements such as non-routine hold times, extract ICOC requirements, etc.
- Loading COCs into TALS

8.4 Laboratory personnel are responsible for:

- receiving samples from custody personnel
- where applicable, checking the chemical preservative of the field samples
- properly storing the samples until the time of analysis
- documenting any non-conformances and notifying the project manager or designee
- using the barcode reader to document each sample's location in the laboratory
- using proper internal chain-of-custody procedures as outlined in this SOP

9.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-PM-001: *Project Management*
- SOP SA-QA-006*: Training Procedures*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

10.0 Attachments

Attachment 1: Eurofins TestAmerica Savannah Chain of Custody
Attachment 2: Analyses with Short Holding Times
Attachment 3: Laboratory Codes
Attachment 4: Sample Acceptance Policy
Attachment 5: After Hours Sample Receipt Form
Attachment 6: Eurofins TestAmerica Savannah Triage Checklist
Attachment 7: West Virginia Sample Temperature Check Form

11.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated Attachments to current documents.

Attachment 1:
Eurofins TestAmerica Savannah Chain-of-Custody Form

Eurofins TestAmerica, Savannah — **Chain of Custody Record**

5102 LaRoche Avenue
Savannah, GA 31404-6019
phone 912.354.7858 fax 912.352.0165

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

TestAmerica Laboratories, Inc. d/b/a Eurofins TestAmerica

Project Manager:

Client Contact	Email:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	_____ of _____ COCs
Address	Analysis Turnaround Time			Sampler:
City/State/Zip	☐ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
(xxx) xxx-xxxx Phone	TAT if different from Below _____			Walk-in Client:
(xxx) xxx-xxxx FAX	☐ 2 weeks			Lab Sampling:
Project Name:	☐ 1 week			
Site:	☐ 2 days			Job / SDG No.:
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ Return to Client ☐ Disposal by Lab ☐ Archive for _____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd:_____ Corr'd:_____ Therm ID No.:_____

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.23, dated 4/16/2019

Attachment 2:
Analyses with Short Holding Times

ANALYSIS	METHOD	HOLDING TIME	DEPARTMENT
BOD	405.1, 5210B	48 Hours	GENERALS
C-BOD	5210B	48 Hours	GENERALS
COLOR	110.2, 2120B	48 Hours	GENERALS
ENCORE SOIL SAMPLERS	8015B or 8015C (GRO), 8260B	48 Hours	VOLATILES
FERROUS IRON (Fe^{2+})	3500-Fe B or 3500-Fe D	Immediately	GENERALS
HEXAVALENT CHROMIUM (CR $^{6+}$)	7196A, 3500-Cr B or 3500-Cr D	24 Hours	GENERALS
MBAS	425.1, 5540C	48 Hours	EXTRACTIONS
NITRATE (NO_3)	353.2, 300.0, or 9056A	48 Hours	GENERALS
NITRITE (NO_2)	353.2, 300.0, or 9056A	48 Hours	GENERALS
ODOR	140.1, 2150B	24 Hours	GENERALS
ORTHOPHOSPHATE (PO_4)	365.1, 365.2, 4500-P F	48 Hours	GENERALS
pH	150.1, 4500-H^+ B	15 Minutes	GENERALS
pH	9040B or 9040C	24 Hours	GENERALS
RESIDUAL CHLORINE (Cl_2)	330.3, 4500-Cl^- B	15 Minutes	GENERALS
SETTLEABLE SOLIDS	2540F	48 Hours	GENERALS
SULFITE (SO_3)	377.1,	24 Hours	GENERALS
SULFITE (SO_3)	4500-SO_3 B	15 Minutes	GENERALS
TURBIDITY	180.1, 2130B	48 Hours	GENERALS
UV ABSORPTION (UV254 / SUVA)	5910B	48 Hours	GENERALS
VOLATILE ORGANIC COMPOUNDS, UNPRESERVED	524.2	24 Hours	VOLATILES
VOLATILE ORGANIC COMPOUNDS, UNPRESERVED	624	3 Days	VOLATILES
VOLATILE ORGANIC COMPOUNDS, UNPRESERVED	8015B or 8015C (GRO), 8260B	7 Days	VOLATILES

Attachment 3:
Laboratory Codes

Location Code	Location
140	Knoxville
160	St. Louis
180	Pittsburgh
190	Michigan
200	Burlington
220	Connecticut
230	Anchorage
240	Canton
250	Portland
280	Denver
300	Richland
310	Cedar Falls
320	Sacramento
360	Westfield
370	Honolulu
400	Pensacola
440	Irvine
460	Edison
480	Buffalo
490	Nashville
500	Chicago
550	Phoenix
560	Corpus Christi
580	Seattle
590	Spokane
600	Houston
640	Tallahassee
660	Tampa
680	Savannah
700	Mobile
720	Pleasanton

Attachment 4:
Sample Acceptance Policy

SAMPLE HANDLING GUIDELINES

Thank you for choosing TestAmerica! This bottle kit contains the containers you requested, with the proper preservatives, sample labels, Chains-of-Custody (COCs), and packing materials. Following the guidelines below will ensure we process your samples efficiently upon arrival at the laboratory. If you have questions, please contact a member of the TestAmerica client services team.

- Use permanent ink on all COCs and container labels. Information listed on each container must match the information on the COC. Container labels and COCs must be filled out completely. Refer to the example completed COC on the back of this form. Circled items are critical items that must be filled out.
- When filling bottles, do not overfill and rinse out the preservative. Fill VOA vials completely to avoid headspace. If provided, list trip blanks on the COC.
- All glass containers should be returned in the bubble wrap bags provided, and packed carefully in the cooler to reduce breakage. Avoid stacking glass containers on top of each other.
- Cut resistant gloves must be worn when reaching into coolers.
- Samples must be packed in ice for transport in order to keep them at a stable temperature. Samples arriving with a temperature greater than 6°C will be noted as being outside the laboratory's acceptable temperature range. Note: Ice packs ("blue ice") are often insufficient to maintain the necessary cooler temperature.
- Many analyses have "holding times" of 7 days or less; therefore, it is crucial that you return your samples to us by overnight express shipping service. If you are shipping containers to arrive on a Saturday, be certain to select the "Saturday Delivery" option. We are open to receive samples Monday - Saturday (except for holidays).
- **Note: The VOA vials contained in TerraCore and Encore kits have been pre-weighed prior to shipment. Pre-printed labels will not be provided for these containers. Do not affix any additional labels on these vials as this affects the tare weight and will bias the final results.**
- **Note: All shipping stickers/labels affixed to outside of the cooler must be removed prior to shipment back to the laboratory.**

Sample Acceptance Policy:
All samples will be evaluated against the criteria listed below. Samples which do not meet the criteria listed below will be qualified using the TALS NonConformance Module and/or Sample Receipt Checklist.

1) Samples must arrive in good condition with a Chain-of-Custody (COC) filled out completely.
2) Samples must be properly labeled.
3) Samples must be in proper containers with adequate volume for the analysis. Aqueous samples submitted for Volatiles analyses must be submitted without headspace. Samples must be dechlorinated and submitted with proper chemical preservation (pH) as required by the analytical test method.
4) Most analytical methods require chilling samples to 4°C. These criteria are met if the samples are chilled to below 6°C and above freezing. Note: Samples hand-delivered to the laboratory immediately after collection are only considered acceptable if there is evidence that the chilling process has begun (i.e., arrival on ice).
5) Samples must be prepared and analyzed with the holding times defined in the analytical test method.

Attachment 5: After Hours Sample Receipt Form

After Hours Sample Receipt Form

Procedure:

When a client brings samples to the laboratory outside normal business hours, the following steps must be taken to maintain proper chain-of-custody procedures and to verify thermal preservation:

1. Meet client in Sample Receiving area (i.e., CU Dept).
2. Initiate After Hours Sample Receipt Form and fill out Sample Receipt Documentation section.
3. Sign COC in "Relinquished To" field. Enter date and time in "Date/Time" field.
4. Utilizing proper PPE, measure cooler temperature using a calibrated IR Thermometer.
   - Select a container at random to verify cooler temperature if the Temperature Blank Bottle is not apparent when the cooler is opened.
   - If using glass bottle, aim IR thermometer at bottle label.
5. Record uncorrected and corrected cooler temperature on COC and form.
6. Give copy of executed COC back to client. If multi-page COC, give last page to the client. If single page COC, make a copy on the copier.
7. Place COC and After Hours Sample Receipt Form in plastic bag and place in the cooler.
8. Place cooler in CUR1 or Walk-in (EXR3 or GER2) Refrigerator.
9. Email "Savannah – Ship/Rec Department" that samples arrived after hours and give location of cooler.

Sample Receipt Documentation:

Employee Name: _____

Date: _____ Time: _____

Client Name: _____

Received on Ice? _____Yes _____No

Cooler Temperature:

Uncorrected _____°C Corrected _____°C

Thermometer ID: _____

Refrigerator ID: _____

FCU062.03.22.17:5

eurofins | Environment Testing TestAmerica

Attachment 6:
TestAmerica Savannah Triage Checklist

TestAmerica Savannah Triage Checklist

- ☐ Date: _____
- ☐ Time:_____
- ☐ Checked By:_____
- ☐ Number of Coolers:_____
- ☐ Cooler Type: __Hard __ Styrofoam ___ Box
- ☐ Ice Type: Wet Dry GelPack None Other____
- ☐ Received Via: Fed-Ex ( ) UPS ( ) Bus
  Client Drop Off US Mail Courier
  Other
- ☐ Client: _____
- ☐ Thermometer ID: _____
- ☐ Uncorrected Cooler Temps (°C):_____
- ☐ Correction Factor:_____
- ☐ Corrected Cooler Temps (°C): _____
- ☐ Other/ Comments:

  _____

  _____
- ☐ West Virginia – Yes / No
- ☐ Foreign soil – Yes / No

FCU066:12.27.18:10

eurofins | Environment Testing TestAmerica

Attachment 7: West Virginia Sample Temperature Check Form

eurofins | Environment Testing TestAmerica

Savannah
WV Sample Temperature Check
FCU068:11.02.17:0

INSTRUCTIONS: The temperature of ALL samples received from the state of West Virginia must be recorded on this form, and this document must then be scanned and attached to the TALS Login.

Sample Receiving Tech Initials: _____ IR Gun # and Correction Factor: _____

Sample ID	Bottle Type	Uncorrected Temperature (°C)	Corrected Temperature (°C)

LIQUID EXTRACTION PROCEDURE: CONTINUOUS LIQUID-LIQUID

(Methods: EPA 3520C, EPA 3520C_LVI, and EPA 600-series)

Approvals (Signature/Date):

Whitney Palefsky — March 4, 2019
Whitney Palefsky — Date
Quality Assurance Manager

Kimberly Chamberlain — March 5, 2019
Kim Chamberlain — Date
Technical Director/Department Manager
Environmental Health & Safety Coordinator

Facility Distribution No. 1 **Distributed To: TALS File System Shares**

1.0 Scope and Application

This SOP gives the procedures for extracting semivolatile organic compounds (SVOCs) in water samples via continuous liquid-liquid extraction (CLLE) and Zymark TurboVap concentration technique. This procedure can be performed using standard volume (i.e., 1000mL) or low volume (i.e., 250mL). The low volume procedure is referred to as LVI (i.e., low-volume initiative).

The following classes of SVOCs can be extracted using the procedures outlined in this SOP:

Fraction	Analytical Method	SOP #
Organochlorine Pesticides & PCBs	EPA 608 EPA 8081B EPA 8082A	SOP SA-SG-045
Diesel Range Organics Oil Range Organics (DRO and ORO)	EPA 8015C	SOP SA-SG-070
Polychlorinated Biphenyls	EPA 680	SOP SA-SM-007
Base Neutrals / Acids & PAHs	EPA 625 EPA 8270D EPA 8270D_LL	SOP SA-SM-033

This SOP includes the Work Instructions outlining the extract clean-up procedures employed by the laboratory. These clean-up procedures may include copper (sulfur) and sulfuric acid as outlined in Attachment 7 through Attachment 8.

A complete target analyte lists, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Extraction Procedures

2.1.1 Continuous Liquid-Liquid Extraction (CLLE) Procedure

A known volume of sample is transferred to a continuous liquid-liquid extractor (CLLE), is adjusted to a specific pH if required by the analytical method, and extracted using the solvent and conditions specified in Attachment 5. The extract is concentrated to an appropriate final volume using the Zymark TurboVap.

2.2 Concentration Procedures

2.2.1 Zymark TurboVap Concentration Procedure

After the CLLE procedure is completed, the solvent portion in the round bottom flask is transferred to a glass Zymark concentration tube. The tube is placed in the Zymark TurboVap

concentration device, which has been heated to a specified temperature. A stream of nitrogen is directed into the tube to evaporate the solvent and to concentrate the target compounds. When the volume of solvent reaches the specified volume, normally 1mL, the nitrogen stream is automatically stopped. An alarm sounds to alert the analyst, and the extract is removed from the device and transferred to a storage vial.

If a solvent exchange is required, the exchange solvent is added to the tube, the solvent is evaporated to the specified final volume, and the extract is transferred to a storage vial.

The extracts are stored at 0-6°C until the time of analysis.

2.3 Extract Clean-up Procedures

Copper, and/or acid clean-up procedures can be performed to remove interferences from extracts as outlined in Attachment 7 through Attachment 8 of this SOP.

2.4 Method References

This SOP is based on the following methods: EPA 3520C (CLLE), and the extractions sections of EPA 608, EPA 625, and EPA 680.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

After cleaning the CLLE apparatus, inspect the glassware for the presence of water, especially in the small tubing of the extractor. Water can block the solvent return tube and prevent efficient extraction of the sample. A thorough acetone rinsing will help to eliminate or minimize this problem.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Acetone, hexane, methylene chloride, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. The clean-ups employed by the laboratory may include sulfuric acid and copper (sulfur) clean-ups as outlined in Attachment 7 through Attachment 8.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be processed first.

4.2.3 Samples with large amounts of sediments or particulate may clog the solvent return line of the CLLE. A layer of glass wool, placed in the bottom of the extractor, may be helpful when sediments or particulate are present.

4.2.4 During extract cleanup an emulsion may form in the acid/solvent interface. A small quantity of sodium sulfate or sodium chloride may be gently added to the emulsion. The salt will generally cause the emulsion to break up.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Methylene chloride is a carcinogen and an irritant. It causes irritation to the respiratory tract and has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Methylene chloride may be absorbed through the skin and can cause irritation and pain to the skin and eyes.

Hexane and acetone are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Sodium hydroxide is a severe corrosive. Contact with the skin can cause irritation or severe burns and scarring. Contact with the eyes can cause irritation, burns, permanent vision impairment or even blindness.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Compressed gasses have specific hazards. The employee must be familiar with the MSDS/SDS for each of the compressed gasses. The employee must also be familiar with the compressed gas section (Section 11) of the Environmental Health and Safety Manual.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” button on the Oasis homepage and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2mg/m^3 Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

6.1.1 CLLE-specific Equipment/Supplies

Continuous Liquid-Liquid Extractor – with compatible condenser, extractor body, and round bottom receiving flask

Heating Mantle and Adjustable Support – The heating mantle must be connected to a rheostat to control the temperature.

Rheostat or Variable Transformer

Receiving Flask – 250mL with Teflon stoppers

Boiling Stones

6.1.2 Concentration-specific Equipment

Zymark TurboVap II concentration device or equivalent – The instrument must be vented into an operating fume hood.

Concentration Tubes – Compatible with Zymark apparatus; 200mL with 1.0mL tip

6.1.3 Support Equipment

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*

Thermometers – Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*

Oven – Capable of maintaining approximately 400°C.

Magnetic Stirrer

6.2 Volumetric Labware

Volumetric Containers and Dispensers – Verify in accordance with SOP SA-AN-100: Laboratory Support Equipment (Verification and Use). Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative or Qualitative)	Use	Verification Frequency	Verification Criteria
Glass Transfer Pipettes	Various	Qualitative	Transfer of samples/extracts	None	None
Class A Volumetric Flasks	Various	QUANTITATIVE	Preparation of spiking mixes	None (Class A)	None (Class A)
Class A Graduated Cylinder	Various	QUANTITATIVE	Determination of initial volumes for QC and samples using reduced volumes	None (Class A)	None (Class A)

Glass Disposable Pipettes	Various	QUANTITATIVE	Determination of final volumes and spike volumes	Per Lot	Accuracy = 2% Precision = 1%
Extract Vials	12mL	Qualitative	Pest/PCB Extract Storage	None	None
Autosampler Vials	2mL	QUANTITATIVE	Final volume determination (SVOCs and DRO)	Per Lot	Accuracy = 2% Precision = 1%
VOA Vials	43mL	Qualitative	Standard Storage	None	None
Gas Tight Syringes	Various	QUANTITATIVE	Determination/delivery of spike volumes	None, if received w/ COA	None, if received w/ COA
Pump-Style Pipettes	Various	Qualitative	Delivery of acids/bases for pH adjustments and solvents.	Initially, upon 1st use	Accuracy = 10% Precision = 10%
Medicine Cups	30mL	Qualitative	Sample containment for pH and residual chlorine checks	None	None

6.3 Laboratory Supplies

pH paper – Narrow range.

Residual Chlorine Check Strips – Potassium Iodide starch strips used to detect residual chlorine. Store in original, capped container. Use within the manufacturer's expiration date.

Filter Paper, grade 414 – 18.5cm diameter

Glass Funnels

Pyrex Glass Wool – rinsed with methylene chloride prior to use

11mm crimp-type capper and decapper

Detergent – Barmaid or equivalent

Shallow baking trays

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. The routine sample collection containers supplied by the laboratory are:

Low Volume Procedure:
250mL amber glass container- purchased with Certificate of Analysis attesting to purity

High Volume Procedure:
1L amber glass container– purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.

7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.

7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date or 6 months from the date opened, whichever is sooner.

7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.

7.1.5 The expiration date for prepared standards is 3 months from the date prepared or the expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Acetone, hexane, methylene chloride, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Purchased Reagents

7.2.1.1 Laboratory Reagent Water – ASTM Type II

7.2.1.2 Methylene Chloride – pesticide quality or equivalent
TALS Reagent Name: EX_MECL2_00001
Storage: Flammables Cabinet

7.2.1.3 Acetone – pesticide quality or equivalent

TALS Reagent Name: EX_Acetone_00001
Storage: Flammables Cabinet

7.2.1.4 Hexane – residue grade or better
TALS Reagent Name: EX_Hexane_00001
Storage: Flammables Cabinet

7.2.1.5 Sodium Hydroxide (NaOH) Solution (10N) – Fisher Part Number FLSS255
TALS Reagent Name: EX-10NNaOH_00001
Storage: cool, dry cabinet or under a hood

7.2.1.6 Sulfuric Acid (H_2SO_4) Solution (1:1 w/v) – Fisher part Number LC25640
TALS Reagent Name: EX_10N_H2SO4_00001
Storage: cabinet, dry cabinet or under a hood

7.2.1.7 Sodium Sulfate – anhydrous, granular
TALS Reagent Name: EX_Na2SO4_00001
Storage: cool, dry place

7.2.1.8 Methanol - pesticide quality or equivalent
TALS Reagent Name: EX_Methanol_00001
Storage: Flammable Cabinet
Expiration: manufacturer's expiration date or 2 years from the date opened, whichever is sooner

7.2.2 Prepared Reagents

7.2.2.1 Sodium Sulfate, Baked – anhydrous, granular, purified by heating for approximately 3 hours at approximately 500°C n a shallow tray. Store in glass containers.
TALS Reagent Name: EX_Baked NaSO4_00001
Storage: cool, dry place

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*. Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

All standards must be stored in accordance with the manufacturer's recommended temperatures (typically room temperature) until opened. Once opened, all purchased standards, and all prepared standards are stored in the refrigerator at 0-6°C, unless otherwise noted below.

Unless otherwise noted, all purchased standards have an expiration date of 6 months from date of opening, and all prepared standard have an expiration date of 3 months from date of preparation.

Note: This standards list is comprised of the routine standards used by the laboratory. Information on project-specific, non-routine standards is found in the Reagent Module in TALS.

7.3.1 Purchased Standards

7.3.1.1 Organochlorine Pesticide Surrogate Stock, 200ug/mL – Purchased from Restek (catalog # 32000)
TALS Reagent Name: SGPESTSURR_XXXXX

7.3.1.2 Pesticide A\B Mix, varied concentrations – Purchased from Restek (Custom# 32415.~~sec~~)
TALS Reagent Name: SG_ABICV_XXXXX

7.3.1.3 Aroclor 1016/1260 Spike Mix, 1000ug/mL – Purchased from Restek (catalog # 32039)
TALS Reagent Name: SG1660CAL_XXXXX

7.3.1.4 O-terphenyl Standard, 10000ug/mL – Purchased from NSI (catalog # C-1341H-TP)
TALS Reagent Name: SG_OTP_XXXXX

7.3.1.5 #2 Diesel Fuel, 100mg/mL – Purchased from Accustandard (catalog # FU-009-D-200X)
TALS Reagent Name: SGDROCAL_XXXXX

7.3.1.6 Decachlorophenyl 13C12. 40ug/mL. Purchased from Cambridge Isotopes (catalog # EC-1410-3)
TALS Reagent Name: DB(680)SURR_XXXXX

7.3.1.7 680 Nonachlorobiphenyl Solution, varied concentrations – Purchased from Ultra Scientific (catalog # RPC-081-S)
TALS Reagent Name: PCB RTmix_XXXXX

7.3.1.8 BNA Surrogate Standard 5000ug/mL. Purchased from Restek (catalog # 567685).
TALS Reagent Name: 8270_SURR_XXXXX

7.3.1.9 8270 surrogate standard 100ug/ml . Purchased from Restek (part # 570814)
TALS Reagent Name : 8270RTSSURR_XXXXXX

7.3.1.10 SM O-Terphenyl Standard, 2000ug/mL. Purchased from Restek (catalog # 31066)
TALS Reagent Name: O-Terp Std_XXXXX

7.3.1.11 BNA Surrogate, 100ug/mL. Purchased from Restek (catalog # 567685).
TALS Reagent Name: 8270RTSSurr _XXXXX

7.3.1.12 8270 List 1/Standard 1, varied concentrations - Purchased from Restek (catalog # 567672)
TALS Reagent Name: 8270 L1/S1_XXXXX

7.3.1.13 8270 List 1/ Standard 11, 2000ug/mL - Purchased from Restek (catalog # 569732)
TALS Reagent Name: 8270 L1/S11_XXXXX

7.3.1.14 8270 List 1/ Standard 9, 2000ug/mL - Purchased from Restek (catalog # 569730)
TALS Reagent Name: 8270 L1/S9_XXXXX

7.3.1.14 8270 List 1/ Standard 10, 2000ug/mL - Purchased from Restek (catalog # 569731)
TALS Reagent Name: 8270 L1/S10_XXXXX

7.3.1.15 680 Calibration Concentration Mix- varied concentrations. Purchased from Ultra (catalog

number CB681-MN)
TALS reagent name: 680conCal_XXXXX

7.3.1.16 Lindane 13C6 Surrogate- 100ug/ml. Purchased from Cambridge (catalog # CLM128)
TALS reagent name: Lindane-13C6_XXXXX

7.3.1.17 4'4-DDT 13C12 Surrogate- 100ug/ml. Purchased from Cambridge (catalog # SDFK-001).
TALS reagent name: 4,4'DDT13C12_XXXXXXX

7.3.2 Prepared Standards

7.3.2.1 Working Pesticide / PCB – prepared by adding 1.25mL Pesticide Surrogate Stock (200ug/mL) diluted to a final volume of 500mL in methanol.
TALS Reagent Name: PESTwkSURR_XXXXX

7.3.2.2 Working Pesticide Spike Mix – prepared by diluting 1.0mL of INTPESTABICV (from SG dept) to 100mL of methanol.
TALS Reagent Name: 608wkSPIKE_XXXXX

7.3.2.3 Working PCB Spike Mix – prepared by adding 1.0mL of Aroclor 1016/1260 Spike Mix diluted to a final volume of 100mL in methanol.
TALS Reagent Name: 1660wkSPIKE_XXXXX

7.3.2.4 Working DRO Surrogate Mix – prepared by adding 1.0mL of O-terphenyl standard (10000 ug/mL) diluted to a final volume of 500mL in acetone.
TALS Reagent Name: DROwkSURR_xxxxx

7.3.2.5 Working DRO Spike Mix – prepared by adding 1.0mL of #2 Diesel Fuel (100 mg/mL) diluted to a final volume of 100mL in acetone.
TALS Reagent Name: DIESELWK_xxxxx

7.3.2.6 Working 680 Surrogate Mix – prepared by adding 3.125mL of Decachlorobiphenyl 13C12 (40ug/ml), 1mL of Lindane 13C6, 1ml of 4,4'DDT and diluted to a final volume of 50mL in acetone.
TALS Reagent Name: 680wkSURR_xxxxx

7.3.2.7 Working 680 Spike Mix – prepared by adding 1.0mL of Concentration Calibration Standard Mix (varied concentrations) and 1.25mL of PCB RT Mix diluted to a final volume of 25mL in acetone.
TALS Reagent Name: 680wkSPIKE_xxxxx

7.3.2.8 Working LLBNA Surrogate Mix – prepared by adding 1mL 8270_SURR and 0.5mL of O-Terphenyl std in 500mL of acetone.
TALS Reagent Name: LLBNAwkSUR_xxxxx

7.3.2.10 Working EX 8270L1 Spike - prepared by adding 10mL 8270 L1/S1, and 5mL 8270 L1/S10 and 5ml L1/S11 in 100mL of methanol
TALS Reagent Name: EX8270SPKL1

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 1L (for high volume extractions) or 250mL (for LVI extractions) amber glass containers without preservative.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation and/or analysis.

Aqueous samples must be prepared within 7 days of collection and analyzed within 40 days of extraction.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

9.1.1 EPA 600-Series Methods

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period. The default QC items required for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) performed per 10 % of samples or 1 per batch – whichever is greater and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, a matrix spike, and a matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, a matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

For high volume samples, the routine container supplied for this method is a 1L container. 1L is required for extraction. Insufficient sample volume is defined as receiving less than a total of 3L. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 500mL.

For LVI, the routine container supplied for this method is a 250mL container. The entire contents of the container are required for extraction. Insufficient sample volume is defined as receiving less than a total of 3 containers for an individual sample unless a unique container for the MS/MSD is provided.

If there is insufficient sample to perform the MS/MSD, the LCS must be prepared in duplicate

(i.e., LCS/LCSD). An NCM must be initiated to denote this situation.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1.2 EPA 3520C Methods

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period. The default QC items required for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

If there is insufficient sample to perform the MS/MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated to denote this situation.

For high volume samples, the routine container supplied for this method is a 1L container. 1L is required for extraction. Insufficient sample volume is defined as receiving less than a total of 3L. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 500mL.

For LVI, the routine container supplied for this method is a 250mL container. The entire contents of the container are required for extraction. Insufficient volume is defined as receiving less than a total of 3 containers for an individual sample unless a unique container for the MS/MSD is provided.

Note: For EPA 8015C, insufficient sample volume defined as receiving less than a total of 3L. Due to the affinity of these compounds for the glass bottle, reduced sample volumes may not be used for this analysis; rather, the contents of the entire container must be extracted.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

Refer to the applicable analytical SOP (Section 1.0) for information on instrument QC.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation – Pre-Prep Steps

10.1.1 Remove the samples from the refrigerator and allow them to come to room temperature.

10.1.2 Scan the samples into the applicable TALS batch.

10.1.3 Preservation and Residual Chlorine Checks

Pour approximately 5mL of sample into a disposable medicine cup. Use this volume for both the pH and residual chlorine verifications.

10.1.3.1 pH Verification.

For each sample:
- Dip the pH paper into the cup containing sample.
- Record the reading as the Initial pH in the TALS batch.
- If the pH is outside the range of 5-9, initiate a Nonconformance Memo.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

10.1.3.2 Residual Chlorine Check

For each sample:
- Dip the residual chlorine strip into the cup containing sample.
- If the strip turns blue to deep purple, this indicates the presence of residual chlorine.
- Indicate in the TALS batch worksheet with Yes or No (i.e., Y or N) for the presence of residual chlorine.
- If residual chlorine is detected, initiate a Nonconformance Memo.

Note: To avoid cross-contamination, use a separate medicine cup and residual chlorine check strip per sample. Do not dip the test strip into the sample container.

10.1.4 Determination of Initial Volume

The sample volume is determined gravimetrically using a top-loading balance.

Weigh each filled sample container and record this weight in the preparation batch in TALS as the gross weight in grams. The empty container is then re-weighed and weight recorded in grams as the tare weight in the preparation batch in TALS. These weights are then used to determine the initial volume in milliliters.

Note: The density is assumed to be 1g/mL.

Note: If samples are received in duplicate, use the sample bottle that contains the most volume. Extracting less than the routine volume of sample as described in Section 9 will result in elevated reporting limits. Selecting the sample bottle with the

most volume will ensure the lowest possible RL is obtained.

Note: If a reduced volume of sample must be extracted in order to provide sufficient sample volume to meet state or other program QC requirements, the sample is thoroughly mixed and an aliquot is measured into a Class A volumetric container to determine the weight and then poured into the extractor body.

10.1.5 Surrogate Addition

Add surrogate spiking solution directly to each sample in the sample container, prior to pouring the sample into CLLE apparatus and prior to pH adjustment. Be sure to use the correct spiking solutions for the analytical procedure of concern. The addition of the surrogates ***must*** be witnessed by another analyst to ensure that the proper spiking solution and volume is added to each sample.

Note: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of surrogate spiking solution must be reduced proportionately.

10.2 QC Sample Preparation

10.2.1 Method Blank – Add appropriate amount of reagent water and surrogate to the extraction vessel. Prepare as a sample following the steps outlined in Section 10.3 or Section 10.4.

10.2.2 Laboratory Control Sample – Add appropriate amount of reagent water, surrogates, and spiking solutions to the extraction vessel. Be sure to use the correct surrogates, spiking solutions, and volumes for the analytical procedure of concern. The addition of the spike solutions ***must*** be witnessed by another analyst to ensure that the proper spiking solutions and volume is added to each sample. Prepare as a sample following the steps outlined in Section 10.3 or Section 10.4.

10.2.3 Matrix Spike(s) – Spike the sample container with the appropriate volume of spiking solution. Be sure to use the correct solutions for the analysis of concern. The addition of the matrix spike mixes ***must*** be witnessed by another analyst to ensure that the proper volume of the spiking solution is added to each sample. Prepare as a sample following the steps outlined in Section 10.3 or Section 10.4.

10.3 CLLE Extraction Procedures

All glassware preparation steps must be performed under or near a fume hood to minimize the evaporation of methylene chloride into the lab.

10.3.1 In a fume hood, add approximately 70mL of methylene chloride to the receiving flask, and add a few boiling stones to the flask. Plug the flask with a Teflon plug until time to attach to an extractor body.

10.3.2 Set the extractor body on the holder in the fume hood. Add methylene chloride (50-100mL) to the extractor body in order to fill the drain tube and ensure that a continuous flow of solvent from the extractor body to the receiving flask is maintained.

Note: Inspect the solvent layer when the sample is added. Large quantities of

sediment may plug the tube such that extraction will not occur.

10.3.3 As soon as possible after the methylene chloride is added to the extractor body, gently pour the entire sample into the extractor body, trying not to let the sample leak into the sidearm. If the sample leaks into the sidearm, the sidearm can be drained back into the sample container and the sample can then be poured back into the extractor body.

The extractor body will be approximately one-half to two-thirds full, and the methylene chloride in the solvent return should be about 3/4 of the way to the receiving flask.

Note: If a sub-sample must be extracted (e.g., half volume is used to perform MS/MSD) measure the sample volume in a graduated cylinder prior to pouring into the extractor body.

10.3.4 Add 50mL of methylene chloride to the sample container (or the volumetric container if a reduced initial volume is used). Swirl the solvent around the inside of the container to thoroughly rinse, and add this rinse to the extractor body.

Adjust the pH of the samples to the range specified in Attachment 5. The pH is adjusted by adding small aliquots of 1:1 sulfuric acid to the liquid body after samples are poured up. Stir the sample gently with a Pasteur pipette/stir rod. To ensure cross-contamination does not occur, use a new pipette/stir rod for each sample. Check the pH after each acid/base addition with pH paper to ensure the sample is in the proper pH range. Record this pH as 1st pH in the TALS worksheet.

Note the following critical step:
Use narrow range pH paper to confirm the pH of samples for EPA 8270D or EPA 625 after adjustment.

Note: Sulfuric acid will cause the pH of the sample to decrease. Sodium hydroxide will cause the pH of the sample to increase.

10.3.5 Slowly add reagent water to the extractor body until the level of the liquid causes the methylene chloride to just flow over to the receiving flask.

Securely attach the prepared receiving flask to the extractor body, and secure the heating mantle around the flask.

10.3.6 Place the condenser securely on the extractor body. Make sure that water is flowing through the condenser and that the extractor body is positioned sitting up straight and not tilted back.

Note: If the extractor body is tilted, it will not allow sufficient cycling.

Turn the heating mantle on. Observe the extraction for the first hour or so to ensure that the solvent is being boiled, condensed, and returned to the receiving flask. Extract the samples for the time listed in Attachment 5. The batch start time and stop time must be recorded in the AD batch in TALS. Start time is recorded as the time the heating mantles are connected. The stop time is recorded as the time the heating mantles are disconnected. If a single pH extraction is performed, skip to Section 10.3.9. If a dual pH extraction must be performed, continue to Section 10.3.7.

Note: Dual pH extraction is only required for EPA 625 and EPA 8270D

Visually inspect the samples. Determine if the samples have multiple layers such as sediment or an oil layer. Consult with the Department Supervisor or Technical Manager if the sample matrix is unusual or difficult to categorize.

For EPA 625 and EPA 8270D Only:
If a dual pH extraction is required, turn the heating mantle "off" to stop the extraction at the appropriate time, and allow the extraction vessel to cool (at least 30 minutes). Remove the receiving flask from the extractor body and cover with a Teflon stopper. Store extract sealed in the liquid room. Place a new receiving flask on the extractor body.

10.3.7 Adjust the pH of the sample (as indicated in Attachment 5) by adding small aliquots 10N sodium hydroxide. Stir the sample with a disposable Pasteur pipette, and check the pH after each addition in accordance with Section 8.1.1.1. Do not dip the pH paper into the sample. Use a pipette to remove a small aliquot of the sample and touch the liquid to the pH paper. Record this pH as 2nd pH in the TALS worksheet.

Note the following critical step:
Use narrow range pH paper to confirm the pH of samples for EPA 8270D or EPA 625 after adjustment.

Note: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

10.3.8 Turn the heating mantle on and extract the sample at the second pH for the time listed in Attachment 5. Record start time for the 2nd extraction in the Batch Information screen of the AD batch.

10.3.9 After the samples have extracted for the required time, turn the heating mantle "off", and allow the extraction vessel to cool to room temperature. Record this as the stop time for the 2nd extraction in the Batch Information screen of the AD batch. Note: It is important to allow the receiving flask to cool before it is removed from the extractor.

10.3.10 Keep the extract covered with aluminum foil or Teflon plug until the concentration step, if it is to be performed the same day. When extracts must be stored on the counter or on carts, they must be shielded from light with a black-out blanket.

10.3.11 Rinse the ground-glass joint of the condenser with acetone. Wrap in aluminum foil if the condenser will immediately be re-used for a subsequent batch. Place the condenser in the rack. Collect the acetone rinsate in a separate container for disposal.

10.3.12 After the receiving flask has been removed from the extractor body, the methylene chloride remaining in the extractor body must be properly disposed of in the chlorinated solvent waste container. The extractor must be handled with minimum agitation to minimize the amount of solvent allowed to evaporate into the laboratory. Place the receiving flask side-arm over the chlorinated waste container. Add tap water to the extractor body to displace the methylene chloride into the waste container.

10.3.13 The liquid remaining in the extractor body must be poured carefully down the sink and flushed continuously with water.

10.3.14 Clean the glassware in accordance with the procedures in Attachment 6.

10.4 Zymark Concentration Procedures

10.4.1 Choose an appropriate size Zymark tube to accommodate the required final volume. Pre-rinse the Zymark tube with methylene chloride and allow to dry.

10.4.2 Turn on TurboVap evaporation unit. Set the water bath temperature to 50°C. Set the control to "auto" and "pressure". Set the gas pressure to zero initially for each cell in the TurboVap unit.

10.4.3 Pre-rinse each concentration cap on the TurboVap evaporation unit with methylene chloride.

10.4.4 Working under a hood, pour the extract into a Zymark concentration tube, and transfer the label from the round bottom flask to the Zymark tube. If samples require EPA 625 or EPA 8270D dual pH extraction, the contents of the two round bottom flasks are transferred to the same Zymark tube.

Rinse the round bottom receiving flask with approximately 5mL of solvent and transfer this rinsate to the Zymark tube. Place the tube into a cell of the evaporation unit.

Repeat for all samples in the analytical batch. The remaining extract must be tightly covered with a piece of aluminum foil to minimize evaporation of the solvent.

Set tubes into the Zymark unit. Cover each tube with the corresponding cap.

NOTE: Make sure the position used in the unit has not been taken out of service. Positions which do not pass the weekly check will be marked with an "X".

10.4.5 Carefully close the cover of the Zymark TurboVap unit completely. Make sure that each tube is seated properly and that the individual covers are positioned directly over each tube.

Note: When the elongated tubes are used, care must be taken to position the tubes to avoid breaking the tube covers and to completely close the instrument cover.

10.4.6 Concentrate the extracts at a constant temperature of 50°C. Start the concentration with a pressure of 10psi. Once the extract volume reaches approximately 70mL, turn the pressure up to 15psi.

10.4.7 Concentrate the extract until the cell alarm sounds. This indicates that the extract volume is near or below 1mL.

Note: Due to loss of target analytes, it is not advised to concentrate extracts to less than 1ml for methods 8270 or 625.

10.4.8 For EPA 608, EPA 8081B, EPA 8081A, and EPA 680, a solvent exchange of hexane is required. Add 10mL of hexane to the Zymark concentration tube and concentrate to 1ml..

10.4.9 Remove Zymark concentration tubes from evaporation unit and transfer to storage vials as outlined in Section 10.5.

10.5 Final Volume Determination and Extract Transfer Procedures

Extracts are quantitatively transferred from the concentration tube to the storage and/or autosampler vial as follows:

1) For methods that require a final volume of 1.0mL, the autosampler vial is used to determine the final extract volume. The volume markings on these vials must be verified per lot.

   Concentrate the extract to approximately 1mL. Using a transfer pipette, transfer all of the volume in the zymark tube to an extract vial. Rinse the tube with the appropriate solvent to rinse off any extract that has deposited/collected on the walls of the tip of the tube. Use this rinsate to establish the 1.0mL final volume using the markings on the autosampler vial.

   Note: If the extract cannot be evaporated to 1mL due to the matrix of the sample, do not force the extract to concentrate. Adjust to an appropriate final volume (e.g., 2mL or 5mL) using the process outlined below.

2) For methods that require a final volume greater than 1mL the disposable pipette or class A volumetric container is used to determine the final extract volume. The volume markings on these pipettes must be must be verified per lot.

   Concentrate the extract to approximately 1mL. Using a 5mL pipette, withdraw the extract from the tube while simultaneously and slowly adding a small amount solvent to rinse the tube and to achieve a final volume of 5mL, as measured in the pipette. Transfer to the appropriate vial for storage. Use additional solvent and a calibrated pipette to achieve the appropriate final volume as needed.

   Transfer to the appropriate vial for storage.

Mark the volume of the extract on the side of the storage container/vial to allow the analyst to judge whether the sample extract has evaporated during storage and handling. Store concentrated extracts at 0-6°C until time of analysis.

10.6 Sample Drying Procedures – EPA 8270D and EPA 625

Extracts for EPA 8270D and EPA 625 should have a neutral pH. An acidic pH indicates the presence of water in the extract, and, as such, the sample will need to be dried as outlined below.

Note: This process should occur after extract concentration and prior to final volume determination and extract transfer to the autosampler/storage vial.

Using a transfer pipette, test the pH of the extract in the concentration tube. Transfer one drop of extract onto pH paper (range 1-6). A pH within a range of 5-6 is neutral and requires

no further drying steps. A pH <5 indicates the extract is acidic and requires additional drying steps to remove the water from the extract.

1. Add approximately 5mL of methylene chloride to the extract in the concentration tube.
2. Add approximately 5mL of DI water to the tube.
3. Swirl and allow the methylene chloride and water layers to separate.
4. Prepare a drying column by placing a small piece of glass wool into a transfer pipette. Pack the transfer pipette with dried sodium sulfate until approximately ¾ full.
5. Pass the methylene chloride layer (i.e., the bottom layer) through the drying column in small aliquots. Any residual water in the extract will be absorbed by the sodium sulfate. Collect the extract in a new concentration tube. Add a small amount of methylene chloride to elute the sample through the sodium sulfate.
6. Proceed to the extract concentration steps as outlined in Section 10.6.
7. Prior to final volume determination, recheck the pH as outlined above. A pH within a range of 5-6 is neutral and requires no further drying steps. A pH <5 indicates the extract is still acidic and requires further drying steps to remove the water from the extract. Repeat steps 1-6, as needed.

10.7 Analysis

Refer to the applicable analytical SOP (Section 1.0) for information on sample analysis.

11.0 Calculations / Data Reduction

11.1 Data Reduction

11.1.1 Data reduction and review tasks include the following items:

- Employ spike witness procedures to ensure proper spiking solutions and volume are used.
- Ensure all TALS batch Data Type fields are completed so that proper traceability is maintained.
- Check to ensure that each sample extract is properly identified and that the extracts are transferred to the analytical groups with proper documentation.
- Mark the final volume of the extract on the outside of the storage container as a check on extract evaporation.
- QC items must be treated in the same manner as samples.
- Ensure samples are evaporated at the appropriate rate. Unacceptable surrogate or spike recoveries may be attributed to evaporating the samples too quickly or to improper solvent exchange.
- Document any unusual circumstances and procedural violations using the LIM Nonconformance Module. This can include: samples problematic matrices such as color, odor, or emulsions; initial or final amount changes; deviations to the SOP, etc.

Batch data must be reviewed and evaluated in accordance with SOP SA-QA-002: Data Generation and Review.

Additional details on data reduction procedures are given in the associated analytical SOPs.

11.1.2 Initiate an NCM, as follows, if the following conditions are noted:

- Samples have color.
  The sample(s) was noted to be the following color: <comma&Merge>. Historically, samples with any color have not recovered within the established quality control limits.

- Sample causes emulsions to form.
  The following sample(s) was noted to have formed an emulsion: <comma&Merge>. Historically, samples with emulsions have not recovered within the established quality control limits.

- Sample appearance/physical properties are unusual.
  The following sample(s) was noted to have a(n) cloudy/opaque/viscous appearance: <comma&Merge>. Historically, samples with this condition have not recovered within the established quality control limits.

  The following sample(s) was noted to have low levels of solids present in the liquid body: <comma&Merge>. Historically, samples with solids have not recovered within the established quality control limits.

  The following sample(s) was noted to have high levels of solids present in the liquid body which required the addition of glass wool to the liquid body: <comma&Merge>. Historically, samples with solids have not recovered within the established quality control limits.

- Additional acid or base was required to adjust the pH.
  The following sample(s) required the addition of more than 2mL of acid to pH the sample to less than 2: <comma&Merge>. Historically, samples requiring additional acid have not recovered within the established quality control limits.

  The following sample(s) required the addition of more than 2mL of base to pH the sample to greater than 11: <comma&Merge>. Historically, samples requiring additional base have not recovered within the established quality control limits.

- Residual chlorine was present.
  The presence of residual chlorine was noted in the following sample: <comma&Merge>. Historically, samples with residual chlorine have not recovered within the established quality control limits.

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.2 Calculations

Details on sample calculations are given in the associated analytical SOPs listed in Section 1.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

12.4.1 EPA 600-Series Methods

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

12.4.2 EPA 3520C and EPA 3520C_LVI

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Methylene chloride extracts – Dispose according to characterization on sample disposal sheets. If non-hazardous, transfer extract to chlorinated waste container. If hazardous, transfer to hazardous waste department for storage.

- Methylene chloride waste – Transfer to chlorinated waste container.

- Hexane extracts – If non-hazardous, transfer to flammable waste containers and dispose of as flammable waste. If hazardous, transfer to the waste disposal department for disposal as hazardous waste.

- Flammable waste (hexane or acetone from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.

- Aqueous acidic waste from samples – Collect into the disposal area and neutralize before release to the sewer system.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Test Methods for Evaluating Solid Waste*, Third Edition (Updates III and IV), SW-846; EPA Office of Solid Waste and Emergency Response: Washington, DC.
    - Chapter 4: *Organic Analytes*; Revision 3, December 1996
    - Chapter 4: *Organic Analytes*; Revision 4, February 2007
    - EPA 3500C: *Organic Extraction and Sample Preparation*; Revision 3, February

2007
- EPA 3520C: *Continuous Liquid-Liquid Extraction*; Revision 3, December 1996
- EPA 3600C: *Cleanup*; Revision 3, December 1996
- EPA 3660B: *Sulfur Cleanup*; Revision 2, December 1996
- EPA 3665A: *Sulfuric Acid/Permanganate Cleanup*; Revision 1, December 1996

- Code of Federal Regulations, Title 40 (Protection of Environment), Chapter 1 (Environmental Protection Agency), Subchapter D (Water Programs), Part 136 (Guidelines Establishing Test Procedures for the Analysis of Pollutants), Appendix A (Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater)
  - EPA 608: *Organochlorine Pesticides and PCBs*
  - EPA 625: *Base/Neutrals and Acids*

16.0 Method Modifications and Clarifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices.

16.1.1 Collection and Handling Procedures

Once the TCLP/SPLP extraction procedure has been performed, the TCLP/SPLP leachate must be transferred to a 1L amber bottle. The TCLP/SPLP leachate must be preserved in accordance with the associated method and stored at 4°C (less than 6°C abut not frozen) until the time of extraction. The leachate must be extracted within 7 days of completion of the TCLP/SPLP leaching procedure.

Note: The sample leachate chosen as the MS/MSD must be spiked prior to adjusting the pH.

16.1.2 Preparation and Analytical Procedures

TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in this SOP. Refer to Attachment 5 for the extraction conditions. The following volumes are utilized for TCLP samples for SVOCs:

TCLP PARAMETER	Volume of Sample Extracted (mL)	Final Volume (mL)
BNA by GC/MS (EPA 8270)	200	1.0
Pesticides by GC (EPA 8081B)	20	10

TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in the associated analytical SOPs (Section 1.0).

16.2 Other Considerations

16.2.1 The analyte lists in Section 1.1 of EPA 608, and in Tables 1, 2, and 3 of EPA 625, have been expanded to include all analytes currently performed by the laboratory.

16.2.2 The volume of solvent added to the receiving flask for CLLE has been modified from the guidance in EPA 3520C and EPA 625. EPA 3520C specifies 300-500mL of methylene chloride to be added to the receiving flask, and EPA 625 specifies that 200-500mL be added to the flask. The laboratory adds 70-100mL to the receiving flask. This volume provides sufficient solvent volume to efficiently extract the sample since the CLLE is a closed system, minimizes solvent exposure to the analyst, and allows the extract to be concentrated faster. Laboratory control standard (LCS) and performance testing (PT) data using the reduced solvent volumes meet or exceed the minimum accuracy and precision criteria specified in the reference method. The Methods Update Rule specifically allows this type of method modification to be made.

16.2.3 A specific temperature set-point for the K-D water bath has not been established at this time, but rather a temperature range of 60-90°C is used. There should be little impact on the analyte recoveries provided the temperature is hot enough the boil the solvent.

16.2.4 The laboratory's default required batch QC items differ from those outlined in the reference methods. For example, there is no method-defined batch precision requirement listed in EPA Method 608; however, the EPA does require precision for all samples analyzed under the Clean Water Act. In order to satisfy this and other client-specific and/or regulatory program requirements and expectations, matrix spike duplicates and laboratory control sample duplicates have been incorporated.

16.2.5 Dependent on capacity, and with the approval of Technical Management, the laboratory may employ the option to reduce CLLE times from those specified in Attachment 5. The batch QC and an additional RLV performed within the batch will be used to verify sensitivity and recovery. An NCM must be initiated to denote this situation.

16.2.6 Chapter 4 of SW-846 specifies to send sample collection bottles containing dechlorination agent for those samples from a chlorinated water source. Since the laboratory often is not made aware of the source of the water, dechlorination agent is not routinely provided. Samples are checked for residual chlorine prior to extraction, and an NCM must be initiated for any samples that test positive.

16.2.7 Unless requested by the client, the laboratory does not perform MDLs, DOCs, and LCSs for the non-routine and site-specific compounds.

16.2.8 Method 608 and 680 only give instructions for prep by separatory funnel (3510C). The laboratory utilized method 3520C for prep of samples for analysis by method 608 and/or 680. All validation has been performed by extraction by method 3520C.

16.2.9 EPA Method 3520C requires drying of the extract utilizing sodium sulfate prior to concentration. In the extraction process, the solvent fraction contained in the round bottom is the extract, and this volume does not make direct contact with the water fraction during the CLLE extraction process. As a result, the drying of the solvent as mentioned in the reference method is not necessary nor utilized as part of the routine procedure. If water is visible in the flask after extraction, the volume will be dried utilizing sodium sulfate.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Extraction Conditions – CLLE
Attachment 6: Glassware Cleaning Procedures
Attachment 7: Sulfur (Copper) Cleanup
Attachment 8: Sulfuric Acid Cleanup
Attachment 9: Zymark Sensor Diagnostic Test and Maintenance Log
Attachment 10: Zymark-specific Maintenance Instructions
Attachment 11: Method Prep Work Instruction
Attachment 12: Sample Spiking Volumes

Attachment 1:
SOP Summary

Sample Preparation Summary

Continuous Liquid-Liquid Extraction Procedure
In continuous liquid-liquid extraction a known volume of sample is adjusted to a specific pH, if required by the analytical method, transferred to a continuous liquid-liquid extractor, and extracted using the solvent and conditions specified in Attachment 5. The extract is concentrated to an appropriate final volume using either the Zymark TurboVap concentration procedure.

Zymark Concentration Procedure
After the extraction procedure is completed, the solvent is transferred to a glass Zymark concentration tube. The tube is placed in the Zymark concentration device, which has been heated to a specified temperature. A stream of nitrogen is directed into the tube to evaporate the solvent and to concentrate the target compounds. When the volume of solvent reaches the specified volume, normally 1mL, the nitrogen is automatically stopped. An alarm sounds to alert the analyst, and the extract is removed from the device and transferred to a storage vial or container.

If a solvent exchange is required, the exchange solvent is added to the tube, the solvent is evaporated to the specified final volume, and the extract is transferred to a storage vial or container. The concentrated extracts are stored at 4°C until the time of analysis.

Sample Analysis Summary
Analyze samples in accordance with the following SOPs:

Fraction	Analytical Method	SOP #
Organochlorine Pesticides & PCBs	EPA 608 EPA 8081B EPA 8082A	SOP SA-SG-045
Diesel Range Organics Oil Range Organics (DRO and ORO)	EPA 8015C	SOP SA-SG-070
Polychlorinated Biphenyls	EPA 680	SOP SA-SM-007
Base Neutrals / Acids & PAHs	EPA 625 EPA 8270D EPA 8270D_LL	SOP SA-SM-033

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time [1]
Water (CLLE)	1L amber glass	1L	500mL	None	0-6°C[2]	None	7 days
Water (CLLE LVI) *Pesticides and PCBs only	250mL amber glass	250mL	250mL	None	0-6°C[2]	None	7 days

[1] Time from collection to initiation of extraction.
[2] Samples must be maintained at 0-6 °C, with no frozen samples.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Up to 20 field samples prepared together within a 24-hour time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample (LCS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample is provided for the MS/MSD	Refer to analytical SOP	Refer to analytical SOP
Matrix Spike (MS)	EPA 600-Series: One per 10% of samples EPA 3520C, 3520C_LVI: One per batch	Refer to analytical SOP	Refer to analytical SOP
Matrix Spike Duplicate (MSD)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per method/analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 (Note: Unsupervised work must not begin until successful IDOC has been obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per method/analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

Zymark Maintenance
It is recommended to change the water in the water bath weekly. Add 1-2 drops of Clear Bath to prevent bacteria and algae growth. Methylene chloride that dissolves in the water bath will damage the sensors.

The Zymark sensor diagnostic test must be performed weekly. If the sensors do not meet criteria the sensor may need replacing. Refer to the manufacturer's manual for replacement procedures if necessary.

The thermometer for the Zymark must be calibrated in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*.

Troubleshooting
Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan
An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5:
Extraction Conditions - CLLE

SEMIVOLATILE GC EXTRACTIONS					
Methods	**Extraction Conditions**	**Extraction Time (Hours)**	**Extraction Solvent**	**Final Solvent**	**Final Volume (mL)**
EPA 608 EPA 8081B EPA 8082A	Single pH at neutral pH (5-9)	18-24	$MeCl_2$	Hexane	10
EPA 608_LVI EPA 8081B_LVI EPA 8082A_LVI	Single pH at neutral pH (5-9)	18-24	$MeCl_2$	Hexane	2.5
EPA 8015C	Single pH <2	18-24	$MeCl_2$	$MeCl_2$	1.0

SEMIVOLATILE GC/MS EXTRACTIONS					
Methods	**Extraction Conditions**	**Extraction Time (Hours)**	**Extraction Solvent**	**Final Solvent**	**Final Volume (mL)**
EPA 625	Dual pH: pH<2 followed by pH>11	24 / 24	$MeCl_2$	$MeCl_2$	1.0
EPA 8270D (TCL) EPA 8270D_LL (TCL)	Single pH <2	18-24	$MeCl_2$	$MeCl_2$	1.0
EPA 8270D (AP9) EPA 8270D_LL (AP9) EPA 8270D TCLP	Dual pH: pH<2 followed by pH>11	18-24 / 18-24	$MeCl_2$	$MeCl_2$	1.0
EPA 680	Single pH at neutral pH (5-9)	18-24	$MeCl_2$	Hexane	1.0

Attachment 6:
Glassware Cleaning Procedures

EX GLASSWARE CLEANING PROCEDURES #1
(SEP FUNNEL & MISC GLASSWARE)

(Includes glassware used for O&G, MBAS, TCLP, funnels, beakers, etc.)

PPE: **Lab coat**
Eye protection
Cut resistant gloves

Note: It is important to segregate heavily contaminated glassware from use until verified clean by the analysis of a method blank. Discard if the condition of the glassware cannot be verified or if the glassware is obviously not salvageable.

1. Rinse all glassware thoroughly with water and discard down the sink drain.

3. Fill dishpan with hot water and add about 60mL FL-70 detergent.

4. Brush vigorously until clean.

5. Rinse three times with tap water.

6. Rinse with sulfuric acid. (OPTIONAL)

   Note: All separatory funnel stopcocks must be removed prior to washing. Stopcocks will not be replaced until separatory funnels have been rinsed with sulfuric acid and rinsed with running water.

   After rinsing with sulfuric acid, rinse glassware thoroughly with tap water. All traces of the acid must be removed from the glassware.

7. Rinse with acetone and place on covered counter or rack to dry.

   Collect acetone rinses in the satellite waste container designated for flammable waste.

EX GLASSWARE CLEANING PROCEDURES #2
(CLLE BODIES)

PPE: **Lab coat**
Eye protection
Cut resistant gloves

This procedure assumes that the round-bottom flasks containing the extracts have been removed and that the CLLE bodies are at room temperature.

1. Remove the condenser and place on the rack. Be careful not to tip the CLLE body forward and cause methylene chloride to leak on to the table top. Gently remove the CCLE body from the rack and carry the CLLE bodies one at a time to the hood.

2. Empty the methylene chloride layer into the satellite waste container designated for chlorinated waste.

3. Turn on the water and pour the water layer from the CLLE into the sink. Rinse the CLLE body with water and discard down the sink. Repeatedly rinse the CLLE body with water until all of the solids are rinsed down the sink.

   Note: Be careful not to pour methylene chloride down the sink-it will dissolve the drain pipes.

4. Fill one side of the sink with hot water and add 800mL of FL-70 detergent.

5. Note the condition of the CLLE body. If heavily contaminated, do not place into the soak water. Keep this glassware separate and contact the Supervisor to determine the best course of action to clean the glassware.

   It is important to segregate heavily contaminated glassware from use until verified clean by the analysis of a method blank. Discard glassware if the condition of the glassware cannot be verified or if the glassware is obviously not salvageable.

6. Allow CLLE bodies that have been excessively soiled to soak for 10-15 minutes.

7. Scrub the inside of the CLLE bodies with a soft brush.

8. Rinse a minimum of three times with hot tap water. It is important to remove all of the soap film at this point.

9. Rinse the entire CLLE body with acetone, paying particular attention to the smaller glass tubing. It is important to remove as much water as possible from the CLLE body at this point. Collect the acetone in a satellite container.

10. Place the CLLE body on the rack inverted to help drain the remaining water and acetone and to dry the CLLE body.

EX GLASSWARE CLEANING PROCEDURES #3
(ZYMARK TUBES & CLLE ROUNDBOTTOM FLASKS)

PPE: **Lab coat**
Eye protection
Cut resistant gloves

1. Note the condition of the Zymark tube or receiving flask. If heavily contaminated, do not place into the sink. Keep this glassware separate and contact the Supervisor to determine the best course of action to clean the glassware.

   Note: It is important to segregate heavily contaminated glassware from use until verified clean by the analysis of a method blank. Discard if the condition of the glassware cannot be verified or if the glassware is obviously not salvageable.

2. Rinse each tube or flask thoroughly with water and discard down the sink drain.

3. Fill one side of sink with hot water and add about 60mL of FL-70 detergent.

4. For Zymark tubes, use a small brush to clean the tip of the tube and a larger brush to clean the walls of the tube.

   For receiving flasks, use a brush that will allow you to scrub the inside walls of the flask.

5. Rinse each tube and flask thoroughly a minimum of three times with hot tap water until no traces of soap are present in the tube. It is important to remove all traces of soap at this point.

6. Rinse each tube and flask thoroughly with acetone and place on covered counter or rack to dry.

   Collect acetone rinses in the satellite waste container designated for flammable waste.

Attachment 7:
Sulfur (Copper) Cleanup

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Work Instruction
FEX096: 05.20.11:1
Page 1 of 2

Sulfur Cleanup Procedures

Method: 3660B

Summary of Procedure
This procedure is based on EPA Method 3660B and is used in conjunction with the following SOPs:

Preparation Method	SOP #
EPA 3510C / EPA 3520C	SA-EX-030
EPA 3546 / EPA 3550B / EPA 3550C	SA-EX-040
Analysis Method	**SOP #**
EPA 508	SA-SG-046
EPA 608 / EPA 8081A / EPA 8081B / EPA 8082 / EPA 8082A	SA-SG-045
EPA 8276	SA-SM-034

The sulfur cleanup uses copper granules to eliminate elemental sulfur from PCB or pesticide extracts. Copper is added to the extract, and the vial is shaken. If sulfur is present, a black precipitate (copper sulfide) will form. The extract is treated with copper until no further precipitate is formed.

Note: All samples to be analyzed for EPA 8276 must undergo sulfur clean-up.

The method blank and LCS must be subjected to the same cleanup steps as the samples.

Perform all cleanup steps under a fume hood or in a well-ventilated area.

Reagents
Copper granules – the surface of the copper should be "shiny".

Cleanup Instructions
1. Ensure the sample extract has been exchanged into the applicable final solvent (e.g., MTBE for EPA 508, Hexane for EPA 608/8081B/8082A) prior to performing the sulfur cleanup procedure.

2. Add approximately 0.1g of "shiny" copper to the vial, and vortex for approximately two minutes.

   Note: If the extract is for EPA 614 or EPA 8141B in addition to one of the analytical methods listed above, transfer an aliquot of the extract to another vial for the copper cleanup.

3. If sulfur is present, a black precipitate will form. Allow the extract to sit for 2-3 minutes for any additional precipitate to form and settle out.

   If the precipitate does not settle out, additional copper treatments and/or filtration may be required. Contact the Technical Manager for instructions on how to proceed.

4. **The sample is now ready for analysis as outlined in Section 10 of the associated analytical SOP.**

Attachment 8: Sulfuric Acid Cleanup

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Work Instruction
FEX097:05.20.11:1
Page 1 of 2

Sulfuric Acid Cleanup Procedures

Method: 3665A

Summary of Procedure
This procedure is based on EPA Method 3665A and is used in conjunction with the following SOPs:

Preparation Method	SOP #
EPA 3510C / EPA 3520C	SA-EX-030
EPA 3546 / EPA 3550B / EPA 3550C	SA-EX-040
EPA 3580A	SA-EX-042
Analysis Method	**SOP #**
EPA 680	SA-SM-007
EPA 608 (PCBs only) / EPA 8082 / EPA 8082A	SA-SG-045
EPA 8276	SA-SM-034

The acid cleanup procedure is used for PCBs and Toxaphene Congeners only. Sulfuric acid is added to the extract, and the vial is shaken. The layers are allowed to separate, and the sample layer is removed. Large organic-soluble compounds that are present in the sample will extract into the acid and are discarded.

Note: This procedure will destroy pesticide compounds. Sulfuric acid cleanups must only be used for PCB samples and cannot be used for samples requesting pesticides by EPA 8081B, EPA 8141B, or EPA 608.

Note: All samples to be analyzed for EPA 8276 must undergo sulfuric acid clean-up.

The method blank and LCS must be subjected to the same cleanup steps.

Perform all cleanup steps under a fume hood or in a well-ventilated area.

Reagents
Sulfuric acid (H_2SO_4) – reagent grade, concentrated

Cleanup Instructions
1. Ensure the sample extract has been exchanged into hexane prior to performing the sulfuric acid cleanup.
2. Transfer an aliquot of the extract to a vial for the cleanup. The recommended aliquot volume is 5.0mL.
3. Add approximately 2mL of concentrated sulfuric acid and cap the vial. Mark the vial to denote the total volume on the vial. Also mark the vial to denote the separation of the acid (bottom) layer and the extract (top) layer.
4. Ensure the vial cap is secure, and gently shake the vial. Open the vial, and allow any pressure that has built up to dissipate. Repeat these steps until no pressure is noted when the cap is opened, and then shake the vial for one additional minute. A vortex mixer can also be used.

5. Allow the extract (top) layer and the acid (bottom) layer to separate. This separation may take a few minutes or several hours depending on the nature of the sample extract. Use the marks on the side of the vial to judge if the volume of extract (top) layer is the same as when it was originally added to the vial.

6. Remove the extract (top) layer from the vial using a disposable Pasteur pipette, and transfer the extract to a clean vial. If greater than 80% of the extract is recovered, no additional preparation is necessary.

7. If an emulsion has formed (i.e., there are bubbles or a cloudy area in between the top and bottom layers), add sodium sulfate crystals to the vial and gently stir the top layer with a glass rod. The sodium sulfate should help to break the emulsion so the top layer can be adequately recovered.

8. If the extract has color, perform an additional cleanup by adding 5mL more of concentrated acid and repeating Steps 3-6.

   Note: Use good judgment when determining how many acid cleanups to use. If it takes more than three cleanups to clean the extract, it is recommended to start over with a smaller aliquot of sample or to dilute the extract before proceeding with additional cleanups. A diluted extract can be concentrated back to the equivalent volume after the cleanup steps. Contact the Technical Manager for assistance with this task.

9. The sample is now ready for analysis as outlined in Section 10 of the associated analytical SOP.

Attachment 9: Zymark Sensor Diagnostic Test and Maintenance Log

ZYMARK MAINTENANCE LOG

Turbo Vap #: _____ Date:_____ Initials:_____
Thermometer #:_____

Sensor Diagnostic Test

SENSOR #	1			2			3			4			5			6		
		P	F		P	F		P	F		P	F		P	F		P	F
INITIAL VALUE																		
FINAL VALUE																		
% VALUE																		
OUT OF SERVICE																		
Turbo-Vap Temperature Display Reading (°C)																		
Temperature - Uncorrected (°C)																		
Temperature - Corrected (°C)																		

Criteria:*		
90 < Initial Value < 410	Final Value (with empty tube) / Initial Value (without tube) =<67%	Temperature:+/- 2°C

*If values meet criteria, mark the Pass (P) cell. If values do not meet criteria, mark the Fail (F) cell and place an "X" in the Out of Service cell. The sensor may need replacing.

Record all non-routine maintenance, including sensor replacement, in the space provided below.

Maintenance Performed:								
Replaced Sensor		____1	_____2	____3	____4	_____5	____6	
Bubble Dislodging Procedure								
Water Bath (empty, clean, refill)								
Other								
Date & Initials:								

FEX035:06.05.15:6

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Attachment 10:
Zymark-specific Maintenance Instructions

Zymark Maintenance Procedures

Sensor Diagnostic Test

(Frequency = Required Weekly)

- Lift the concentrator's cover, remove any tubes, and turn the power on.
- Press the SELECT DISPLAYED CONDITION button within 4 seconds.
  The CONDITION and VALUE displays show the current software version, for example:
  [ ] [1.0]
  After 3 seconds the displays reflect the pushwheel settings.
  - ⇒ Press the CELL ONE button and keep it depressed for the next step.
    The CONDITION and VALUE displays show the maximum bath temperature rating for cell one's sensor. This tests the Sample Temperature Rating.
    [50] [°C]
  - ⇒ Release the CELL ONE button, the CONDITION and VALUE displays show

    COND. VALUE
    [1.2] [10]
    ↑ ↑
    Cell # Sensor Output Value

    The first digit displayed is the sensor location. The remaining digits are the sensor output value. In the above example, the sensor location is 1 and the sensor output value is 210.
  - ⇒ Record initial sensor output value.
  - ⇒ Place a clean, empty Zymark tube into the cell.
  - ⇒ Record the final sensor output value.
- Repeat the above process (marked with an arrow) for cells 2-6.
- When all sensors are tested, press ENDPOINT SELECT to exit the diagnostic.
- Repeat the entire process for all TurboVaps.

Cleaning and Refilling the Water Bath

(Frequency = Recommended Weekly)

- Turn the unit off and unplug the power cord. Remove all glassware. Remove the top plate.
- Carefully lift the rack out of the bath.
- Siphon off the water in the bath.
  - ⇒ Close siphon bulb vent.
  - ⇒ Place siphon's suction tube in the water bath.
  - ⇒ Place drain tubing in sink.
  - ⇒ Squeeze siphon bulb to start.
- Wipe and rinse the bath walls. Clean the rack by rinsing with water.
- Replace the rack in the water bath. Replace the top plate.
- Place a concentrator tube in *five* positions.
- Pour approximately 1L of distilled water through the empty position.
- Add 15 drops of *Clear Bath*.
- Add more distilled water until the level is as high as the initial solvent level in the sample tube without causing an overflow when all six tubes are in position.
- Plug in the power cord and turn the power on.
- Allow 20-30 minutes for the bath to reach temperature, the air to come out of solution, and for most bubbles to dissipate.
- Perform the Bubble Dislodging Procedure.

Bubble Dislodging Procedure

Insert a clean Zymark tube. Using a pumping motion, raise and lower the tube approximately an inch, several times.

FEX082:02.18.13:1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Attachment 11
Method Prep Work Instruction

Aqueous Prep Volumes Summary
(CLLE)

Analysis Method	Prep Method	Initial Volume	Final Volume	Solvent	Dept	Box
625 8270	625 3520C	1000mL	1mL	$MeCl_2$	SM	M
625 8270	625_LVI 3520C_LVI	250mL	0.5mL*	$MeCl_2$	SM	M
8270_LL	3520C	1000mL	1mL	$MeCl_2$	SM	LL
8270_LLPAH	3520C	1000mL	1mL	$MeCl_2$	SM	LL
8270_LLPAH	3520C_LVI	250mL	0.5mL*	$MeCl_2$	SM	LL
680	680	1000mL	1mL	Hexane**	SM	M680
8015_DRO	3520C	1000mL	1mL	$MeCl_2$	SG	F
608 8081_8082	608 3520C	1000mL	10mL	Hexane**	SG	White Box
608 8081_8082	608_LVI 3520C_LVI	250mL	2.5mL	Hexane**	SG	White Box
8081 / 8082_LL	3520C_LVI	250mL	0.5mL*	Hexane**	SG	P

* When Final Volume is 0.5mL for ANY test, use the Zymarks designated for 0.5mL volumes.

** Requires solvent exchange with HEXANE during concentration.

Blue Tape = Solvent Lot Test. Use same lot of solvent to bring to Final Volume.

Yellow Tape = LVI prep. See above for Final Volumes.

Red Tape = Initial Volume reduced from SOP amount. Final Volume must be reduced accordingly.

Green Tape = KD concentration required.

Orange Tape = Final Volume to be reduced to 1/2 routine. Initial Volume is at SOP volume.

Attachment 12: Sample Spiking Volumes

Method	Spike Mix	Volume	Surrogate Mix	Volume	Initial Volume	Final Volume
8081/8082	608wkSpike	1mL	PESTwkSurr	1mL	1000mL	5mL
	1660wk Spike	1mL	---	---	---	---
8015	DIESELWK (DRO)	1mL	DROwkSURR	1mL	1000mL	1mL
8270	EX8270SPKL1	1mL	8270RTSURR	1mL	1000mL	1mL
	8270 L1/S9	20ul	---	---	---	---
	Ap9Mix 1	500uL	---	---	---	---
	Ap9Mix 2	500uL	---	---	---	---
8270LL	EX8270SPKL1	100uL	LLBNAwkSurr	1mL	1000mL	1mL
	8270 L1/S9	5uL	---	---	---	---
	Ap9Mix 1	50uL	---	---	---	---
	Ap9Mix2	50uL	---	---	---	---
680	680wkSpike	1mL	680wkSurr	1mL	1000mL	1mL
8081/8082_LVI	608wkSPIKE	250uL	PESTwkSurr	250uL	250mL	2.5mL
	1660wkSPIKE	250uL	---	---	---	---

18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated detergent to reflect what is currently in use. Section 6.3
- Revised final volume determination procedures. Section 10.5
- Revised sample spiking volumes to reflect actual laboratory practice. Attachment 12

MICROWAVE AND SONICATION EXTRACTION PROCEDURES
(Methods: EPA 3546 and EPA 3550C)

Approvals (Signature/Date):

Whitney Palefsky — April 4, 2019
Whitney Palefsky — Date
Quality Assurance Manager

Kimberly Chamberlain — April 4, 2019
Kimberly Chamberlain — Date
Technical Director
Department Manager
Environmental Health & Safety Coordinator

1.0 Scope and Application

This SOP gives the procedures for extracting semivolatile organic compounds (SVOCs) via microwave and sonication procedures.

Microwave extraction is used as the laboratory's default procedure for routine soil matrices and methods. Sonication extraction is used for tissue samples, only, as outlined in Attachment 12.

The following classes of SVOCs can be extracted using the procedures outlined in this SOP:

Analyte Class	Analytical Method	Analytical SOP
PCB Homologues	EPA 680	SA-SM-007
Diesel Range Organics (DRO)	EPA 8015C	SA-SG-070
Oil Range Organics (ORO)	EPA 8015C	SA-SG-070
Product Identification	EPA 8015C	SA-SG-070
Organochlorine Pesticides	EPA 8081A EPA 8081B	SA-SG-045
PCBs as Aroclors	EPA 8082 EPA 8082A	SA-SG-045
SVOCs	EPA 8270D	SA-SM-033

This SOP also includes the following information:

- Work Instructions for the extract clean-up procedures employed by the laboratory. These clean-up procedures include copper (sulfur) and sulfuric acid as outlined in Attachment 7 and Attachment 8.
- Work Instruction for the determination of the percent lipid content of a tissue sample as outlined in Attachment 13.

Note: Soil results are routinely reported on a dry weight basis. The procedure for determining the moisture content of soil samples is given in SOP SA-GE-190: *Solid/Residue Determinations*.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Microwave Extraction Procedure

A known weight of a sample is transferred to a Teflon extraction vessel. The sample is

spiked with surrogate compounds and an analyte-specific solvent is added to the vessel. The vessel is placed in the microwave instrument, and the sample extracted at an elevated temperature and pressure. The vessel is cooled to room temperature, the extract is passed through sodium sulfate to remove the water from the sample, and the extract is collected in a concentration tube. The solvent is evaporated, and the extract is concentrated to an appropriate final volume. Attachment 5 defines the solvents and extraction conditions for the applicable analytical procedures.

2.2 Extract Clean-up Procedures

Sulfur (copper) and acid clean-up procedures can be performed to remove interferences from extracts as outlined in the Attachment 7 and Attachment 8.

2.4 Tissue Extraction Procedures

Preparation of tissue samples utilizes sonication extraction. The procedure is summarized in Attachment 12.

2.3 Percent Lipids Determination Procedures

Percent lipids analysis is routinely requested for tissue samples prepared via sonication procedures. The percent lipids procedure is summarized in Attachment 13 and involves weighing the extract before and after drying overnight. The ratio of the two weights is equivalent to the percent lipids content of the sample.

2.5 Method References

This SOP is based on the following methods: EPA Method 3546 (for microwave) and EPA Method 3550C (for sonication). Biological tissue-specific guidance is taken from EPA Region IV Method 0B 10.90 *"Extraction and Analysis of Organics in Biological Tissue."*

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Acetone, hexane, methylene chloride, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to preparation and/or analysis to reduce or eliminate the interferences.

4.2.2 Samples for PCB-only analysis are often acid cleaned in accordance with Attachment 8.

4.2.3 In addition to acid clean-ups, copper clean-ups can be performed to remove interferences from extracts as outlined in Attachment 7.

4.2.4 Interfering contamination may occur when a sample containing low concentrations of analytes is processed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be processed first.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of the chemicals used in this procedure has not been precisely defined. Each chemical must be treated as a potential health hazard, and exposure to these chemicals must be minimized.

Methylene chloride is a carcinogen and an irritant. It causes irritation to the respiratory tract and has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Methylene chloride may be absorbed through the skin and can cause irritation and pain to the skin and eyes.

Hexane and acetone are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Compressed gases have specific hazards. The employee must be familiar with the MSDS/SDS for each of the compressed gases. The employee must also be familiar with the compressed gas section (Section 11) of the Environmental Health and Safety Manual.

The microwave procedure produces high temperatures (~100°C) and high pressures (~200psi) during the extraction process. The analyst must be familiar with the manufacturer's instructions for properly loading and unloading samples from the device and for the safe use and handling of the extraction vessels.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Florisil	Irritant	10mg/m^3 TLV 5mg/m^3 PEL	May cause irritation if inhaled or adsorbed through the skin.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
TWA = Time Weighted Average STEL = Short Term Exposure Limit TLV = Threshold Limit Value PEL = Permissible Exposure Limit			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Microwave extractor – CEM MARS Model 907501, includes carrousel. The unit has temperature feedback control and is capable of sensing temperature within +/- 2.5°C and adjusting microwave field output power within two seconds of sensing.

Microwave extraction vessels – Teflon

Zymark TurboVap II concentration device or equivalent – The instrument must be vented into an operating fume hood.

Concentration Tubes – 200mL with 1.0mL tip. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

6.2 Volumetric Containers

Various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 6 for Glassware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Disposable Graduated Pipettes	Various	QUANTITATIVE	Final Volume Determination	Per Lot	Accuracy = 2% Precision = 1%
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Dilution, Reagent, and Standard Preparation	None (Class A)	None (Class A)
Autosampler Vials	1.5mL	QUANTITATIVE	Extract storage and final volume determination for ≤1.0mL	Per Lot	Accuracy = 2% Precision = 1%
Extract Vials	12mL	Qualitative	Storage of extracts for final volumes ≥1.5mL	None	None
Disposable Transfer Pipettes	Various	Qualitative	Sample Transfer	None	None

6.3 Laboratory Supplies

Detergent – FL-70 or equivalent, used for washing non-disposable labware

Filter Funnel - Large glass funnels with 18.5cm filter paper

Stainless Steel Spatulas

11mm crimp-type capper and decapper

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

8oz glass soil jars – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Acetone, hexane, and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Blank Matrix – Ottawa Sand. Purified in the oven by heating on the self clean cycle for 3 hours in a shallow tray. Used for the preparation of soil QC samples.
Storage: sealed glass jar at room temperature
Expiration:
Unopened: Manufacturer's expiration date
Opened: 1 year from purification date

7.2.2 Sodium Sulfate, granular, anhydrous – Purified in the oven by heating on the self clean cycle for 3 hours in a shallow tray. Used as a drying agent.
Storage: sealed glass jar at room temperature
Expiration:
Unopened: Manufacturer's expiration date
Opened: 1 year from purification date

7.2.3 Methylene Chloride – pesticide grade or better. Used as extraction solvent.
Storage: Flammables cabinet
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date

7.2.4 Acetone – pesticide grade or better. Used for glassware rinsing and as solvent for some spiking mixes and extraction solvent.
Storage: Flammables cabinet
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date

7.2.6 Hexane – residue grade or better.
Storage: Flammables cabinet
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*. Certificates of analysis or purity must be received with all purchased standards, and scanned and attached to reagents in TALS.

All of the standards used from this SOP must be stored in the refrigerator at a temperature of 0-6°C (i.e., less than 6°C but not frozen). Unless otherwise noted, all purchased standards have an expiration date of 6 months from date of opening, and all prepared standard have an expiration date of 3 months from date of preparation.

Note: This standards list is comprised of the routine standards used by the laboratory. Information on project-specific, non-routine standards is found in the Reagent Module in TALS.

7.3.1 Purchased Standards

7.3.1.1 Organochlorine Pesticide Surrogate Stock, 200ug/mL – Purchased from Restek (catalog # 32000)
TALS Reagent Name: SGPESTSURR_XXXXX

7.3.1.2 Pesticide A\B Mix, varied concentrations – Purchased from Restek (Custom# 32415.sec)
TALS Reagent Name: SG_ABICV_XXXXX

7.3.1.3 Aroclor 1016/1260 Spike Mix, 1000ug/mL – Purchased from Restek (catalog # 32039)
TALS Reagent Name: SG1660CAL_XXXXX

7.3.1.4 O-terphenyl Standard, 10000ug/mL – Purchased from NSI (catalog # C-1341H-TP)
TALS Reagent Name: SG_OTP_XXXXX
#2 Diesel Fuel, 100mg/mL – Purchased from Accustandard (catalog # FU-009-D-200X)
TALS Reagent Name: SGDROCAL_XXXXX

7.3.1.5 Decachlorophenyl 13C12. 40ug/mL – Purchased from Cambridge Isotopes (catalog # EC-1410-3)
TALS Reagent Name: DB(680)SURR_XXXXX

7.3.1.6 680 Nonachlorobiphenyl Solution, varied concentrations – Purchased from Ultra Scientific (catalog # RPC-081-S)
TALS Reagent Name: PCB RTmix_XXXXX

7.3.1.7 BNA Surrogate Standard, 100ug/mL – Purchased from Restek (catalog # 568728)
TALS Reagent Name: 8270RTSURR_XXXXX

7.3.1.8 8270 Surrogate Standard, 100ug/mL – Purchased from Restek (part # 570814)
TALS Reagent Name: 8270RTSSURR_XXXXXX

7.3.1.9 SM O-Terphenyl Standard, 2000ug/mL – Purchased from Restek (catalog # 31066)
TALS Reagent Name: O-Terp Std_XXXXX

7.3.1.10 AP9 Spike Mix – Purchased from NSI (catalog number is Q-5131)
TALS Reagent Name: EXAP9SPK_XXXXX

7.3.1.11 8270 List 1/Standard 1, varied concentrations – Purchased from Restek (catalog # 567672)
TALS Reagent Name: 8270 L1/S1_XXXXX

7.3.1.12 8270 List 1/ Standard 11, 2000ug/mL – Purchased from Restek (catalog # 569732)
TALS Reagent Name: 8270 L1/S11_XXXXX

7.3.1.13 8270 List 1/ Standard 9, 2000ug/mL - Purchased from Restek (catalog # 569730)
TALS Reagent Name: 8270 L1/S9_XXXXX

7.3.1.14 8270 List 1/ Standard 10, 2000ug/mL – Purchased from Restek (catalog # 569731)
TALS Reagent Name: 8270 L1/S10_XXXXX

7.3.1.15 680 Calibration Concentration Mix, varied concentrations – Purchased from Ultra (part number CB681-MN)
TALS Reagent Name: 680conCal_XXXXX

7.3.1.16 Lindane 13C6 Surrogate, 100ug/mL – Purchased from Cambridge (catalog # CLM128)
TALS Reagent ZName: Lindane-13C6_XXXXX

7.3.2 Prepared Standards

7.3.2.1 Working Pesticide / PCB – prepared by adding 1.25mL Pesticide Surrogate Stock (200ug/mL) diluted to a final volume of 500mL in methanol.
TALS Reagent Name: PESTwkSURR_XXXXX

7.3.2.2 Working Pesticide Spike Mix – prepared by diluting 1.0mL of INTPESTABICV (from SG dept) to 100mL of methanol.
TALS Reagent Name: 608wkSPIKE_XXXXX

7.3.2.3 Working PCB Spike Mix – prepared by adding 1.0mL of Aroclor 1016/1260 Spike Mix diluted to a final volume of 100mL in methanol.
TALS Reagent Name: 1660wkSPIKE_XXXXX

7.3.2.4 Working DRO Surrogate Mix – prepared by adding 1.0mL of O-terphenyl standard (10000 ug/mL) diluted to a final volume of 500mL in acetone.
TALS Reagent Name: DROwkSURR_xxxxx

7.3.2.5 Working DRO Spike Mix – prepared by adding 1.0mL of #2 Diesel Fuel (100 mg/mL) diluted to a final volume of 100mL in acetone.
TALS Reagent Name: DIESELWK_xxxxx

7.3.2.6 Working 680 Surrogate Mix – prepared by adding 3.125mL of Decachlorobiphenyl 13C12 (40ug/mL), 1mL Lindane13C6, diluted to a final volume of 50mL in acetone.
TALS Reagent Name: 680wkSURR_xxxxx

7.3.2.7 Working 680 Spike Mix – prepared by adding 1.0mL of Concentration Calibration Standard Mix (varied concentrations) and 1.25mL of PCB RT Mix diluted to a final volume of 25mL in acetone.
TALS Reagent Name: 680wkSPIKE_xxxxx

7.3.2.8 Working LLBNA Surrogate Mix – prepared by adding 1mL 8270_SURR and 0.5mL of O-Terp std in 500mL of acetone.
TALS Reagent Name: LLBNAwkSUR_xxxxx

7.3.2.9 Working EX 8270L1 Spike – prepared by adding 10mL 8270 L1/S1, 5mL of 8270 L1/S10, 5mL of 8270 L1/S1 in 100mL of methanol.
TALS Reagent Name: EX8270SPKL1

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Soil Samples

Soil samples are routinely collected in 8z glass soil containers.

Samples must be iced at the time of collection. Sample receipt temperature must be 0-6°C (i.e., less than 6°C with no frozen samples). Samples and extracts must be stored in the refrigerator at 0-6°C until preparation and/or analysis. Samples must be prepared within 14 days of collection.

Refer to the associated analytical SOPs for information on analysis holding times.

8.2 Tissue Samples

Tissue samples are routinely collected in unpreserved glass containers, with the size dependent upon the type of tissue being collected, or wrapped in aluminum foil. Plastic jars or plastic baggies can be used.

Upon receipt, tissue samples must be placed in the freezer at -10° to -20°C, if preparation cannot be completed that day, and must be kept frozen until the time of preparation. A holding time of six months from the date of collection for frozen fish fillets is recommended by Alabama Department of Environmental Management (ADEM) and will be used for all biological tissues.

Once the tissue sample has been thawed, it must be stored at 0-6°C (less than 6°C but not frozen), and preparation must take place within 14 days.

Tissue samples must be homogenized prior to preparation since organic compounds are generally not evenly distributed throughout biological tissue. The entire sample should be ground into a homogenous consistency. To do this, chop the sample into one inch or smaller

chunks and place in the blender or meat grinder. Note: Slightly frozen tissue is easier to work with at this point. Add dry ice to the blender or meat grinder. Blend or grind on appropriate power level until the tissue is thoroughly ground and homogenized. Inspect the tissue for the presence of "chunks" of tissue. Remove the well-homogenized tissue and blend the remaining "chunks" until homogenized. Mix the tissue thoroughly and separate into 10g portions for the required analyses. If the extraction is not to take place soon after homogenization, the homogenized tissue or the individual 10g portions can be frozen. Note, however, that the holding begins with the date of the original thawing process.

Refer to the associated analytical SOPs for information on analysis holding times.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 of each analytical SOP provide requirements for evaluating QC data.

9.1 Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items. The minimum default QC items performed for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

The routine container supplied for this method is a 8oz (200g) container. Generally 15g of sample is used for the extraction Reduced sample initial amounts may be necessary to achieve the required batch matrix spike frequency; Note: Spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample submitted to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). Insufficient sample is defined as receiving less than a total of 60g. If insufficient sample is provided to perform the MS/MSD, an NCM must be initiated on all samples within the batch to denote this situation.

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

Batch QC must meet the criteria given in Attachment 3 of the associated analytical SOP.

9.2 Instrument QC

The instrument QC for the analytical procedures associated with this extraction procedure are given in the analytical SOPs listed in Section 1.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and*

Corrective Action Procedures and the QC Summary Table in Attachment 3 of the associated analytical SOPs. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Field Sample and QC Sample Preparation Procedures

Utilizing the TALS Backlog program, select and schedule extraction batches based on the analysis methods requested. Generate the Sample Request Form (SRF) and print any TALS Worksheet, Method, or Login Notes.

Note: Some agencies and clients have specific extraction and/or spiking requirements. Verify the Project Requirement Summary (PRS) and TALS Notes prior to beginning the extraction to ensure state and/or project-specific requirements are followed.

10.1.1 Remove the samples from the refrigerator and allow them to come to room temperature.

Scan the samples into the TALS batch. Add the appropriate QC as per section 9.1. Additional LCS, MS, and MSD may be required dependent on the spiking list requested.

Prior to extraction, the samples must be homogenized in accordance with SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples*.

Note: Prior to homogenization, the amount of standing water present in the sample container must first be visually confirmed. If the amount of standing water present is approximately 25% or greater, the standing water must be decanted. An NCM must be initiated to denote this situation.

10.1.2 To prepare the method blank and laboratory control samples (LCS/LCSD), weigh an amount of Ottawa sand equivalent to that used for samples into a labeled a microwave extraction vessel. Add the appropriate spiking mix to the LCS/LCSD. Process the method blank and LCS/LCSD in the same manner as the associated field samples, beginning with Section 10.1.5.

Refer to Attachment 5 for sample weights for each of the analytical methods.

Refer to Attachment 14 for sample spiking volumes.

Note: The addition of the LCS/LCSD spiking solutions must be witnessed by another analyst to ensure the correct spiking solution and the proper volume of spiking solution is added to each sample.

10.1.3 To prepare the field samples, weigh the appropriate amount of homogenized sample into a labeled microwave extraction vessel. Refer to Attachment 5 for sample weights for each of the analytical methods.

10.1.4 To prepare the matrix spike (MS) and matrix spike duplicate (MSD), weigh two additional portions of a sample in the batch into labeled microwave extraction vessels. Add the appropriate spiking solution to the MS/MSD. Process the MS/MSD in the same manner as the associated field samples, beginning with Section 10.1.5.

The addition of the MS/MSD spiking solutions must be witnessed by another analyst to ensure the correct spiking solution and the proper volume of spiking solution is added to each sample.

Note: The MS/MSD or MS/SD may be specified by the client or may be chosen at random from the samples in the batch.

10.1.5 Add the appropriate surrogate spiking solution (as listed in Attachment 5) to each sample and QC item.

Note: The addition of all spiking solutions must be witnessed by another analyst to ensure the correct spiking solution and the proper volume of spiking solution is added to each sample.

Note: If the final volume of the extract must be reduced to achieve the required reporting limits, the amount of surrogate and matrix spiking solutions must be reduced proportionately.

10.1.6 Add 15mL of Acetone to each Teflon tube.

**NOTE for 8015_DRO, Acetone is NOT utilized. Proceed to section utilizing Methylene Chloride ONLY.

10.1.7 Make sure that the plugs are in place and caps are fastened properly on each extraction vessel.

Shake Teflon vial for approximately 15 seconds by hand. Sample should be free-flowing in the tube.

After shaking, add 15mL of Methylene Chloride or Hexane (see attachment 5 for appropriate solvent) to the Teflon tube.

Note: Add sufficient solvent to ensure the sample is completely covered in the tube. For method 8015, this will be a total of 30mls of Methylene chloride.

10.1.8 Place the vessels on the carrousel in the appropriate slot.

10.1.9 Select the program for the extraction on the keyboard. There are two programs available dependent on the number of samples (i.e., less than 24 samples or greater than 24 samples). The default parameters are as follows:

Parameter	Program
Time	10 minutes to reach 110°C; then hold 10 minutes
Temperature	110°C

Pressure	50-150psi
Watts	<24 Samples: 800 watts >24 samples: 1600 watts

Once the microwave extraction has completed, perform the following steps:

- Allow the extracts to reach room temperature.
- Add a piece of folded filter paper to a clean steel funnel.
- Add approximately 30g of baked sodium sulfate to the funnel.
- Place the funnel over a Zymark tube.
- Working under a hood, rinse the filtering funnel and labeled Zymark tube with the appropriate solvent (methylene chloride or hexane as outlined in Attachment 5). Discard the solvent in the waste container.
- Working with one sample at a time and continuing to work under a hood, pour the extract from the extraction vessel into the funnel. It is important that the extract contact the sodium sulfate to remove the water from the extract.
- Rinse the microwave vessel with approximately 10mL of extraction solvent and pour through the funnel and collect in the Zymark tube. Repeat with an additional 10mL aliquots of solvent.
- Rinse the funnel with a 10mL aliquots of solvent and allow the solvent to drain and collect in the tube.
- Place the tube into a cell on the evaporation unit. Repeat for remaining samples in the batch.
- Concentrate as described in Section 10.4.

10.2 Zymark Concentration Procedures

Note: If the volume of sample extracted has been reduced to provide sufficient sample to meet state or other program QC requirements, the final volume of the extract must be reduced proportionately.

10.2.1 Turn on TurboVap evaporation unit. Set the water bath temperature to 50°C. Set the gas pressure to zero initially for each cell in the TurboVap unit.

10.2.2 Pre-rinse each concentration cap on the TurboVap evaporation unit with methylene chloride.

Note: Rinse the cap with methylene chloride each time a new sample is added.

10.2.3 If the sample extracts have been collected in any container other than a Zymark tube, transfer the extract to a Zymark tube. Place the Zymark tube into a cell of the evaporation unit. Repeat for all samples in the batch. The remaining extract must be tightly covered with a piece of aluminum foil to minimize evaporation of the solvent.

10.2.4 Carefully close the cover of the Zymark TurboVap unit completely. Make sure that each tube is seated properly and that the individual covers are positioned directly over each tube.

Note: When the elongated tubes are used, care must be taken to position the tubes to avoid breaking the tube covers and to completely close the instrument cover.

10.2.5 Concentrate the extracts at a constant temperature of 50°C. Start the concentration with a pressure of 4-5psi. Gradually increase the psi until dimpling is seen on the top layer. Once the extract volume reaches approximately 70mL, turn the pressure up to 15psi.

10.2.6 Concentrate the extract until the cell alarm sounds. This indicates that the extract volume is near or below 1mL.

10.2.7 Remove Zymark concentration tubes from evaporation unit and transfer to storage vials as outlined in Section 10.6.

10.5 Final Volume Determination and Extract Transfer Procedures

Extracts are quantitatively transferred from the concentration tube to the storage and/or autosampler vial as follows:

1) For methods that require a final volume of 1.0mL, the autosampler vial is used to determine the final extract volume. The volume markings on these vials must be verified per lot.

   Concentrate the extract to less than 1mL. Using a transfer pipette, rinse the tube with the extract to rinse off any extract that has deposited/collected on the walls or the tip of the tube. Transfer the extract from the tube to an autosampler vial. Add a small amount of methylene chloride to the tube, and use it to further rinse the tube. Use this rinsate to establish the 1.0mL final volume using the markings on the autosampler vial.

   Note: If the extract cannot be evaporated to 1mL due to the matrix of the sample, do not force the extract to concentrate. Adjust to an appropriate final volume (e.g., 2mL or 5mL) using the process outlined below.

2) For methods that require a final volume of greater than 1mL , the disposable pipette is used to determine the final extract volume. The volume markings on these pipettes must be must be verified per lot.

   Concentrate the extract to approximately 1mL. Using a 5mL pipette, withdraw the extract from the tube while simultaneously and slowly adding a small amount solvent to rinse the tube and to achieve the final volume required, as measured in the pipette. Alternatively, a class A volumetric container may be used to determine final volume. Transfer the entire contents of the zymark tube to the volumetric. Rinse the zymark with the appropriate final solvent and transfer to the volumetric until the sample reaches the marks.

   Transfer to the appropriate vial for storage.

Mark the volume of the extract on the side of the storage container/vial to allow the analyst to judge whether the sample extract has evaporated during storage and handling. Store concentrated extracts at 0-6°C until time of analysis.

10.7 Analysis

Details on sample analysis are given in the associated analytical SOPs listed in Section 1.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data reduction and review tasks include the following items:

- Employ spike witness procedures to ensure proper spiking solutions and volume are used.
- Ensure all TALS batch Data Type fields are completed so that proper traceability is maintained.
- Check to ensure that each sample extract is properly identified and that the extracts are transferred to the analytical groups with proper documentation.
- Mark the final volume of the extract on the outside of the storage container as a check on extract evaporation.
- QC items must be treated in the same manner as samples.
- Ensure samples are evaporated at the appropriate rate. Unacceptable surrogate or spike recoveries may be attributed to evaporating the samples too quickly or to improper solvent exchange.
- Document any unusual circumstances and procedural violations using the TALS Nonconformance Module. This can include: samples problematic matrices such as color, odor, or emulsions; initial or final amount changes; deviations to the SOP, insufficient sample provided to perform batch QC (such as MS/MSD), etc.

Batch data must be reviewed and evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

Additional details on data reduction procedures are given in the associated analytical SOPs.

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.2 Calculations

Details on sample calculations are given in the associated analytical SOPs listed in Section 1.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits.*

Note: Tissue matrices are non-routine, and the laboratory is not currently NELAC certified for this matrix. Additionally, Ottawa sand (as opposed to a "true" tissue matrix) is used for tissue QC samples unless the laboratory is specifically requested by the client to procure a "true" tissue matrix. As such, the soil MDL is converted to tissue units and used to satisfy the MDL requirement for this matrix.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for the non-SW846 procedures are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items for the non-SW846 procedures. The control limits for the batch QC items (LCS and MS/MSD) for the SW846 procedures are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items for the SW846 procedures.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

Note: Tissue matrices are non-routine, and the laboratory is not currently NELAC certified for this matrix. Additionally, Ottawa sand (as opposed to a "true" tissue matrix) will be used for tissue QC samples unless the laboratory is specifically requested by the client to procure a "true" tissue matrix. As such, the soil DOC is used to satisfy the DOC requirement for this matrix.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Methylene chloride extracts – Dispose according to characterization on sample disposal sheets. If non-hazardous, transfer extract to chlorinated waste container. If hazardous, transfer to hazardous waste department for storage.

- Standards containing methylene chloride - Transfer to a satellite container designated for chlorinated waste and transfer to waste disposal department when the container is full.

- Methylene chloride waste – Transfer to a satellite container designated for chlorinated waste and transfer to waste disposal department when the container is full.

- Hexane extracts – If non-hazardous, transfer to flammable waste containers and dispose of as flammable waste. If hazardous, transfer to the waste disposal department for disposal as hazardous waste.

- Standards containing hexane - Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.

- Flammable waste (hexane or acetone from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.

- Aqueous acidic waste from samples – Collect into the disposal area and neutralize before release to the sewer system.

- Silica Gel Contaminated with methylene chloride from Clean-up Process – Transfer to a satellite container designated for chlorinated waste and transfer to waste disposal department when the container is full.

- Flammable Waste (acetone from extracts, rinsings, standards, and carbon disulfide) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.

- Methylene chloride used to rinse glassware, etc. – Transfer to a satellite container designated for chlorinated waste and transfer to waste disposal department when the container is full.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-010: *Validation of New Analytical Capabilities and Instrumentation*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Test Methods for Evaluating Solid Waste*, Third Edition (Updates III and IV), SW-846; EPA Office of Solid Waste and Emergency Response: Washington, DC.
    - Chapter 4: *Organic Analytes*; Revision 3, December 1996
    - Chapter 4: *Organic Analytes*; Revision 4, February 2007
    - EPA 3500C: *Organic Extraction and Sample Preparation*; Revision 3, February 2007
    - EPA 3550C: *Ultrasonic Extraction*; Revision 3, February 2007
    - EPA 3600C: *Cleanup*; Revision 3, December 1996
    - EPA 3645: *Microwave Extraction*; Revision 0, February 2007
    - EPA 3660B: *Sulfur Cleanup*; Revision 2, December 1996
    - EPA 3665A: *Sulfuric Acid/Permanganate Cleanup*; Revision 1, December 1996
- Tekmar sonicator instruction manual.
- *"Extraction and Analysis of Organics in Biological Tissues"*, Method 0B 10/90, USEPA Environmental Services Division, Region IV, Analytical Support Branch, Athens, GA.

16.0 Method Modifications

16.1 This procedure may be modified to analyze other matrices (e.g., wipe samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project. The instructions for the preparation of wipe samples are given in the Wipe-specific Work Instruction (FQA086).

16.2 The initial amount and final volumes used for pesticide and PCB extractions have been changed from 30g and 10mL to 15g and 10mL.

16.3 Some additional batch QC items may be performed which differ from those specified in the reference methods (e.g., LCSD) to satisfy common state regulatory requirements and/or client requests for precision data and/or to facilitate scheduling and data evaluation. The laboratory has incorporated the batch QC items as outlined in Section 9.1 which include method blank, LCS, and MS/MSD, and LCSD (if insufficient sample is provided for MS/MSD).

16.4 The reference methods do not address how to proceed when solid samples contain a substantial amount of water. Therefore, the laboratory has implemented the requirement to decant standing water prior to homogenizing solid samples if amount of standing water is >/= 25% of total sample volume.

16.5 The reference method states that sodium sulfate is used to dry samples prior to microwave extraction. Samples are dried with sodium sulfate after the microwave extraction is completed to remove excess water.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Extraction Solvents and Volumes
Attachment 6: Sample and QC Sample Spiking Solutions
Attachment 7: Sulfur Cleanup
Attachment 8: Sulfuric Acid Cleanup
Attachment 9: Zymark Sensor Diagnostic Test and Maintenance Log
Attachment 10: Zymark Instructions for the Sensor Diagnostic Test and Cleaning and Refilling the Water Bath
Attachment 11: Glassware Cleaning Procedures
Attachment 12: Tissue Extraction Procedures
Attachment 13: Determination of Percent Lipids
Attachment 14: Sample Spiking Volumes

Attachment 1:
SOP Summary

Sample Preparation Summary

Microwave Extraction Procedure

A known weight of a sample is transferred to a Teflon extraction vessel. The sample is spiked with surrogate compounds and an analyte-specific solvent is added to the vessel. The vessel is placed in the microwave instrument, and the sample extracted at an elevated temperature and pressure. The vessel is cooled to room temperature, the extract is passed through sodium sulfate to remove the water from the sample, and the extract is collected in a concentration tube. The solvent is evaporated, the extract is exchanged into another solvent – if required by the analytical procedure, and the extract is concentrated to an appropriate final volume. Attachment 6 defines the solvents and extraction conditions for the applicable analytical procedures.

Refer to Attachment 13 for the sonication extraction procedures for tissue samples.

Sample Analysis Summary

Samples are analyzed in accordance with the appropriate method-specific SOP.

Analyte Class	Analytical Method	Analytical SOP
PCB Homologues	EPA 680	SA-SM-007
Diesel Range Organics, Oil Range Organics, Product Identification (DRO/ORO)	EPA 8015C	SA-SG-070
Organochlorine Pesticides	EPA 8081B	SA-SG-045
PCBs as Aroclors	EPA 8082A	SA-SG-045
SVOCs / BNAs	EPA 8270D	SA-SM-033

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time
Soil	8oz glass soil jar	15g (See Attachment 6)	15g (See Attachment 6)	Not Applicable	Not Applicable	4°C1	14 days
Tissue	Various	10g	10g	Not Applicable	Not Applicable	<-10°C	6 months frozen; 14 days from thawing

[1]Samples are collected on ice and maintained at <6°C with no frozen samples.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Up to 20 field samples extracted together within 24-hour time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample (LCS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample provided for MS/MSD	Refer to analytical SOP	Refer to analytical SOP
Matrix Spike (MS)	One set per batch	Refer to analytical SOP	Refer to analytical SOP
Matrix Spike Duplicate (MSD)	One set per batch	Refer to analytical SOP	Refer to analytical SOP
Initial Demonstration of Capability (IDOC)	Initially, per analyst/matrix/method/analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 (Unsupervised work cannot begin until acceptable IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst/matrix/method/analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Frequency	Criteria	Corrective Action
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:
- type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance
Refer to the instrument manufacturer's guides for trouble-shooting items.

Chipped or broken glassware must be removed from service.

Microwave Maintenance
A microwave power/wattage check and temperature calibration should be performed annually per the manufacturer's instructions. Additionally, the inside of the microwave unit should be wiped down on an as needed basis, with a paper towel moistened with reagent water.

The service technician is contacted if the equipment malfunctions. Unacceptable recoveries for laboratory control samples (LCS) could indicate a service call is warranted.

Zymark Maintenance
It is recommended to change the water in the water bath weekly. Add 1-2 drops of Clear Bath to prevent bacteria and algae growth. Methylene chloride that dissolves in the water bath will damage the sensors.

The Zymark sensor diagnostic test must be performed weekly. If the sensors do not meet criteria the sensor may need replacing. Refer to the manufacturer's manual for replacement procedures if necessary.

The thermometer for the Zymark must be calibrated in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique, or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5:
Extraction Volumes and Solvents

Method	Analyte Class	Extraction Amount (g)	Extraction Solvent	Final Volume (mL)
EPA 8081B	Chlorinated Pesticides	15	Hexane	5.0
EPA 8082A	PCBs			
EPA 8270D	BNAs	15	$MeCl_2$	1.0
EPA 680	PCB Homologues	15	Hexane	1.0
EPA 8015C	DRO, ORO, & Product Identification	15	$MeCl_2$	1.0

Note: Other extraction and/or final solvents or solvent ratios may be used provided they are fully validated in accordance with SOP SA-QA-010: *Validation of New Analytical Capabilities and Instrumentation*.

Attachment 6:
Sample and QC Sample Spiking Solutions

Method	Analyte Class	Surrogate Standard	LCS/MS/MSD Standard
EPA 8081B	Chlorinated Pesticides	PESTwkSURR_xxxxx	608wkSPIKE_xxxxx
EPA 8082A	PCBs		1660wkSPIKE_xxxxx
EPA 8270D	BNAs	BNAwkSURR_xxxxx	BNAFULLSPK_xxxxx BENZIDINEwk_xxxxx
	_LLPAH	LLBNAwkSURR_xxxxx	BNAFULLSPK_xxxxx BENZIDINEwk_xxxxx
EPA 680	PCB Homologues	680wkSURR_xxxxx	680wkSPIKE_xxxxx
EPA 8015C	DRO	DROwkSURR_xxxxx	DIESELwk_xxxxx
	ORO		OROwkspike_xxxxx
	Product Identification		Project specific

Attachment 7:
Sulfur (Copper) Cleanup

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Work Instruction
FEX096: 12.16.16:2
Page 1 of 1

Sulfur Cleanup Procedures

Method: 3660B

Summary of Procedure
This procedure is based on EPA Method 3660B and is used in conjunction with the following SOPs:

Preparation Method	SOP #
EPA 3520C	SA-EX-030
EPA 3546	SA-EX-040
Analysis Method	**SOP #**
EPA 508	SA-SG-046
EPA 608 / EPA 8081A / EPA 8081B / EPA 8082 / EPA 8082A	SA-SG-045

The sulfur cleanup uses copper granules to eliminate elemental sulfur from PCB or pesticide extracts. Copper is added to the extract, and the vial is shaken. If sulfur is present, a black precipitate (copper sulfide) will form. The extract is treated with copper until no further precipitate is formed.

The method blank and LCS must be subjected to the same cleanup steps as the samples.

Perform all cleanup steps under a fume hood or in a well-ventilated area.

Reagents
Copper granules – the surface of the copper should be "shiny".

Cleanup Instructions
1. Ensure the sample extract has been exchanged into the applicable final solvent (e.g., MTBE for EPA 508, Hexane for EPA 608/8081B/8082A) prior to performing the sulfur cleanup procedure.

2. Add approximately 0.1g of "shiny" copper to the vial, and vortex for approximately two minutes.

   Note: If the extract is for EPA 614 or EPA 8141B in addition to one of the analytical methods listed above, transfer an aliquot of the extract to another vial for the copper cleanup.

3. If sulfur is present, a black precipitate will form. Allow the extract to sit for 2-3 minutes for any additional precipitate to form and settle out.

   If the precipitate does not settle out, additional copper treatments and/or filtration may be required. Contact the Technical Manager for instructions on how to proceed.

4. The sample is now ready for analysis as outlined in Section 10 of the associated analytical SOP.

Attachment 8:
Sulfuric Acid Cleanup

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Work Instruction
FEX097:12.16.16:2
Page 1 of 2

Sulfuric Acid Cleanup Procedures

Method: 3665A

Summary of Procedure
This procedure is based on EPA Method 3665A and is used in conjunction with the following SOPs:

Preparation Method	SOP #
EPA 3520C	SA-EX-030
EPA 3546	SA-EX-040
EPA 3580A	SA-EX-042
Analysis Method	**SOP #**
EPA 680	SA-SM-007
EPA 608 (PCBs only) / EPA 8082 / EPA 8082A	SA-SG-045

The acid cleanup procedure is used for PCBs and Toxaphene Congeners only. Sulfuric acid is added to the extract, and the vial is shaken. The layers are allowed to separate, and the sample layer is removed. Large organic-soluble compounds that are present in the sample will extract into the acid and are discarded.

Note: This procedure will destroy pesticide compounds. Sulfuric acid cleanups must only be used for PCB samples and cannot be used for samples requesting pesticides by EPA 8081B or EPA 608.

The method blank and LCS must be subjected to the same cleanup steps.

Perform all cleanup steps under a fume hood or in a well-ventilated area.

Reagents
Sulfuric acid (H_2SO_4) – reagent grade, concentrated

Cleanup Instructions
1. Ensure the sample extract has been exchanged into hexane prior to performing the sulfuric acid cleanup.
2. Transfer an aliquot of the extract to a vial for the cleanup. The recommended aliquot volume is 5.0mL.
3. Add approximately 2mL of concentrated sulfuric acid and cap the vial. Mark the vial to denote the total volume on the vial. Also mark the vial to denote the separation of the acid (bottom) layer and the extract (top) layer.
4. Ensure the vial cap is secure, and gently shake the vial. Open the vial, and allow any pressure that has built up to dissipate. Repeat these steps until no pressure is noted when the cap is opened, and then shake the vial for one additional minute. A vortex mixer can also be used.
5. Allow the extract (top) layer and the acid (bottom) layer to separate. This separation may take a few minutes or several hours depending on the nature of the sample

extract. Use the marks on the side of the vial to judge if the volume of extract (top) layer is the same as when it was originally added to the vial.

6. Remove the extract (top) layer from the vial using a disposable Pasteur pipette, and transfer the extract to a clean vial. If greater than 80% of the extract is recovered, no additional preparation is necessary.

7. If an emulsion has formed (i.e., there are bubbles or a cloudy area in between the top and bottom layers), add sodium sulfate crystals to the vial and gently stir the top layer with a glass rod. The sodium sulfate should help to break the emulsion so the top layer can be adequately recovered.

8. If the extract has color, perform an additional cleanup by adding 5mL more of concentrated acid and repeating Steps 3-6.

   Note: Use good judgment when determining how many acid cleanups to use. If it takes more than three cleanups to clean the extract, it is recommended to start over with a smaller aliquot of sample or to dilute the extract before proceeding with additional cleanups. A diluted extract can be concentrated back to the equivalent volume after the cleanup steps. Contact the Technical Manager for assistance with this task.

9. The sample is now ready for analysis as outlined in Section 10 of the associated analytical SOP.

Attachment 9:
Zymark Sensor Diagnostic Test and Maintenance Log

ZYMARK MAINTENANCE LOG

Turbo Vap #: _____ Date:_____ Initials:_____
Thermometer #:_____

Sensor Diagnostic Test

SENSOR #	1			2			3			4			5			6		
		P	F		P	F		P	F		P	F		P	F		P	F
INITIAL VALUE																		
FINAL VALUE																		
% VALUE																		
OUT OF SERVICE																		
Turbo-Vap Temperature Display Reading (°C)																		
Temperature - Uncorrected (°C)																		
Temperature - Corrected (°C)																		

Criteria:*		
90 < Initial Value < 410	Final Value (with empty tube) / Initial Value (without tube) =<67%	Temperature:+/- 2°C

*If values meet criteria, mark the Pass (P) cell. If values do not meet criteria, mark the Fail (F) cell and place an "X" in the Out of Service cell. The sensor may need replacing.

Record all non-routine maintenance, including sensor replacement, in the space provided below.

Maintenance Performed:												
Replaced Sensor		____1	_____2	____3	____4	_____5	____6					
Bubble Dislodging Procedure												
Water Bath (empty, clean, refill)												
Other												
Date & Initials:												

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Attachment 10:
Zymark-specific Maintenance Instructions

Zymark Maintenance Procedures

Sensor Diagnostic Test
(Frequency = Required Weekly)

- Lift the concentrator's cover, remove any tubes, and turn the power on.
- Press the SELECT DISPLAYED CONDITION button within 4 seconds.
  The CONDITION and VALUE displays show the current software version, for example:
  [ ] [1.0]
  After 3 seconds the displays reflect the pushwheel settings.
  - ⇒ Press the CELL ONE button and keep it depressed for the next step.
    The CONDITION and VALUE displays show the maximum bath temperature rating for cell one's sensor. This tests the Sample Temperature Rating.
    [50] [°C]
  - ⇒ Release the CELL ONE button, the CONDITION and VALUE displays show

    COND. VALUE
    [1.2] [10]
    ↑ ↑
    Cell # Sensor Output Value

    The first digit displayed is the sensor location. The remaining digits are the sensor output value. In the above example, the sensor location is 1 and the sensor output value is 210.
  - ⇒ Record initial sensor output value.
  - ⇒ Place a clean, empty Zymark tube into the cell.
  - ⇒ Record the final sensor output value.
- Repeat the above process (marked with an arrow) for cells 2-6.
- When all sensors are tested, press ENDPOINT SELECT to exit the diagnostic.
- Repeat the entire process for all TurboVaps.

Cleaning and Refilling the Water Bath
(Frequency = Recommended Weekly)

- Turn the unit off and unplug the power cord. Remove all glassware. Remove the top plate.
- Carefully lift the rack out of the bath.
- Siphon off the water in the bath.
  - ⇒ Close siphon bulb vent.
  - ⇒ Place siphon's suction tube in the water bath.
  - ⇒ Place drain tubing in sink.
  - ⇒ Squeeze siphon bulb to start.
- Wipe and rinse the bath walls. Clean the rack by rinsing with water.
- Replace the rack in the water bath. Replace the top plate.
- Place a concentrator tube in *five* positions.
- Pour approximately 1L of distilled water through the empty position.
- Add 15 drops of *Clear Bath*.
- Add more distilled water until the level is as high as the initial solvent level in the sample tube without causing an overflow when all six tubes are in position.
- Plug in the power cord and turn the power on.
- Allow 20-30 minutes for the bath to reach temperature, the air to come out of solution, and for most bubbles to dissipate.
- Perform the Bubble Dislodging Procedure.

Bubble Dislodging Procedure

Insert a clean Zymark tube. Using a pumping motion, raise and lower the tube approximately an inch, several times.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Attachment 11:
Glassware Cleaning Instructions

EX GLASSWARE CLEANING PROCEDURES #1
(SEP FUNNEL & MISC GLASSWARE)

(Includes glassware used for O&G, MBAS, TCLP, funnels, beakers, etc.)

PPE: **Lab coat**
Eye protection
Cut resistant gloves

Note: It is important to segregate heavily contaminated glassware from use until verified clean by the analysis of a method blank. Discard if the condition of the glassware cannot be verified or if the glassware is obviously not salvageable.

1. Rinse all glassware thoroughly with water and discard down the sink drain.
3. Fill dishpan with hot water and add about 60mL FL-70 detergent.
4. Brush vigorously until clean.
5. Rinse three times with tap water.
6. Rinse with sulfuric acid. (OPTIONAL)

   Note: All separatory funnel stopcocks must be removed prior to washing. Stopcocks will not be replaced until separatory funnels have been rinsed with sulfuric acid and rinsed with running water.

   After rinsing with sulfuric acid, rinse glassware thoroughly with tap water. All traces of the acid must be removed from the glassware.

7. Rinse with acetone and place on covered counter or rack to dry.

   Collect acetone rinses in the satellite waste container designated for flammable waste.

EX GLASSWARE CLEANING PROCEDURES #3
(ZYMARK TUBES & CLLE ROUNDBOTTOM FLASKS)

PPE: **Lab coat**
Eye protection
Cut resistant gloves

1. Note the condition of the Zymark tube or receiving flask. If heavily contaminated, do not place into the sink. Keep this glassware separate and contact the Supervisor to determine the best course of action to clean the glassware.

   Note: It is important to segregate heavily contaminated glassware from use until verified clean by the analysis of a method blank. Discard if the condition of the glassware cannot be verified or if the glassware is obviously not salvageable.

2. Rinse each tube or flask thoroughly with water and discard down the sink drain.

3. Fill one side of sink with hot water and add about 60mL of FL-70 detergent.

4. For Zymark tubes, use a small brush to clean the tip of the tube and a larger brush to clean the walls of the tube.

   For receiving flasks, use a brush that will allow you to scrub the inside walls of the flask.

5. Rinse each tube and flask thoroughly a minimum of three times with hot tap water until no traces of soap are present in the tube. It is important to remove all traces of soap at this point.

6. Rinse each tube and flask thoroughly with acetone and place on covered counter or rack to dry.

   Collect acetone rinses in the satellite waste container designated for flammable waste.

Attachment 12:
Extraction Procedures for Tissue Matrices

1.0 Summary of Method

A known weight of a sample is combined with anhydrous, purified sodium sulfate to form a free flowing, sandy mixture. The sample is spiked with surrogate compounds; an analyte-specific solvent is added; and the sample is extracted using an ultrasonic disrupter (i.e., sonicator) for three minutes. The solvent is decanted, and the extraction is repeated two more times. The extract is filtered, solvent exchanged (if required by the analytical procedure), and concentrated to an appropriate final volume. Attachment 6 defines the solvents and extraction conditions for the applicable analytical procedures.

2.0 Equipment and Instrumentation

Ultrasonic Disrupter (sonicator) – Tekmar Model or equivalent with ¾-inch horn-type titanium-tipped sonication probe. The sonicator must be capable of operating in the pulse mode at full power. The sonicator must have a minimum power wattage of 300 watts.

Sonabox – the sonicator must be placed in the sonabox to reduce noise. The sonabox must be placed under a fume hood.

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Stainless Steel Blender or Meat Grinder – heavy-duty type capable of blending tissues.

3.0 Sample Collection, Preservation, Shipment and Storage

Tissue samples are routinely collected in unpreserved glass containers, with the size dependent upon the type of tissue being collected, or wrapped in aluminum foil. Plastic jars or plastic baggies can be used.

Upon receipt, tissue samples must be placed in the freezer at -10° to -20°C, if preparation cannot be completed that day, and must be kept frozen until the time of preparation. A holding time of six months from the date of collection for frozen fish fillets is recommended by Alabama Department of Environmental Management (ADEM) and will be used for all biological tissues.

Once the tissue sample has been thawed, it must be stored at 0-6°C (less than 6°C but not frozen), and preparation must take place within 14 days.

4.0 Extraction Procedure

Tissue samples must be homogenized prior to preparation since organic compounds are generally not evenly distributed throughout biological tissue. The entire sample should be ground into a homogenous consistency. To do this, chop the sample into one inch or smaller chunks and place in the blender or meat grinder. Note: Slightly frozen tissue is easier to work with at this point. Add dry ice to the blender or meat grinder. Blend or grind on appropriate power level until the tissue is thoroughly ground and homogenized. Inspect the tissue for the presence of "chunks" of tissue. Remove the well-homogenized tissue and blend the remaining "chunks" until homogenized. Mix the tissue thoroughly and separate into 10g portions for the required analyses. If the extraction is not to take place soon after homogenization, the homogenized tissue or the individual 10g portions can be frozen. Note, however, that the holding begins with the date of the original thawing process.

To prepare the field samples, weigh the appropriate amount of homogenized sample into a labeled 500mL extraction bottle. A weight of 10g is standard for tissue matrix.

> Note: A larger amount of tissue or solid may be extracted if lower reporting limits (RLs) are required for the project. Refer to project notes or project-specific quality assurance plans (QAPP) for required RLs.

To prepare the method blank and laboratory control samples (LCS/LCSD), weigh an amount of Ottawa sand equivalent to that used for samples into a labeled 500mL extraction bottle. Add the appropriate spiking mix to the LCS/LCSD. Process the method blank and LCS/LCSD in the same manner as the associated field samples.

> Note: If a "control tissue" has been obtained for biological tissue QC, use the control tissue for the method blank and LCS/LCSD, treating them in the same manner as the sample tissues.

To prepare the matrix spike (MS) and matrix spike duplicate (MSD), weigh two additional portions of a sample in the batch into labeled 500mL extraction bottles. Add the appropriate spiking solution to the MS/MSD. Process the MS/MSD in the same manner as the associated field samples.

Add the appropriate surrogate spiking solution (as listed in Attachment 7) to each sample and batch QC item.

> Note: The addition of all surrogate and spiking solutions must be witnessed by another analyst to ensure the correct spiking solution and the proper volume of spiking solution is added to each sample.

Add approximately 60g of granular, purified sodium sulfate to each sample and stir with a glass rod or stainless steel spatula to form a sandy, free-flowing mixture. The sodium sulfate combines with the water in the sample to "dry" the sample (i.e., remove the water). Additional sodium sulfate may be required if the sample is very wet.

Immediately add 70mL of the appropriate extraction solvent as outlined in Attachment 6. Additional solvent may be needed to cover the sample approximately one inch above the solids. Stir the sample to break up any lumps that may have formed.

Place each extraction bottle under the hood near the sonicator.

Check the "tune" of the sonicator using the procedure outlined in Attachment 4. Document the pass or fail of the tune in the Sonicator Logbook.

Place the tip of the sonicator horn in the center of the beaker about ½ inch below the surface of the solvent but above the solid portion. More solvent may be added to bring the level to approximately one inch above the solid layer.

Sonicate each sample for three minutes with the output control knob set at 10, mode switch set to pulse, and percent duty cycle set at 50%. If the sonication is properly performed, the solids and solvent will vigorously mix each time the sonicator pulses.

Decant the extract through the filter funnel containing sodium sulfate and a filter. Collect the extract in a beaker or directly into a labeled, "elongated" Zymark tube. Note: If the sample is very wet and requires a large amount of sodium sulfate and a larger volume of solvent, the extracts are collected in separate Zymark tubes and combined prior to analysis.

Repeat two more times with fresh 70mL portions of extraction solvent, collecting all extracts in the same Zymark tube.

After the third sonication, pour the solids portion of the sample into the filter funnel. Rinse the extraction vessel and solids in the filter funnel with several small aliquots of the extraction solvent, collecting all of the extraction solvent in the Zymark tube.

Note: Clean the sonicator horn between samples by rinsing with methylene chloride.

Note: The extraction steps from weighing to sonication should be carried out quickly to minimize the loss of SVOC through evaporation.

Cover the Zymark tube with aluminum foil, and leave it under the hood until ready to perform the concentration steps as outlined in the body of this SOP.

5.0 Maintenance and Troubleshooting

Refer to Attachment 4 to additional information.

Sonicator Weekly Maintenance

- Turn the instrument off. Disconnect both horns from the back of the instruments.
- Place both horns on a clean padded surface.
- Remove the 1" tip from the horn. Inspect the tip for any pitting and any irregularities. The tip is to be replaced with a new tip if any pitting or irregularities are found. Clean the tip and its threads with a paper towel and methylene chloride.
- Check to ensure that the connection with the horn is tight and free of any foreign matter.
- Carefully reinsert the 1" tip into the horn and tighten the tip. Care must be taken to ensure that the tip is as tight as possible, re-inspect the tip for any irregularities after tightening.
- Perform the above procedure on the second horn.

- After maintenance is complete, re-connect the bottom horn and re-tune the instrument. If the horn will not re-tune, see the department manager.
- Any horn that cannot be re-tuned will be replaced. If the instrument will not tune, a service technician will be called. The instrument will not be used until all tuning criteria are met.

Sonicator Tuning

A tune check must be performed on the sonic dismembrator each day it is used. This tune is to ensure that the sonic dismembrator is operating within the manufacturer's specifications and that the instrument is delivering the minimum wattage output specified by the procedure. Full maintenance is performed as needed by the Department Manager or Supervisor.

- All checks and adjustments to the sonicator must be documented in the maintenance log.
- Unplug the top horn connection on the rear panel of the instrument.
- Check to ensure that the bottom horn connection is as tight as possible.
- Turn the pulser switch off.
- Turn the output to 10.
- Check to ensure that the horn being tuned is free of obstruction.
- Carefully push the tune switch in. At the same time, adjust the tuning knob so that the output meter reads as low as possible. The output meter must read below 20%. If the instrument will not tune to below 20%, a shift supervisor must be informed immediately.

Note: The tuning switch must not be held in for longer than 10 seconds. Holding the switch in for more than 10 seconds may cause the horn being tuned to overheat.

Attachment 13:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Work Instruction
FEX100.03.16.09.0
Page 1 of 1

Determination of Percent Lipids

Summary of Procedure
Percent lipids is determined by weighing a sample prior to and after drying. The ratio of the weights is equivalent to the percent lipids content of the sample. The percent lipids are determined on the tissue sample extract prior to GPC extract cleanup. The final volume of the extract must be adjusted to 10mL prior to the determination of the percent lipids.

Standards
Cod liver oil - commercially available.
Storage: room temperature;
Expiration: Unopened: Manufacturer's expiration date; Opened: Manufacturer's expiration date

Percent Lipids QC Check Standard – add 10g of cod liver oil to a 100mL volumetric flask and dilute to volume with methylene chloride. Each 1.0mL aliquot of this solution contains 100mg of lipid.
Expiration: 12 months from preparation date

Procedure
Clean an aluminum weigh boat. Note: From this point onward the boat must be handled with tongs and protected from particulates. Record the weight of the clean aluminum weigh boat. Transfer 1.0mL of the extract (final volume 10mL) into the aluminum weigh boat. Place the boat in a hood and allow the solvent to evaporate overnight. Reweigh the boat.

Calculate the percent lipids as follows:

$$\%\,Lipids = \frac{(Wr - Wb)}{SV \otimes \frac{Wsample}{FV}} \otimes 100$$

Where:
W_r = weight of residue and aluminum weigh boat (g)
W_b = tare weight of the aluminum weigh boat (g)
W_{sample} = weight of sample extracted (g)
SV = volume of sample added to the aluminum weigh boat
FV = final volume of the extract (mL)

Note: If percent lipids are to be determined on the samples, a method blank and laboratory control standard (LCS) must be prepared and analyzed. The LCS is prepared by adding 1.0mL of the percent Lipids QC Check Standard to 10g of Ottawa sand. The method blank is prepared by adding 1mL of solvent (MeCl2 or Hexane depending on project) to 10g of Ottawa sand. These are extracted in the same manner as the samples. Assuming a sample weight of 10g, the percent lipids in the QC Check sample is 1.0%. The QC check sample must recover with 0.80 to 1.20%.

Attachment 14: Sample Spiking Volumes

Method	Spike Mix	Volume	Surrogate Mix	Volume	Initial Weight	Final Volume
8081/8082	608wkSpike	500uL	PESTwkSurr	500uL	15g	5mL
	1660wk Spike	500uL	---	---	---	---
8015	DIESELWK (DRO)	2mL	DROwkSURR	1mL	15g	1mL
8270	EX8270SPKL1	1mL	8270RTSURR	1mL	15g	1mL
	8270 L1/S9	50uL	---	---	---	---
	EX8270SPKL1	500uL	---	---	---	---
	EX8270SPKL2	500uL	---	---	---	---
8270LL	EX8270SPKL1	100uL	LLBNAwkSurr	1mL	30g	1mL
	8270 L1/S9	5uL	---	---	---	---
	EX8270SPKL1	50uL	---	---	---	---
	EX8270SPKL2	50uL	---	---	---	---
680	680wkSpike	1mL	680wkSurr	1mL	15g	1mL

Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Revised reagents and standards to reflect what is in current use. Section 7
- Changed amount of solvent added to Teflon tube after shaking from 30 mL to 15mL. Section 10.1.7
- Specified that 30mL methylene chloride must be added to tube for EPA method 8015. Section 10.1.7
- Added requirement to gradually increase the psi until dimpling is seen on the top layer during concentration. Section 10.2.5
- Included alternative of utilizing a class A volumetric to determine final volume. Section 10.5
- Changed routine and minimum sample sizes for soils to 15g. Attachment 2
- Changed final volume for chlorinated pesticides and PCBs to 5mL. Attachment 5

IGNITABILITY

(Method: EPA 1030)

Approvals (Signature/Date):

08/28/19

Adriana Geiger — Date
Department Manager

08/28/19

Kimberly Chamberlain — Date
Quality Assurance Manager/Technical Director
Environmental Health & Safety Coordinator

Facility Distribution No. 1 **Distributed To: TALS File System Shares**

1.0 Scope and Application

This SOP gives the procedure for determining the ignitability (i.e., the burn rate) of soil samples, using a ceramic tile tester.

This test is used, in part, to determine whether a sample should be classified as hazardous (as outlined in 40CFR Part 261.21). Ignitability is used in Department of Transportation (DOT) shipping and safety regulations to define flammable and combustible materials.

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Ignitability

In a preliminary test, the soil sample is formed into an unbroken strip 250mm in length. A propane torch is applied to one end of the test material to determine whether combustion will propagate along 200mm of the strip within a specified time period. Materials that propagate burning along the 200mm strip within the specified time period are then subjected to a burning rate test where time is measured over a distance of 100mm and the rate of burning is determined. Samples with a burn rate >2.2mm/sec (or 0.17mm/sec for metals) are considered to have a positive result for ignitability.

Note: The terms "ignitability" and "flashpoint" are often incorrectly used interchangeably. This ignitability procedure determines how quickly a sample will burn over a specified distance. The flashpoint procedure determines the temperature at which a sample combusts, and is obtained via EPA Method 1010A.

2.2 This SOP is based on the following method: EPA 1030 (Ignitability).

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus.

All sample collection containers are single-use disposable containers which limits the potential for contamination.

4.1.1 All tests must be carried out inside a fume hood with the test apparatus situated perpendicular (90°) to the direction of airflow. Airflow parallel to the test apparatus results in non-reproducible burning rates.

The rate of airflow through the fume hood affects the burning rate. Too high an airflow distorts the flame and retards its horizontal propagation. The optimum airflow is in the range of 0.7-1 meter per second (i.e., 140-197 feet/min).

Particle size of test material can affect not only the burning rate, but also the ignition of the material. Therefore, the particle size of the test material should be the same for each test run.

Materials that are moisture sensitive (i.e., readily absorb moisture from air) should be tested as quickly as possible after removal from the sample container.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The analyst must observe the following safety precautions when using the ignitability testing apparatus (i.e., the propane torch):

- do not work alone when performing this test.
- wear a protective face shield (recommended) or glasses, and gloves and a lab coat.
- ensure the hood is functioning properly.
- light the propane torch according to the manufacturer's instructions.
- exercise extreme care when the flame is initially applied to the sample because low flashpoint samples can cause a much higher than expected flash.
- Secure the propane torch cylinders when not in use.

Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). If the sample displays explosivity or extreme flammability, do not conduct this test.

5.2 Primary Materials Used

There are no materials used in this procedure, which have a serious or significant hazard rating.

6.0 Equipment and Supplies

Anemometer – used for ignitability determination to measure air velocity. An optimum air velocity is 0.7-1.0m/sec (i.e., 140-197 feet/min). The anemometer must be calibrated annually by an external service provider. A new anemometer may be purchased annually, if this is more cost effective than recalibration.

Propane Torch - Propane gas burner, capable of maintaining temperature greater than 1000°C.

Ceramic Tile - low-heat conducting, non-combustible, impervious, of approximate dimension of 25cm x 25cm x 2.5cm

Marker - High temperature marker for marking ceramic plates

Powder Train Mold – For molding powdered and granular materials for the burn rate test

Timer – Used to determine burn time. The timer must be verified annually in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

IR Thermometer – Used to obtain temperature of the sample. This thermometer must be verified quarterly in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Thermocouple – Capable of measuring temperatures greater than 1000°C, used to verify flame temperature prior to analysis. The thermocouple must be calibrated annually by an external service provider. A new thermocouple may be purchased annually, if this is more cost effective than recalibration.

6.1 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. Sample collection containers are purchased with Certificate of Analysis attesting to purity.

The routine sample collection containers supplied by the laboratory are: 8oz glass containers

7.0 Reagents and Standards

Reagents and standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it.

7.2 Reagents

Potting Soil - Used as the method blank
Storage: Room temperature
Expiration: 5 years from preparation date

Excelsior – Used as the laboratory control sample (LCS). Comprised of fine pencil shavings.
Storage: Room temperature
Expiration: 5 years from preparation date

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Soil Samples

Samples are routinely collected in 8oz glass containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of analysis. Soil samples are analyzed on an as-is basis (i.e., dry weight correction is not performed).

There is no method-defined holding time for performing the burn rate test to determine ignitability. As such, the EPA 1030 method has been set up in TALS with the holding time condition set to "NONE". The analyst should make every effort to analyze samples within 7 days of collection; however, holding time qualifiers (i.e., H-flags) are not required if this internal holding time is not met. Samples should be tested as soon as possible after

removal from the sample container to avoid drying, absorbing moisture, or loss of volatiles.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC Requirements

9.1.1 Batch QC Frequency

Batch QC must meet the criteria given in Attachment 3 of this SOP.

An analytical batch consists of up to 20 environment samples and the associated quality control items.

- The minimum QC items required for each analytical batch for are: a method blank, a laboratory control sample (LCS), and a sample duplicate.

If there is insufficient sample provided to perform the sample duplicate, the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated to denote this situation.

Note: To ensure consistent results are obtained, the sample duplicate must be performed at the same temperature as the original sample (i.e., within 4°F).

9.1.1 Batch QC Criteria

Refer to Attachment 3 for Batch QC criteria.

9.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Other than removing the samples from the refrigerator, allowing them to come to room temperature, and homogenizing in accordance with SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples,* there are no sample preparation steps associated with this procedure. Record the temperature of the sample (uncorrected and corrected) in the TALS AD batch information screen.

Soil samples are analyzed on an as-is basis (i.e., dry weight correction is not performed). Triplicate determinations of the burning rate must be conducted for samples that burn significantly.

Note: The rate of airflow through the fume hood affects the burning rate. Too high an airflow distorts the flame and retards its horizontal propagation. The optimum airflow is in the range of 0.7-1 meter per second (140-197 feet/min). Adjust the hood sash accordingly to achieve this rate. Record the fume hood air velocity as well as the anemometer ID in the TALS AD batch information screen.

Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). If the sample displays explosivity or extreme flammability, do not conduct this test.

10.1.1 Preliminary Screening Test

10.1.1.1 The preliminary ignitability test is conducted on all waste materials. On a clean, impervious ceramic tile, clearly mark a 250 mm long test path. Make another mark at exactly 200 mm from the start of the sample path.

10.1.1.2 Prepare the test material in its “as received” form by forming an unbroken strip or powder train of sample 250 mm long by 20 mm wide by 10 mm high on the ceramic tile. Use the mold to form the material as in Section 10.1.2.3.

10.1.1.3 Place the ceramic tile with the loaded sample in a fume hood about 20 cm (~8 inches) from the front of the hood and in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test. The air velocity should be approximately 0.7 meters/second. Measure the air velocity with an anemometer.

10.1.1.4 Light the propane torch and adjust the height of the flame (6.5 to 7.5 cm) by adjusting the propane gas and air flows. Measure the temperature of the flame (tip of the flame) by thermocouple. The temperature of the flame must be at least 1000°C. Indicate thermocouple ID and that the flame is of appropriate temperature in the TALS AD batch information screen.

10.1.1.5 Apply the tip of the flame to one end of the sample strip. The test period will depend on the sample matrix as follows:

- If the sample is non-metallic, hold the flame tip on the sample strip until the sample ignites or for a maximum of 2 minutes. If combustion occurs, begin timing with a timer and note whether the combustion propagates up to the 200mm mark within the 2-minute test period. Note the timer ID in the TALS AD batch information screen.

- If the sample is a metal or metal-alloy powder, hold the flame tip on the sample strip until the sample ignites or for a maximum of 5 minutes. If combustion occurs, begin timing with a stop watch and note whether the combustion propagates up to the 200mm mark within the 20 minute test period. Note the timer ID in the TALS AD batch information screen.

10.1.1.6 If the sample does not ignite and propagate combustion either by burning with open flame or by smoldering along 200mm of sample strip within the 2 minute test period (or 20 minute test period for metal powders), the sample is not considered flammable and no further testing is required. If the sample propagates burning of 200mm of the test strip within the 2 minute test period (or 20 minute test period for metals), the material must be evaluated by the burning rate test as outlined in the following section.

10.1.2 Burning Rate Test

10.1.2.1 The preparation of the test sample for the burning rate test will depend on the physical characteristics of the waste. Wastes that exist in a powered or granular state are molded into a powder train mold (See Section 10.1.2.3). Pasty materials are formed into a rope (See Section 10.1.2.4). All tests for the burn rate test are performed on a clean, ceramic plate which has been equilibrated to ambient temperature.

10.1.2.2 On a clean, impervious ceramic tile, clearly mark a 250 mm long test path. Make two additional timing marks at 80 mm and 180 mm from the start of the sample path. The distance between the two marks (100 mm) will be used to calculate the rate of burn in Section 10.1.2. 9.

10.1.2.3 For powdered or granular materials: Tighten the side plates on the mold. Place the mold on the base plate. Pour the material to fill the triangular cross-section of the mold loosely. Drop the unit from a height of 2 cm onto a solid surface three times to settle the powder. Remove the side supports. Lift the mold off the base plate. Place a clean ceramic test plate with the appropriate timing marks (Section 10.1.2.2) face down on top of the mold. Invert the setup and remove the mold.

10.1.2.4 For pasty wastes: Prepare by spreading the waste on a marked ceramic tile (Section 10.1.2.2) in the form of a rope 250 mm in length with a cross-section of 1 cm^2.

10.1.2.5 Place the ceramic tile with the loaded sample prepared in Sections 10.1.2.3 or 10.1.2.4 in a fume hood about 20 cm (~8 inches) from the front of the hood and in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test The air velocity should be approximately 0.7 meters/second. Measure the air velocity with an anemomenter.

10.1.2.6 Light the propane torch and adjust the height of the flame (6.5 to 7.5 cm) by adjusting the gas and air flows. Measure the temperature of the flame (tip of the flame) by a thermocouple. The temperature of the flame must be at least 1000°C.

10.1.2.7 Apply the tip of the flame to one end of the sample strip to ignite the test strip as described in Section 10.1.1.5.

10.1.2.8 When the test strip or powder train has burned up to the 80mm time marker, begin timing the rate of combustion with a stop watch. Stop the timer when the burned strip reaches the 180mm time marker. Record the amount of time (in seconds) required to burn the 100mm test strip.

10.1.2.9 Calculate the rate of burning by dividing the length of the burn test strip (100mm) by the total time (seconds). Results of the burn rate test should be reported in mm/sec.

10.1.2.10 Samples that have a rate of burning of more than 2.2 mm/sec (or burn time of less than 45 seconds for 100mm) are considered to have a positive result for ignitability according to DOT regulations. For metals, this time is 10 minutes or less for 100mm (or a burn rate of more than 0.17mm/sec). The burn rate must be conducted in triplicate for any samples that produce a burn significantly (positive result).

10.1.2.11 QC samples are be handled and analyzed in the same manner as field samples. A method blank is performed using potting soil. An LCS is performed using pencil shavings. A sample duplicate is performed by selecting a sample at random (or by selecting the client-defined sample, if provided) and analyzing in duplicate. To ensure consistent results are obtained, the sample duplicate must be performed at the same temperature as the original sample (i.e., within 4°F).

11.0 Calculations / Data Reduction

Data must be reviewed and evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

If the burn rate test is performed in triplicate, the final result is reported as the average.

Results of the burn rate test must be reported in mm/sec.

Samples that have a burn rate of more than 2.2mm/sec (or burn time of less than 45 seconds for 100mm) are considered to have a positive result for ignitability according to DOT regulations. For metals, this time is 10 minutes or less for 100mm (or a burn rate of more than 0.17mm/sec).

11.1 Calculations

Burn Rate = Length / Time

Where:
Length = length of test strip (i.e., 100mm)
Time = time it takes sample to burn from 80mm mark to 180mm mark on the test strip

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method detection limits as generated in accordance with 40CFR Part 136 Appendix B are not applicable to this procedure.

12.2 Reporting Limit Verifications

Verification of the reporting limit as described in SOP SA-QA-007: *Determination and Verification and Detection and Reporting Limits* is not applicable to this procedure.

12.3 QC Limit Generation, Control Charting, and Trend Analysis

The laboratory has incorporated additional batch QC items above those specified in the reference method (i.e., method blank and/or LCS), to satisfy NELAC requirements. The LCS is considered acceptable if its burn rate is >2.2mm/sec. Since a percent recovery is not reported for the LCS, control charting is not generally performed for this procedure.

The method specifies that precision between sample duplicates should be within 10% RPD.

12.4 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision, analysis of client samples with statistically indistinguishable results when compared to another certified analyst, or by obtaining acceptable PT or single blind QC results. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT or single blind QC study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.5 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Flammable waste samples: Transfer to satellite container for flammable waste. Transfer to hazardous waste section when satellite container is full. Dispose as flammable waste.

14.2 Waste Disposal

Refer to the table below for additional waste disposal information:

Material	Treatment	Destination
Samples, Burn Rate >2.2mm/sec	None	RCRA Waste
Samples, Burn Rate <2.2mm/sec	None	Unconfirmed

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Traceability*

- SOP SA-AN-100: *Laboratory Support Equipment (verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Updates III and IV) USEPA Office of Solid Waste and Emergency Response, Washington, DC.

16.0 Method Modifications and Clarifications

16.1 The laboratory has incorporated additional batch QC items above those specified in the reference method (i.e., method blank and/or LCS), to satisfy NELAC requirements. The method blank is considered acceptable if it does not burn. The LCS is considered acceptable if its burn rate is >2.2mm/sec.

> Note: This procedure generates a positive value for the LCS (as opposed to a percent recovery as is obtained for most methods); therefore, the LCS for this procedure is not generally provided in the final report.

There are no method-defined precision criteria for sample duplicates. The laboratory has adopted 10% RPD as the criteria, as this is consistent with other general chemistry tests.

16.2 There is no method-defined holding time for ignitability. As such, this method has been set up in TALS with the holding time condition set to “NONE”. The analyst should make every effort to analyze samples within 7 days of collection; however, holding time qualifiers (i.e., H-flags) are not required if this internal holding time is not met.

16.3 The reference method gives the optimal anemometer range as 0.7-1.0m/sec. The laboratory’s anemometer gives readings in ft/m. 0.7-1.0m/sec is equivalent to 140-197ft/min. The conversion factor for meters/second to feet/minute is as follows:

$$\frac{0.70\text{m}}{\text{sec}} \times \frac{3.2808\text{ft}}{\text{m}} \times \frac{60\text{sec}}{\text{min}} = 140\text{ft/min}$$

$$\frac{1.0\text{m}}{\text{sec}} \times \frac{3.2808\text{ft}}{\text{m}} \times \frac{60\text{sec}}{\text{min}} = 190\text{ft/min}$$

16.4 EPA Method 1030 requires the flame used to conduct this test to be greater than 1000°C. The laboratory uses a propane/air torch which has an adiabatic temperature of 1967°C.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting

Attachment 1:
SOP Summary

Burn Rate and Ignitability – EPA 1030

In a preliminary test, the soil sample is formed into an unbroken strip 250mm in length. A propane torch is applied to one end of the test material to determine whether combustion will propagate along 200mm of the strip within a specified time period. Materials that propagate burning along the 200mm strip within the specified time period are then subjected to a burning rate test where time is measured over a distance of 100mm and the rate of burning is determined. Samples with a burn rate >2.2mm/sec (or 0.17mm/sec for metals) are considered to have a positive result for ignitability.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Ignitability

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time
Soils	8oz glass	Varies	Varies	None	None	0-6°C[1]	None[2]

[1]Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of analysis.
[2]There is no method-defined holding time for ignitability. As such, the EPA 1030 method have been set up in TALS with the holding time condition set to "NONE". The analyst should make every effort to analyze samples within 7 days of collection; however, holding time qualifiers (i.e., H-flags) are not required if this internal holding time is not met.

Samples should be tested as soon as possible after removal from the sample container to avoid drying, absorbing moisture, or loss of volatiles.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	No more than 20 samples, processed together within a 24-hour time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	No Burn	Do not proceed with sample analysis until a successful method blank is obtained
Lab Control Sample (LCS)	One per batch	Burn Rate > 2.2mm/sec	Do not proceed with sample analysis until a successful LCS is obtained
Lab Control Sample (LCSD)	One per batch, if insufficient sample is provided for Sample Duplicate	Burn Rate > 2.2mm/sec	Do not proceed with sample analysis until a successful LCSD is obtained
Sample Duplicate (SD)	One per batch	RPD ≤ 10%	Barring gross failure, if LCS is acceptable, then qualify data
Initial Demonstration of Capability (IDOC)	Initially; Per analyst, per matrix/method/analyte combination.	Within the method specified accuracy and/or precision criteria, or, if using a single blind PT or QC sample, within vendor's acceptance ranges	Refer to SOP SA-QA-006 Note: Unsupervised work may not begin until successful IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually; Per analyst, per matrix/method/analyte combination	Within the method specified accuracy and/or precision criteria, or, if using a single blind PT or QC sample, within vendor's acceptance ranges	Refer to SOP SA-QA-006

Attachment 4:
Instrument Maintenance and Troubleshooting

There are no analytical instruments associated with this procedure. Rather, the only equipment used in this procedure is ancillary, support equipment (i.e., thermometer and anemometer).

- Thermometers must be calibrated in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*
- The anemometer must be calibrated annually by an external service provider. Note: A new anemometer may be purchased annually, if this is more cost effective than recalibration.

Instrument/Equipment Labeling

Each piece of equipment must be labeled with its name or ID (e.g., MSA, ICP-D, etc.), the date of calibration, and the date the next calibration is due. Additionally, non-operational equipment must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

Maintenance logs have not been established for support equipment as no maintenance is performed. Any malfunctioning equipment is removed from service and replaced.

Attachment 5: Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES - GENERAL LAB

MISCELLANEOUS APPARATUS

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse at least three times with DI water.
4. Air-dry and store inverted in a cabinet or capped.

NOTE:

1. Do not use soap for cleaning COD digestion tubes.
2. Burets may be cleaned by filling with 10% KOH in methanol, allowing to stand for one hour, then rinsing thoroughly with DI water.
3. Evaporating dishes used for TDS determinations are cleaned by rinsing thoroughly with tap water, soaking for several hours in Nochromix, rinsing thoroughly with tap water, and rinsing three times with DI water. The dishes are then oven-dried.

TKN / TOTAL-P DIGESTION TUBES

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse once with Nochromix.
4. Rinse at least three times with DI water.

CYANIDE DISTILLATION APPARATUS

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse with 10% nitric acid, being certain to force at least 10mL through each dispersion tube.
4. Rinse thoroughly with DI water.

ODOR-FREE GLASSWARE

1. Scrub with hot tap water and Liquinox.
2. Rinse with acid cleaning solution.
3. Rinse thoroughly with odor-free water.
4. Glassware used in odor testing should be reserved for that purpose only.

FGE003:08.17.18:2

eurofins | Environment Testing TestAmerica

18.0 Revision History

Summary of Changes:

- Updated SOP signatories to reflect current responsibilities and titles.
- Updated Company name and logo to EurofinsTestAmerica where applicable.

SEMI-VOLATILE COMPOUNDS BY GC/MS

(Methods: EPA 625, EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270D_LL, EPA 8270E_LL, EPA 8270D_LL_PAH, and EPA 8270E_LL_PAH)

Approvals (Signature/Date):

Kimberly Chamberlain 08/15/19

Kimberly Chamberlain Date
Quality Assurance Manager / Technical Director
Environmental Health & Safety Coordinator

Brad Mullis 08/15/19

Brad Mullis Date
Department Manager

1.0 SCOPE AND APPLICATION

This SOP contains the procedures for the determination of extractable semi-volatile organic compounds (SVOC) by gas chromatography/mass spectrometry (GC/MS).

The routine matrices performed by this procedure are waters and soils. Other matrices which may be performed include wipes, leachates, tissues, and wastes.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

2.0 SUMMARY OF METHOD

A measured volume or weight of sample is extracted using continuous liquid-liquid or microwave extraction procedures. The extract is then analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the relative abundance of the characteristic masses determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

This SOP is based on the following methods: EPA 625, EPA 625.1, EPA 8270E, and EPA 8270D.

Note: This SOP contains the procedures for several variations of the SW-846 methods. These variations include:

- EPA 8270D/EPA 8270E = Routine EPA 8270D/EPA 8270E method.
- EPA 8270D_LL/EPA 8270E_LL = Low-level EPA 8270D/EPA 8270E method, providing lower RLs and MDLs than the routine method. This low-level method can incorporate a polynuclear aromatic hydrocarbon (PAH) sublist option to accommodate low-level PAH reporting.
- EPA 8270D_LL_PAH/EPA 8270E_LL_PAH = Low-level EPA 8270D/ EPA 8270E PAH-only method, providing lower RLs and MDLs than the routine method. This method is used for low-level PAH analytes only. This method is used only for DOD QSM V5 samples, and incorporates ortho-terphenyl (OTP) as the surrogate compound. Refer to the DOD QSM document for analytical specifications required in addition to those listed in this SOP.

These three sets of procedures incorporate slightly different standard concentrations, surrogate compounds, instrument configuration, and/or QC evaluation criteria, which are outlined separately in the applicable sections of this SOP.

This SOP also gives the procedures for analyzing and reporting samples using the MS in the Selective Ion Monitoring (SIM) mode.

3.0 DEFINITIONS

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 INTERFERENCES

4.1 Procedural Interferences

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

High purity reagents and solvents are used to help minimize interference problems. Acetone and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The extract may require dilution prior to analysis to reduce or eliminate the interferences.

Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

If there is interference with the primary ion, then secondary ions may be used for quantification. If a secondary ion is used for quantification, the linearity of the secondary ion must be established by meeting the criteria in Section 11.

The basic conditions of the initial extraction may cause hydrolysis and degradation of some target compounds. The degradation may be pronounced in phthalate esters.

Refer to Section 11.1.5 for more information on the chemical relationships of these compounds.

5.0 SAFETY

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Methylene chloride is a carcinogen and an irritant. It causes irritation to the respiratory tract and has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. It may be absorbed through the skin and can cause irritation and pain to the skin and eyes.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1]Exposure limit refers to the OSHA regulatory exposure limit.

6.0 EQUIPMENT AND SUPPLIES

6.1 Equipment and Instrumentation

Gas Chromatograph - Agilent 6890, 7890, or equivalent with compatible autosampler, split/splitless injector, and direct capillary interface.

Mass Spectrometer- Agilent 5973, 5975, or equivalent

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). CHROM software has the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectrum from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software also allows integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as or construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Volumetric Labware

Volumetric Containers must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 12 for Glassware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Gas-Tight Microliter Syringes	Various	QUANTITATIVE	Dilution, Reagent, and Standard Preparation	None (if received with COA)	None (if received with COA)
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Dilution, Reagent, and Standard Preparation	None (Class A)	None (Class A)
Disposable Transfer Pipettes	Various	Qualitative	Sample Transfer	None	None
Autosampler Vials (w/Crimp Cap)	1mL	Qualitative	Instrument Analysis	None	None
Standard Storage Vials (Amber, Glass, w/ Screw Cap)	2mL	Qualitative	Standard Storage	None	None

6.4 Lab Supplies

Column – Rxi-5 SilMS 30ms; 0.25mmx0.25um or equivalent

Injector Liner – 4mm ID quartz or 4mm glass, deactivated

7.0 REAGENTS AND STANDARDS

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability*.

Acetone and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Methylene chloride – pesticide grade, for preparation of analytical standards
Storage: Room temperature
Expiration: Unopened and Opened - Manufacturer's expiration date

Acetone – pesticide grade, for cleaning glassware.
Storage: Room temperature
Expiration: Unopened and Opened - Manufacturer's expiration date

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability.* Certificates of analysis or purity must be received with all purchased standards, and attached to the standard in TALS.

Refer to Attachment 8 for the laboratory's current standards and recipes.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 1L amber glass containers with PTFE-lined lids.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 7 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction. Sample extracts must be allowed to come to room temperature prior to analysis.

Note: In the presence of samples containing residual chlorine, phenol-d6 has been known to react to form chlorinated phenolic compounds that are not detected as the original spiked surrogate. Therefore, aqueous samples must be evaluated for the presence of residual chlorine prior to extraction. The presence or absence of residual chlorine is documented on the preparation sheet completed at extraction.

8.2 Soil Samples

Soil samples are routinely collected in 16oz soil containers with PTFE-lined lids.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction. Sample extracts must be allowed to come to room temperature prior to analysis.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items.

For EPA 625, the laboratory's default QC items performed for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) per 10% of samples extracted, and a matrix spike duplicate (MSD).

For EPA 8270D, EPA 8270D_LL, EPA 8270D_LL_PAH, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, and EPA 625.1 the laboratory's default QC items performed for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

Note: LCS and LCSD are performed if insufficient sample is provided for MS/MSD. If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Refer to applicable preparation SOP listed in Section 10.1 for further information on batch QC.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1.1 Poor Performers

As indicated in SW-846 and/or via assessment of laboratory control sample (LCS) recoveries and control charts, the compounds listed in Attachment 10 are poor performers and/or behave erratically. These compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count for EPA 8270D or EPA 8270E, as outlined in SOP SA-QA-017: *Evaluation of Batch QC Data,* provided they are qualitatively detected.

Note: An NCM must be initiated to denote this situation.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

9.2 Instrument QC

9.2.1 DFTPP Tune

A solution containing DFTPP (decafluorotriphenyl phosphine) must be analyzed at the beginning of each analytical clock as a check on the tune of the instrument. The analytical clock begins with the injection of the DFTPP standard. The analytical clock is defined as 12 hours for EPA 8270D, EPA 8270D_LL, EPA 8270D_LL_PAH, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH and EPA 625.1. The analytical clock is defined as 24 hours for EPA 625.

Meeting the DFTPP tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions including the mass range, scan rate, and multiplier voltage must be identical.

A 1uL aliquot of the 50ng/uL DFTPP/Column Evaluation solution is utilized for EPA 625, EPA 625.1, EPA 8270E and EPA 8270D. A 1uL aliquot of the 5ng/uL DFTPP/Column Evaluation solution is utilized for EPA 8270D_LL, EPA 8270D_LL_PAH, EPA 8270E_LL, and EPA 8270E_LL_PAH.

The DFTPP solution must also contain benzidine, pentachlorophenol, and p,p'-DDT at the following concentrations:

EPA 625, EPA 625.1, EPA 8270E and EPA 8270D – 50ug/mL
EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL and 8270D_LL_PAH – 5ug/mL

Refer to Section 10 for the DFTPP evaluation criteria.

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards for EPA 625 and a minimum of 5 standards for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270D_LL, EPA 8270E_LL, EPA 8270E_LL_PAH and EPA 8270D_LL_PAH. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary to determine if this curve type is prohibited.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

EPA 8270D / EPA 8270E/ EPA 625 / EPA 625.1

Standard Level	Concentration (ug/mL)
1	5
2	10
3	20
4	40
5	60
6	80
7	100
8	200

EPA 8270D_LL/ EPA8270D_LL_PAH/ EPA 8270E_LL/EPA_8270E_LL_PAH

Standard Level	Concentration (ug/mL)
1	0.20
2	1.0
3	2.0
4	5.0
5	10
6	15
7	20

Refer to Attachment 8 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

ICAL evaluation steps and acceptance criteria vary between the methods. Refer to Attachment 1 for a summary of the method-specific requirements.

9.2.2.1 ICAL Readback / % Error Criteria

Each calibration curve must be evaluated to ensure that the quantitation over the entire working range is accurate. This is accomplished via CHROM by reviewing the % Error for each analyte in each calibration level.

The re-calculated concentrations of each analyte should be within 50% for the low point and within 30% for the remaining points.

If these criteria are not met, an alternate curve type that meets criteria should be utilized to ensure a better fit, or another initial calibration curve should be performed.

9.2.3 Instrument Blanks

The instrument must be shown to be free from contamination by the analysis of instrument blanks or method blanks. Instrument blanks are analyzed at the beginning of each clock before analysis of any samples. The instrument blanks should be analyzed following the ICV or CCV. Analysis of a batch method blank may substitute for the analysis of an instrument blank provided analysis is acceptable.

Instrument blanks must be <1/2RL to be acceptable.

9.2.4 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

Refer to Attachment 8 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

ICV evaluation steps and acceptance criteria vary between the methods. Refer to Attachment 1 for a summary of the method-specific requirements.

9.2.5 Continuing Calibration Verification (CCV)

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard. The analytical clock is defined as 12 hours for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH and is defined as 24 hours for EPA 625. The initiation of the clock begins with the injection of the DFTPP.

Refer to Attachment 8 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

An additional, reporting limit CCV (RLCCV) is incorporated into the procedure for EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH. This CCV is used to demonstrate sensitivity at the reporting limit can be obtained. Refer to the applicable CCV section of Attachment 1 for more information on how the RLCCV is utilized.

Note: CCV evaluation steps and acceptance criteria vary between the methods. Refer to Attachment 1 for a summary of the method-specific requirements.

9.2.6 Isomer Resolution Criteria

The GC resolution of structural isomers must be monitored in the ICAL and CCV. In the ICAL, use the calibration level that will be utilized as the CCV.

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. These criteria are important for the identification of benzo(b)fluoranthene and benzo(k)fluoranthene.

Note: The resolution should be monitored utilizing the signal resolution of the extracted ion profiles for the quantitation ion for each analyte. Sufficient GC resolution is needed to identify the individual isomeric peaks by retention time. If the software is able to integrate and define separate peaks for the isomeric pairs, then sufficient resolution has been achieved. If there is sufficient evidence to support the identification of the individual component, then the component is identified, quantified, and reported.

9.2.7 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. The internal standards used are 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12.

Prior to analysis, the internal standards must be added to all standards, samples, and QC items. The concentration of the internal standards must be the same in all calibration samples, field samples, and QC samples. A concentration of 40ug/mL is used for EPA 8270D, EPA 8270E, EPA 625 and EPA 625.1. A concentration of 2.0ug/mL is used for EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL and EPA 8270D_LL_PAH.

9.2.7.1 ICV/CCV ISTD Criteria

The response of the ISTD in the ICV/CCVIS must be within a factor of 2 of the response of the ISTD in the CCV-level standard in the initial calibration sequence, identified as the ICIS. Due to the number of analytes reported in this method, multiple CCVs can be analyzed. The primary CCV is defined as the CCVIS and is used to monitor ISTD response in samples. If the response is outside of the required range, the analysis of the CCVIS must be repeated and any samples associated with the CCVIS must also be re-analyzed. Repeated failure of the ISTD response may require instrument maintenance, re-preparation of standards, and/or re-calibration.

9.2.7.1.2 Sample and Batch QC ISTD Criteria

The response of the ISTD in samples and batch QC items must be within a factor of 2 of the response of the CCVIS in the associated analytical clock. If the response is outside of this range, corrective action must be taken. Corrective actions include re-analysis (for field samples) and/or re-extraction (for batch QC items).

The response of the ISTD in instrument blanks must be within 30% of the response of the previous CCVIS. If outside these limits, consult the Supervisor or Department Manager for approval.

9.2.8 Surrogates

This procedure uses surrogates to evaluate the extraction process.

The surrogates for EPA 625, EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL and EPA 8270D_LL are phenol-d6 (acid), 2-fluorophenol (acid), 2,4,6-tribromophenol (acid), nitrobenzene-d5 (base), 2-fluorobiphenyl (base), and p-terphenyl-d14 (base),

The surrogate for EPA 8270D_LL_PAH and EPA 8270E_LL_PAH is ortho-terphenyl (OTP).

Prior to preparation, the surrogate analytes are added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 100ug/mL is used for EPA 625, EPA 625.1, EPA 8270E and EPA 8270D. A concentration of 10ug/mL is used for EPA 8270D_LL and EPA 8270E_LL. A concentration of 2.0ug/mL is used EPA 8270D_LL_PAH and EPA 8270E_LL_PAH.

The percent recovery of the surrogate in all field samples and QC samples must be within the control limits listed in the Method Limit Groups (MLGs) in TALS.

9.2.8.1 Surrogate Dilution Factor Threshold

Due to the level of dilution required for samples, surrogates may be diluted out. As such, recoveries will be reported as "0D" in dilutions at 1:10 or greater. Control limits will not apply to samples analyzed at dilutions of 1:10 or greater.

An NCM must be initiated to denote this situation.

9.2.8.2 One Acid / One Base Surrogate Exception

The laboratory allows one acid and one base surrogate compound to be outside acceptance limits, in field samples and MS/MSD, provided their recovery is greater than 10%. All surrogate compounds must pass in method blanks and LCS/LCSD.

An NCM must be initiated to denote this situation.

Note: SC DHEC does not permit this allowance. All surrogates must meet criteria for samples originating from South Carolina.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally

permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Preparation

10.1.1 Aqueous Sample Preparation

For continuous liquid-liquid extraction, the sample is transferred to a continuous liquid-liquid extractor, adjusted to a specific pH, as required by the analyte list, and extracted using methylene chloride. The extract is concentrated to a predetermined final volume (typically 1mL) using the Zymark nitrogen blow-down concentrator procedure.

Refer to SOP SA-EX-030: *Liquid Extraction Procedures: Continuous Liquid-Liquid and Separatory Funnel* for specifics on the aqueous and leachate sample preparation process.

10.1.2 Soil Sample Preparation

For microwave extraction, the sample is transferred to a Teflon extraction vessel. A 1:1 acetone/methylene chloride mixture is added to the sample in the vessel. The vessel is placed on the instrument and the sample is extracted at an elevated temperature and pressure. The vessel is cooled to room temperature; the extract is passed through sodium sulfate to remove the water from the sample; and the extract is collected in a concentration tube. The extract is concentrated to a predetermined final volume (typically 1mL) in methylene chloride using the Zymark nitrogen blow-down concentrator procedure.

Refer to SOP SA-EX-040: *Soil Extraction Procedures (Microwave and Sonication)* for specifics on the soil sample preparation process.

10.2 Analysis

10.2.1 Instrument Operating Conditions

Note: The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the MDL.

Example GC Parameters
Injector: 250-280°C
Injector Mode:

- Split – EPA 8270D, EPA 8270E, EPA 625, and EPA 625.1
- Splitless – EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL and EPA 8270D_LL_PAH

Column – Rx-5SilMS 30M x 0.25mm x 0.25um; or equivalent

Carrier Gas Flow: Helium at 0.5-1.0mL/min (column)
Mass spectrometer interface: 280-300°C
Mass spectrometer source temperature: 230°C
Mass spectrometer quad temp: 150C
Mass range: 35-500amu, with a minimum scan time of 1.0 scan per second

Temperature Program:

Initial Temp:	55°C
Initial Hold:	1.5 min
Program Rate:	30°C/min to 190°C, 32°C/min to 320°C
Final Temp:	320°C (hold until elution time of Benzo(ghi)perylene or last eluting analyte)
Injected Volume:	1-2uL per column
Inlet Purge Time:	0.8 minutes

The injection volume must be the same for all standards and sample extracts.

10.2.2 DFTPP Analysis and Evaluation

The DFTPP standard must be analyzed at the beginning of each clock. The analytical clock is defined as 12 hours for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH, and is defined as 24 hours for EPA 625. The initiation of the clock begins with the injection of the DFTPP.

10.2.2.1 DFTPP Spectrum Criteria

The spectrum of the DFTPP must meet the criteria for each method listed in Attachment 5. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. CHROM software is programmed to perform this subtraction during processing of the DFTPP. The time utilized for the background subtraction is displayed on the tune report generated in CHROM.

Background subtraction is designed to eliminate column bleed and instrument background ions.

Note: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.2.2 Peak Tailing Factor Criteria

The analysis of benzidine and pentachlorophenol serves as a check on the system performance. The tailing factor for pentachlorophenol and benzidine is calculated as outlined in Attachment 6 and must meet the following criteria:

EPA 625:
pentachlorophenol <5
benzidine <3

EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH:
pentachlorophenol <2
benzidine <2

If the criteria above are not met, perform injector port and column maintenance and reanalyze the DFTPP standard.

10.2.2.3 DDT Breakdown Criteria

DDT breakdown must be evaluated for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH.

Percent breakdown is calculated using the areas from the total ion chromatogram using following equation:

$$\%DDTBreakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\,100$$

The percent breakdown of p,p'-DDT must not exceed 20% for the methods cited above.

There is no DDT percent breakdown requirement for EPA 625.

10.2.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.4, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5 and Attachment 1.

10.2.4 Sample Analysis

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The clock time starts with the injection of the DFTPP. The analysis standards, QC and samples may continue until the clock time expires. Sample analysis must initiate within the clock time defined by the applicable method. For EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH, the clock time is 12 hours. For EPA 625, the clock time is 24 hours.

10.2.5 Example Analytical Sequence

Description	Comments
DFTPP	Clock time begins.
Initial Calibrations	
ICVs	
Instrument Blank	
Samples & Batch QC Items	EPA 625: Clock time ends at 24 hours from DFTPP EPA 625.1 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH: Clock time ends at 12 hours from DFTPP
DFTPP	Clock time begins.
CCVs	
RL CCV	EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH only
Instrument Blank	
Samples & Batch QC Items	EPA 625: Clock time ends at 24 hours from DFTPP EPA 625.1 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH: Clock time ends at 12 hours from DFTPP

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data*. This SOP includes specific information regarding the evaluation

of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-002: *Data Generation and Review*.

11.1.1 Target Analyte Identification

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the standard spectral library. CHROM utilizes the NIST/NBS library for reference spectra..

The following criteria must be met in order to positively identify a compound:

1) Elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound. RRT is calculated as follows:

$$RRT = \frac{retention time of the target compound}{retention time of the associated internal standard}$$

2) Correspondence of the target compound spectrum and the standard component mass spectrum.

   All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

   The relative intensities of the ions present in the sample component spectrum should agree within ±30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum. The intensities of the ions in the standard reference spectrum are updated utilizing the continuing calibration standard. If the relative intensities are outside 30% in sample, the data will be flagged with an R by CHROM.

3) The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

4) Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC

resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

If the above criteria are not met, the analyst should seek help from a senior analyst or Supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported. If there is not sufficient evidence to support identification of the component, then an NCM must be generated to note that the isomers should be identified as the isomeric pair.

5) Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

   Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2 Evaluation of Tentatively Identified Compounds (TICs)

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Note: TICs can not be provided from a SIM analysis.

Refer to SOP SA-QA-008: *Evaluation of Chromatographic Data* for the laboratory's TIC procedures.

11.1.3 Dilutions

It is acceptable to report results from a single analysis as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. If the laboratory does not have historical data and acquires data for a sample with passing internal standards and surrogates but one or more analytes exceed the calibration range, the laboratory will report the over-range (i.e., E-values) with an "Acceptable" TALS status and re-analyze the extract only for the compounds that exceeded the calibration range. The secondary analysis will be reported as a "DL" in TALS, and surrogates will not be reported from this analysis. This is known as the "Condensed Dilution Format". When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require the laboratory to provide lower detection limits, a general guide is to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample is re-analyzed at a dilution factor of 1/5 to provide lower reporting limits for the remaining compounds.

Note: If samples are analyzed at a dilution factor of 1:10 or greater, report the surrogate result as 0%D, to denote a dilution above the Dilution Threshold. Control limits will not apply to samples analyzed at dilutions of 1:10 or greater.

Dilute samples according to the following table:

Dilution Factor	Extract Aliquot (uL)	ISTD Aliquot (uL)		Final Volume* (mL)
		Routine	Low Level	
2	500	10	5	1.0
5	200	16	8	1.0
10	100	18	9	1.0
20	50	19	9.5	1.0
50	20	20	10	1.0
100	10	20	10	1.0

*Final solvent = methylene chloride

11.1.4 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Notes and/or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

11.1.5 Chemical Relationships

The analyst must be aware of the following chemical relationships:

- Several analytes reported as "total" are summed from the individual components. For example, Total Cresols are summed as follows:

  Total cresols = m/p-cresols + o-cresol

  Note that o-cresol and m/p-cresol are also reported as individual analytes.

- Several analytes are identified as summary analytes. These analytes break into multiple peaks upon analysis. Each component's response must be utilized to calculate a response for the summary analyte. The following are examples of summary analytes and their associated components.

  Aramite, Total = aramite peak 1 + aramite peak 2
  Diallate, Total = diallate peak 1 + diallate peak 2
  Isosafrole, Total = Isosafrole peak 1 and Isosafrole peak 2

- Chromatographically unresolved isomers are reported together since isomers cannot be resolved by differences in mass. For example, m-cresol and p-cresol are reported as m&p-cresols.

- It is important that closely eluting isomers be resolved chromatographically so that the analyte can be properly identified. The most critical separation is benzo(b)fluoranthene and benzo(k)fluoranthene.

- Acid/Base Compounds

  Basic Compounds: In aqueous samples, several target compounds are soluble in methylene chloride only at basic pH (>12). These compounds form methylene chloride insoluble salts at acidic pH and remain in the aqueous phase. When the pH is adjusted to basic, the ionic compound reverts to its original form and can be extracted out of aqueous solution. Examples of these compounds include pyridine (TCLP, AP9), benzidine (625PP), and a,a-dimethylphenethylamine (AP9). If acid-only extraction is performed, basic compounds will not be extracted and detected.

  Neutral Compounds: In aqueous samples, neutral compounds can extract into methylene chloride at either acidic pH (<2) or at basic pH (>12). That is, these compounds do not convert to salts or ionic forms at either acidic pH. If the acid pH is performed first, the compounds partition into the methylene chloride and would be detected in the acid fraction; if the basic pH extraction is performed first, these compounds partition into the methylene chloride and would be detected in the base fraction. Exceptions include the phthalate esters (e.g., dimethyl phthalate, diethyl phthalate) and other esters which may be irreversibly converted (hydrolyzed) to salts if subjected to the basic pH extraction first.

  Acid Compounds: In aqueous samples, the acidic compounds can be extracted into methylene chloride at acid pH (pH<2). The acidic compounds are the phenols and benzoic acid. At basic pH, the phenols forms water soluble salts which are not soluble in methylene chloride. When the pH is adjusted to <2 the salt is converted back to the phenol or acid form. Some phenols (2,4-dimethyl phenol and the cresols) do not completely ionize at basic pH and may be present in both the acid and base fractions of a dual pH extraction.

- Single pH (<2) Extractions

  Single pH extractions are performed at pH<2 and include the compounds that are soluble in methylene chloride at acidic pH. The primary application of the single pH extraction is for the routine compound list, which includes most of the compounds that are monitored for and are detected in field samples. The advantages of the single pH extraction are a shorter extraction time and efficient extraction of all phenolic compounds and compounds subject to hydrolysis under basic pH conditions. The drawback is that basic compounds are not extracted under single pH conditions.

- Dual pH Extractions

  Dual pH extractions may be performed with the basic pH extraction first followed by the acidic pH or the acidic pH extraction may be performed first followed by the basic pH extraction. The table below summarizes some of the positive and negative aspects of dual pH extractions.

Extraction	Pros	Cons
Acidic pH first followed by basic pH Examples: TCLP, Appendix IX	No hydrolysis of phthalate esters	Acid and base/neutral surrogates are both extracted into solvent at acidic pH. There is no surrogate to determine whether the sample pH was adjusted to basic pH, to determine the extraction efficiency of the basic pH extraction
Basic pH first followed by acidic pH Example: EPA 625 PP	Acid compounds (phenols) and base/neutral compounds can be separated into two extracts. The partitioning of the target analytes into separate extracts can sometimes help to minimize the effect of the sample matrix on the target compounds.	Some compounds may be partitioned into both the acid and base/neutral extracts. Examples include 2,4-dimethylphenol and the cresol compounds.
		Phthalate esters are converted (hydrolyzed) to salts under basic conditions which causes irreversible loss of the compounds. Dimethyl phthalate and diethyl phthalate are the most effected compounds.
		Some phenolic compounds may have reduced recoveries

- Compounds with similar structures and properties are often found together in a sample or in the samples from the same project or site. That is, when one of that type of compound is detected, the analyst should be looking for other compounds of that type. For example, when one PAH compound (e.g., naphthalene, phenanthrene, benzo(a)pyrene, etc.) is detected, the analyst should expect other PAH compounds to be present. When chlorinated benzenes (e.g., 1,2-DCB, 1,3-DCB, 1,4-DCB) are present, the analyst should be aware that other chlorinated benzenes may be present. When pentachlorophenol is detected, the analyst should also look for tetrachlorophenols and trichlorophenols.

11.1.6 Confirmation of GC Analyses

If required, GC/MS can be used to confirm some GC-only analytes. Based on the response of the standard, concentrate the SVOC GC extract to an appropriate final volume.

Note for SVOC: The pesticide extracts cannot be used to confirm the presence of herbicides since the extract has not been properly prepared.

- Add an appropriate volume of internal standard to the extract or sample to give the same concentration as in the calibration standard. Analyze the extract under the same conditions as the standard.

- Compare the retention time of the sample to the retention time of the standard.

If a peak is detected at the retention time of the target compound containing the selected masses in the same ratio as the standard, the peak is confirmed as the target

compound and the concentration is calculated. The relative intensities of the ions in the sample should agree within ±20% of the intensities of the ions in the standard.

If a peak is not present at the appropriate retention time or if the ratios of the ions are not the same as the standard, the analyte is not confirmed.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent (applicable to soil samples, only)

Notes:

- **Note: This calculation assumes all applicable unit correction factors are applied.**
- The calculation used for water samples is the same as above with the exception that there is no % Solids factor applied.
- All dry weight corrections are performed automatically in TALS.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated

(statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

12.4.1 EPA 625 and EPA 625.1

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

12.4.2 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the SOP criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures.*

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the EHSM.
- Flammable waste (acetone from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.
- Methylene chloride extracts – Dispose according to characterization on sample disposal sheets. If non-hazardous, transfer extract to chlorinated waste container. If hazardous, transfer to hazardous waste department for storage.
- Methylene chloride used to rinse glassware, etc. – Transfer to chlorinated waste container.
- Excess aqueous samples – Dispose according to characterization on sample disposal sheets. If non-hazardous, dispose down drain/sewer. If hazardous, transfer to hazardous waste department for storage.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.
- Excess oil samples – Transfer to waste department for storage/disposal

14.2 Waste Disposal

Material	Treatment	Destination
Samples (Non-Hazardous Water)	Neutralization if applicable	Sink
Samples (Hazardous Water)	Neutralization if applicable	RCRA Hazardous Water
Samples (Unconfirmed Soils)	Characterize for toxicity, ignitability, corrosivity, and reactivity	Non-hazardous: landfill Hazardous: RCRA Hazardous Soils
Samples (Hazardous Soils)	None	RCRA Soils
Autosampler Vials	None	Hexane Vials
Excess Standards (Non-PCB)	None	Hexane Vials
Excess Standards (PCB)	None	PCB Waste

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-EX-015: *Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)*
- SOP SA-EX-030: *Liquid Extraction Procedures: Continuous Liquid-Liquid and Separatory Funnel*

- SOP SA-EX-040: *soil Extraction Procedures (Microwave and Sonication)*
- SOP SA-EX-042: *Waste Dilution Extraction*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method 8000B: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Method 8270D: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Method 8270E: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- EPA Method 625*: Base/Neutrals and Acids.* 40 CFR Part 136, Appendix A, July 1, 1995.
- EPA Method 625.1*: Base/Neutrals and Acids by GC/MS.* 40 CFR Part 136, Appendix A, December 2016.

16.0 Method Modifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine aqueous RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, and tissues unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures

Waste (Oil) Samples:
Waste (oil) samples are collected in 8oz soil containers with PTFE-lined lids. Waste (oil) samples must be iced at the time of collection and maintained at 0-6°C until the time of

preparation. Samples must be prepared within 14 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

Wipe Samples:
Wipe samples are routinely collected in 40mL VOA vials. Wipe samples must be iced at the time of collection and maintained at 0-6°C until time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

Tissue Samples:
Tissue samples are routinely collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, samples must be placed in the freezer at <-10°C if extraction/digestion cannot be completed that day. Samples must be prepared within 14 days of defrosting. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

TCLP/SPLP Leachate Samples
Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a 1L glass container. TCLP/SPLP leachates must be stored at 0-6°C until the time of preparation. The leachate sample must be prepared within 7 days of completion of the TCLP/SPLP extraction. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

16.1.2 Preparation and Analytical Procedures

Wipe, waste, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-EX-040. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-EX-030. Refer to the applicable preparation SOPs for more information.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

16.2 Other Considerations

16.2.1 SW-846 allows alternate criteria to be used for DFTPP evaluation. As such the laboratory has incorporated criteria from the following method:

- EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH tune criteria are taken from EPA 625.

16.2.2 The laboratory allows one acid and one base surrogate compound to be outside acceptance limits, in field samples and MS/MSD, provided their recovery is greater than 10%. All surrogate compounds must pass in method blanks and LCS/LCSD.

16.2.3 EPA Method 625 does not require the analysis of an ICV. NELAC requires an ICV; however, it does not list specific criteria. The laboratory has adopted the default criteria listed in Section 9.2.3 for ICVs for these methods.

Additionally, EPA Method 8270D lists recommended criteria of 30%D for the ICV and acknowledges that alternative acceptance limits may be appropriate based on project-specific data quality objectives. The laboratory defaults to the criteria outlined in this SOP; however, more stringent, project-specific requirements can be accommodated upon client request.

16.2.4 The laboratory has defined the analytes listed in Attachment 10 as poor or erratic performers and allows for exceptions to the ICV, CCV, LCS, MS/MSD, and Sporadic Marginal Exceedance criteria for these analytes by EPA method 8270D as outlined in this SOP.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

16.2.6 The reference methods do not require the analysis of an instrument blank; however, the laboratory routinely analyzes instrument blanks items and has adopted in-house criteria as outlined in this SOP.

16.2.8 EPA Method 8270D does not place a cap on an individual analyte's %D or %RSD when evaluating the CCV. The laboratory has adopted more stringent in-house requirements as outlined in this SOP.

16.2.9 Due to maintenance procedures, the laboratory allows internal standard retention times in continuing calibration checks (CCVs) for EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH to vary by more than 30 seconds when compared to the retention time of the internal standards in the most recent initial calibration. A component of column maintenance is to remove a portion of the front of the column to eliminate reactive spots caused by injection of field samples. As the column is shortened, the retention times of the internal standards are also shortened. If peak resolution and sensitivity are maintained by meeting the CCV criteria, calibration of the analytical system is not required.

Note: This modification is used for CCVs only. The laboratory has made no similar modification to sample ISTD evaluation.

16.2.10 EPA Method 8270D and 8270E indicate indeno(1,2,3)pyrene and di-n-octylphthalate will be quantitated using ISTD Perylene-12. The laboratory uses Chrysene-d12 as this compound is more stable.

16.2.11 EPA Method 8270D states the method blank must be less than the MDL, 5% of the regulatory limit, or 5% of the sample result, whichever is greater. The laboratory's criteria for the method blank is <1/2RL. More stringent, project-specific requirements can be accommodated upon client request.

16.2.12 EPA Method 8270D specifies a reporting limit standard quantitation criteria of 30% when utilizing a linear fit for the ICAL. With Technical Management approval, the laboratory allows analysis to proceed for analytes with recovery outside 30% of the expected value, provided reasonable sensitivity is achieved.

16.2.13 EPA 8270D_LL_PAH and EPA 8270E_LL_PAH is performed for DOD samples only. This procedure incorporates the ortho-terphenyl surrogate included in the EPA 8310B reference method (i.e., PAHs via HPLC analysis).

16.2.14 The routine calibration standards have been split into two groups with a standard prepared at 160ug/mL for each of the two groups. Each day before starting the instruments, the LIST1A-160 and LIST1B-160 are combined in equal portions to allow analysis of a single standard that contains all the List 1 compounds at an 80ug/mL concentration. The same process is used with the 20ug/mL standards to produce a 10ug/mL reporting limit standard. The 160ug/mL standards are not used for the initial calibration.

16.2.15 Method 8270D states in section 11.6.1 The reference mass spectrum must be generated by the laboratory using the conditions of this method. The reference spectra utilized by the laboratory in CHROM for peak identification is generated by the NIST library. The mass ratios for the ions are updated by the continuing calibration analysis.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Preventative Maintenance and Troubleshooting
Attachment 5: DFTPP Criteria
Attachment 6: Example Tailing Factor Calculation
Attachment 7: Target Compound Information: Quant ions and ISTDs
Attachment 8: Standard Preparation Postings
Attachment 9: Procedures for SIM Analyses
Attachment 10: Poor Responder Information
Attachment 11: EPA 8270D Minimum RRF Table
Attachment 12: Glassware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation Summary
Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP
Water	SA-EX-030
Soil	SA-EX-040

Aqueous Sample Preparation:
For continuous liquid-liquid extraction, the sample is adjusted to a specific pH, as required by the analyte list, transferred to a continuous liquid-liquid extractor, and extracted using methylene chloride. The extract is concentrated to a predetermined final volume (typically 1mL) using the Zymark nitrogen blow-down concentrator procedure.

Soil Sample Preparation:
For microwave extraction, the sample is transferred to a Teflon extraction vessel. A 1:1 acetone/methylene chloride mixture is added to the sample in the vessel. The vessel is placed on the instrument and the sample extracted at an elevated temperature and pressure. The vessel is cooled to room temperature, the extract is passed through sodium sulfate to remove the water from the sample, and the extract is collected in a concentration tube. The extract is concentrated to a specified final volume (typically 1mL) in methylene chloride using the Zymark nitrogen blow-down concentrator procedure.

Sample Analysis Summary
The extract is analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the relative abundance of the characteristic masses determined from NIST library. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Example Analytical Sequence

Description	Comments
DFTPP	Clock time begins.
Initial Calibrations	
ICVs	
Instrument Blank	
Samples & Batch QC Items	EPA 625: Clock time ends at 24 hours from DFTPP EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH: Clock time ends at 12 hours from DFTPP
DFTPP	Clock time begins.
CCVs	
RL CCV	EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH only
Instrument Blank	
Samples & Batch QC Items	EPA 625: Clock time ends at 24 hours from DFTPP EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH: Clock time ends at 12 hours from DFTPP

Initial and Continuing Calibration Requirements Summary

1.0 EPA 625

1.1 Initial Calibration (ICAL)

1.1.1 Linearity Criteria

All compounds must be evaluated for linearity. The following steps outline the evaluation:

1. The relative standard deviation (%RSD) of the calibration standards must be ≤35% for the initial calibration curve to be acceptable.
2. A minimum of 3 points is required. The lowest calibration point must at or lower than the reporting limit (RL).
3. If the calibration is utilizing enough points to establish a linear or quadratic fit, then a regression curve may be used. Utilize a regression curve in accordance with SOP SA-QA-016. The criteria for the regression coefficient is r^2 ≥0.990. If the %RSD is less than 35%, a regression curve may still be used if it provides a better calibration model over the calibration range than the average RF.

1.2 Second Source Initial Calibration Verification (ICV)

1.2.1 ICV Criteria

The initial calibration verification (ICV) is acceptable if the %D is ≤35% for each analyte.

If the %D criteria are not met, re-calibration is required.

1.3 Continuing Calibration

1.3.1 %D Criteria

All analytes in the CCV must be ≤20% of the true value to be acceptable.

2.0 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH

2.1 Initial Calibration (ICAL)

2.1.1 Minimum Relative Response Factor (RRF) Criteria

The minimum RRF criteria and control analytes are listed in Attachment 12. If the minimum RRF criteria for each compound in each level of the ICAL are not met, analysis of an RLCCV is required in each clock. An NCM is required to denote this situation.

2.1.2 Linearity Criteria

All compounds must be evaluated for linearity. The following steps outline the evaluation:

1. Determine the %RSD for each compound. The %RSD of each compound must be ≤20% to be acceptable.

2. If the %RSD criteria are not met, the next option is to utilize a linear regression curve for that compound, in accordance with SOP SA-QA-016. For a linear regression curve to be acceptable, the regression coefficient (r^2) must be ≥0.990.

   Note: When a linear regression curve is used, the reporting limit standard must be re-quantitated as a sample and recover within 30% of the expected value. If these criteria are not met, the initial calibration curve may be evaluated utilizing a quadratic curve or the compound may be quantitated using the average response factor as outlined in Item #3, below.

   For a quadratic curve to be acceptable, the regression coefficient (r^2) must be ≥0.990.

3. If the r^2 criteria are not met, the only remaining option is to utilize the average response factor and report that compound as estimated. In this situation, an NCM must be initiated to describe the issue.

   Note: SW-846 does not put a cap on an individual analyte's %RSD; however, the laboratory has adopted the requirement that no individual analyte can exceed 50% RSD. Therefore, if any analyte's %RSD is >50 %, then re-calibration is required.

4. If more than 10% of the analytes do not meet both the %RSD criteria or meet the r^2 criteria, then recalibration is required.

   Note: Several standard mixes are utilized to perform an initial calibration for the full list of target analytes (e.g., TCL, Appendix IX, etc). Each of these mixes constitutes its own initial calibration. Therefore, when evaluating the numbers of acceptable analytes, each mix will be evaluated separately. Re-calibration need only involve the affected mixes.

5. For any analyte associated with a calibration that does not meet RSD or 0.990 or minimum response factor, an RLCCV must be analyzed with each subsequent clock.

   The RLCCV is a CCV analyzed at the reporting limit for the affected analyte. The criterion for the RLCCV is detection only; however, the standard qualitative identification criteria in the method must be met. An NCM must be initiated to denote this situation. Any positive results should be noted as estimated. Non-detect results for analyte may be reported without flagging if there has been an analysis of the RLCCV.

2.2 Second Source Initial Calibration Verification (ICV)

The initial calibration verification (ICV) is acceptable if the %D of each analyte in the ICV

is less than or equal 30%.

Note: Analysis may proceed for analytes >30%D provided the %D is <50%; however, any detections for these analytes must be reported as estimated and addressed via an NCM.

2.2.1 ICV Poor Performers

Refer to Attachment 10 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >30% but must be <50%D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

2.3 Continuing Calibration Verification (CCV)

2.3.1 Minimum RRF Criteria

The minimum RRF for each compound is listed in Attachment 12.

If the minimum RRF criteria are not met, analysis may proceed with the analysis of the RLCCV to demonstrate sensitivity. An NCM must be initiated for any detections associated with a minimum RRF failure.

Note: The RRF criteria for certain analytes have been modified for the EPA 8270D_LL method.

2.3.2 %D Criteria

The CCV %D criteria are ≤20%.

The reference method makes the exception, however, such that due to the large numbers of analytes in each CCV, some analytes may not meet these criteria.

- If more than 20% of the analytes in a CCV exceed the %D criteria, the CCV is unacceptable, and re-analysis is required.

- If less than 20% of the analytes in a CCV exceed the %D criteria, analyze an RLCCV. Analytes that do not meet CCV %D criteria may be reported if the affected analytes are qualitatively identified in the RL CCV.
  - If the affected analyte is not detected in the associated client sample, the result is reported without qualification.
  - If the affected analyte is detected in the associated client sample, the result must be reported as estimated.
  - Note: An NCM must be initiated to denote this situation.

Note: SW-846 does not put a cap on an individual analyte's %D; however, the laboratory has the requirement that no individual analyte can exceed 50%D. Therefore, if any analyte's %D is >50%, then corrective action is required. Corrective actions include instrument maintenance, re-injection, and/or re-calibration.

Note: The routine calibration standards have been split into two groups with a standard prepared at 160ug/mL for each of the two groups. Each day before starting the instruments, the LIST1A-160 and LIST1B-160 are combined in equal portions to allow analysis of a single continuing calibration verification standard that contains all the List 1 compounds at an 80ug/mL concentration. The same process is used with the 20ug/mL standards to produce a 10ug/mL reporting limit standard. The 160ug/mL standards are not used for the initial calibration.

CCV Poor Performers

Refer to Attachment 10 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation. The poor performers must be counted in the total number of allowances within a CCV analysis.

3.0 **EPA 625.1**

3.1 Initial Calibration (ICAL)

3.1.1 Linearity Criteria

All compounds must be evaluated for linearity. The following steps outline the evaluation:

1. The relative standard deviation (%RSD) of the calibration standards must be ≤35% for the initial calibration curve to be acceptable.
2. A minimum of 5 points are required. The lowest calibration point must at or lower than the reporting limit (RL).
3. If the calibration is utilizing enough points to establish a linear or quadratic fit, then a regression curve may be used. Utilize a regression curve in accordance with SOP SA-QA-016. The criteria for the regression coefficient is r^2 ≥0.990. If the %RSD is less than 35%, a regression curve may still be used if it provides a better calibration model over the calibration range than the average RF.

3.2 Second Source Initial Calibration Verification (ICV)

3.2.1 ICV Criteria

The initial calibration verification (ICV) is acceptable if the recovery meets the method defined limits established in EPA 625.1.

If the %D criteria are not met, re-calibration is required.

3.3 Continuing Calibration

3.3.1 %D Criteria

All analytes in the CCV must meet the method defined recovery limits established in EPA 625.1.

If the %D criteria are not met, then corrective action is required. Corrective actions include instrument maintenance, re-injection, and/or re-calibration.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	1L amber glass	1L	500mL	None	0-6 °C[1]	None	7 days to extract 40 days to analyze
Soil	16oz glass soil jar	30g	15g	None	0-6°C[1]	None	14 days to extract 40 days to analyze

[1]Samples must be maintained at 0-6 °C, with no frozen samples.

Attachment 3:
QC Summary

QC Item	Method	Frequency	Criteria	Corrective Action
Clock Time	EPA 625	24 hours	Clock time starts with the injection of the DFTPP. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new DFTPP.	Not applicable
	All Other Methods	12 Hours		
Tune/Column Evaluation Standard (DFTPP) - Spectrum Criteria	All Methods	At beginning of each clock	Spectrum Criteria: Refer to Attachment 6.	- Perform instrument maintenance - Re-tune.
Tune/Column Evaluation Standard (DFTPP) - Tailing Factor Criteria	EPA 625	At beginning of each clock	Pentachlorophenol <5 Benzidine <3	- Perform instrument maintenance - Re-tune.
	All Other Methods		Pentachlorophenol <2 Benzidine <2	
Tune/Column Evaluation Standard (DFTPP) - Breakdown Criteria	All Other Methods	At beginning of each clock	<20%	- Perform instrument maintenance - Re-tune.
	EPA 625		None	

QC Item	Method	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	EPA 625	Upon instrument set-up, and after unsuccessful CCV	3-point Minimum %RSD <35%	-Re-analyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Replace column and/or clean source, and reanalyze standards
	EPA 625.1		5-point Minimum %RSD <35%	
	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH		5-point Minimum RRF per Attachment 11. %RSD ≤20%. If %RSD > 20%, use curve fit w/ r^2 ≥0.990; If linear fit, RL Level: 30% of true; If r^2 <0.990, use %RSD (allowed for <10% of total # analytes; no analyte >50% RSD). %RSE <50% for low point; <30% for all other points.	
	SIM		3-point Minimum r^2 ≥0.990	
Initial Calibration Verification	EPA 625	After each ICAL	%D <35%	-Reanalyze standard -Prepare new standard and

(ICV) - Second Source	EPA 625.1		Within method-defined limits (in TALS MLG)	reanalyze -Recalibrate
	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH		%D <30% Poor performers %D <50% as per Attachment 10. An NCM is required to denote analytes outside criteria.	

QC Item	Method	Frequency	Criteria	Corrective Action
Continuing Calibration Verification (CCV)	EPA 625	Per clock (Analyze after DFTPP)	%D ≤20%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate Note 1: If <20% total # analytes >20%D, evaluate RL CCV. RL CCV must be qualitatively identified. - If client sample is ND for affected analyte, report unqualified result. - If client sample has detection for affected analyte, report result as estimated.
	EPA 625.1		Within method-defined limits (in TALS MLG)	
	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH		RRF per Attachment 11. %D ≤20%. Poor performers per Attachment 10. See Note 1.	
Tune/Column Evaluation Standard (DFTPP) - Spectrum Criteria	All Methods	At beginning of each clock	Spectrum Criteria: Refer to Attachment 6.	- Perform instrument maintenance - Re-tune.
Reporting Limit Continuing Calibration Verification (RL CCV)	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH	Per clock, if needed. (Analyze after CCV) When RRF criteria is not met in ICAL, or CCV criteria not met.	Affected analytes must be qualitatively identified.	- Perform instrument maintenance - Reanalyze affected samples. - Recalibrate

QC Item	Method	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	All Methods	Spiked in all CCVIS, samples, and batch QC items	CCVIS: Area within 50% to +200% of corresponding level in the ICAL. Samples & batch QC items: - Area within 50% to +200% of CCVIS. - RT within +/-30 seconds from previous CCVIS.	Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	All Methods	Spiked (during extraction procedure) in all samples and batch QC items.	Within MLG limits 1 Acid / 1 Base Allowance: samples and MS/MSD, only, with %R >10% Surrogate Threshold Dilution Factor = 10	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Extraction Batch Definition	All Methods	Extracted together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	All Methods	One per extraction batch	<1/2RL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	All Methods	One per extraction batch	Within TALS MLG Limits	Refer to SOP SA-QA-017

QC Item	Method	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	All Methods	Spiked in all CCVIS, samples, and batch QC items	CCVIS: Area within 50% to +200% of corresponding level in the ICAL. Samples & batch QC items: - Area within 50% to +200% of CCVIS. - RT within +/-30 seconds from previous CCVIS.	Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	All Methods	Spiked (during extraction procedure) in all samples and batch QC items.	Within MLG limits 1 Acid / 1 Base Allowance: samples and MS/MSD, only, with %R >10% Surrogate Threshold Dilution Factor = 10	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Laboratory Control Sample Duplicate (LCSD)	All Methods	One per extraction batch, when insufficient sample is provided for MS/MSD/SD	Within TALS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike (MS)	All Methods	One per extraction batch	Within TALS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	All Methods	One per extraction batch	Within TALS MLG Limits	Refer to SOP SA-QA-017

QC Item	Method	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	All Methods	Spiked in all CCVIS, samples, and batch QC items	CCVIS: Area within 50% to +200% of corresponding level in the ICAL. Samples & batch QC items: - Area within 50% to +200% of CCVIS. - RT within +/-30 seconds from previous CCVIS.	Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	All Methods	Spiked (during extraction procedure) in all samples and batch QC items.	Within MLG limits 1 Acid / 1 Base Allowance: samples and MS/MSD, only, with %R >10% Surrogate Threshold Dilution Factor = 10	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Initial Demonstration of Capability (IDOC)	All Methods	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	All Methods	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Method	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	All Methods	Spiked in all CCVIS, samples, and batch QC items	CCVIS: Area within 50% to +200% of corresponding level in the ICAL. Samples & batch QC items: - Area within 50% to +200% of CCVIS. - RT within +/-30 seconds from previous CCVIS.	Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	All Methods	Spiked (during extraction procedure) in all samples and batch QC items.	Within MLG limits 1 Acid / 1 Base Allowance: samples and MS/MSD, only, with %R >10% Surrogate Threshold Dilution Factor = 10	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Reporting Limit Verification (RLV)	All Methods	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Method	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	All Methods	Spiked in all CCVIS, samples, and batch QC items	CCVIS: Area within 50% to +200% of corresponding level in the ICAL. Samples & batch QC items: - Area within 50% to +200% of CCVIS. - RT within +/-30 seconds from previous CCVIS.	Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	All Methods	Spiked (during extraction procedure) in all samples and batch QC items.	Within MLG limits 1 Acid / 1 Base Allowance: samples and MS/MSD, only, with %R >10% Surrogate Threshold Dilution Factor = 10	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Detection Limit Study (MDL)	All Methods	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Method	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	All Methods	Spiked in all CCVIS, samples, and batch QC items	CCVIS: Area within 50% to +200% of corresponding level in the ICAL. Samples & batch QC items: - Area within 50% to +200% of CCVIS. - RT within +/-30 seconds from previous CCVIS.	Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	All Methods	Spiked (during extraction procedure) in all samples and batch QC items.	Within MLG limits 1 Acid / 1 Base Allowance: samples and MS/MSD, only, with %R >10% Surrogate Threshold Dilution Factor = 10	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
MDL Verification (MDLV)	All Methods	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Preventative Maintenance and Troubleshooting

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
Septum							X	Replace, recommended daily
Splitless Disc							X	Replace, recommended daily
Column/Injector							X	Change sleeve and cut front of column, recommended daily
Autosampler							X	Clean syringe as needed; replace syringe as needed
Injector Port							X	Replace injector port as needed
Lines							X	Flush lines as needed; replace lines as needed
Column							X	Change column as needed
Mass Spectrometer							X	Clean as needed
Rough Pump							X	Change oil as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

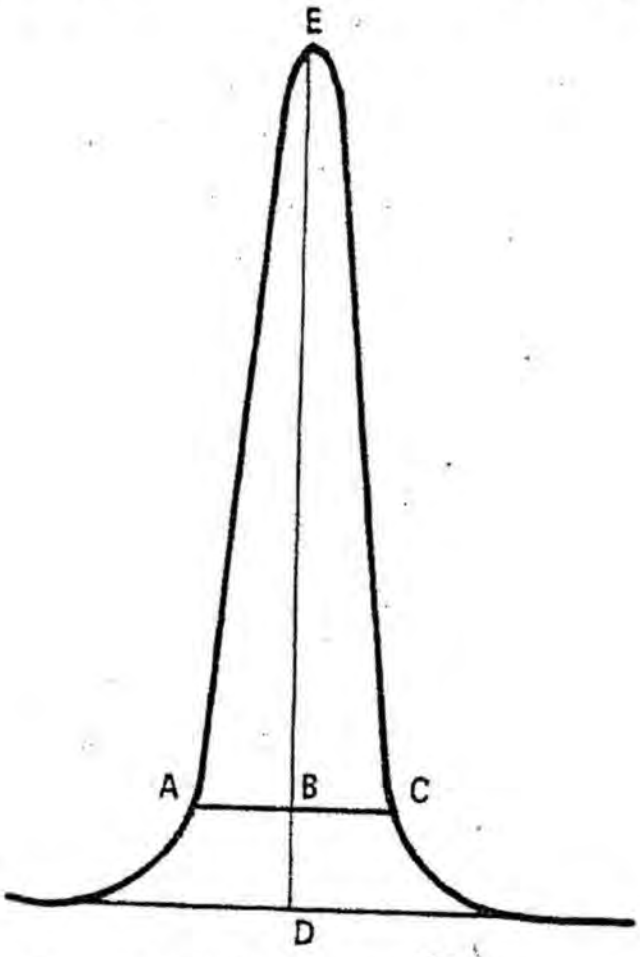
Instrument Labeling

Each instrument must be labeled with its name or identification. Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Attachment 5:
DFTPP Criteria

m/z	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Attachment 6:
Example Tailing Factor Calculation



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Attachment 7:
Target Compound Information: Quant Ions and ISTDs*

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
1,1'-Biphenyl	154	76		3	X	
1-Chloronaphthalene	162	127	164	3	X	
1,2,3-Trichlorobenzene	216	214		1		X
1,2,4,5-Tetrachlorobenzene	216	214	179	3		X
1,2,4-Trichlorobenzene	180	182	145	2	X	
1,2-Dichlorobenzene	146	148		1	X	
1,2-Diphenylhydrazine	77	105	182	4	X	
1,3,5-Trichlorobenzene	180	145		1		X
1,3,5-Trinitrobenzene	213	74	120	4		X
1,3-Dichlorobenzene	146	148	111	1	X	
1,3-Dinitrobenzene	168	76	50	3		X
1,4-Dichlorobenzene	146	148	111	1	X	
1,4-Dioxane	88	58	45	1	X	
1,4-Naphthoquinone	158	104	76	3		X
1,4-Dinitrobenzene	168	76	50	3		
1-Diallate	86	43	234	4		X
1-Methylnaphthalene	142	141		2	X	
1-Naphthylamine	143	115	116	3		X
2,2'-oxybis[1-chloropropane]	45	121		1	X	
2,3,4,5-Tetrachlorophenol	232	230	131	3		
2,3,4,6-Tetrachlorophenol	232	230	131	3	X	
2,3,5,6-Tetrachlorophenol	232	96	131	3	X	
2,3,6-Trichlorophenol	196	198		2	X	
2,3-Dimethylphenol	107	122	121	1	X	
2,4 & 2,5-Dimethylphenol	107	122	121	1	X	
2,4,5-Trichlorophenol	196	198	200	3	X	
2,4,6-Trichlorophenol	196	198	200	3	X	
2,4-Dinitrochlorobenzene	202	110	75	2		X
2,4-Dichlorophenol	162	164	98	2	X	
2,4-Dimethylphenol	122	107	121	2	X	
2,4-Dinitrophenol	184	63	154	3	X	
2,4-Dinitrotoluene	165	89	63	3	X	
2,5-Dimethylphenol	107	122	121	1	X	
2,5-Dinitrophenol	184	63		1	X	
2,6-Dichlorophenol	162	164	98	2	X	
2,6-Dimethylphenol	107	122	121	1	X	
2,6-Dinitrotoluene	165	89	63	3	X	
2-Acetylaminofluorene	181	180	223	5		X
2-Chloronaphthalene	162	164	127	3	X	

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
2-chloronitrobenzene/4-chloronitrobenzene	157	111	75	2		X
2-Chlorophenol	128	130	64	1	X	
2-Diallate	86	43	234	4		X
2-Mercaptobenzothiazole	167	108		5		X
2-Methylnaphthalene	142	141		2	X	
2-Methylphenol	107	108	77	1	X	
2-Naphthylamine	143	115	116	3		X
2-Nitroaniline	65	92	138	3	X	
2-Nitrobiphenyl	152	115		3		X
2-Nitrophenol	139	109	65	2	X	
2-Picoline	93	66		1		X
2-Toluidine	106	107	79	2		X
3 & 4 Methylphenol	107	108		1	X	
3,3'-Dichlorobenzidine	252	254	126	5	X	
3,3'-Dimethylbenzidine	212	196	106	5		X
3,4-Dichloronitrobenzene	109	133	191	2		X
3,4-Dimethylphenol	107	122	121	1	X	
3-Methylcholanthrene	268	252	253	5		X
3-Nitroaniline	138	108	92	3	X	
3-Nitrobiphenyl	152	199		4		X
3-Nitrochlorobenzene	157	111	75	2		X
3-Nitrophenol	139	65		1	X	
4,6-Dinitro-2-methylphenol	198	105	121	4	X	
4-Aminobiphenyl	169	168	170	4		X
4-Bromophenyl phenyl ether	248	250	141	4	X	
4-Chloro-3-methylphenol	107	144	142	2	X	
4-Chloroaniline	127	129	65	2	X	
4-Chloronitrobenzene	157	111	75	2		X
4-Chlorophenol	65	128		2	X	
4-Chlorophenyl phenyl ether	204	141	206	3	X	
4,4'-Methylene bis(2-chloroaniline)	231	266	268	5		X
4-Nitroaniline	138	108	92	3	X	
4-Nitrobiphenyl	152	199		4		X
4-Nitrophenol	65	109	139	3	X	
4-Nitroquinoline-1-oxide	174	101	128	4		X
5-Nitro-o-toluidine	152	77	106	3		
6-Methylchrysene	242	241	239	5		X
7,12-Dimethylbenz(a)anthracene	256	239	241	6		X
Acenaphthene	154	153	152	3	X	
Acenaphthylene	152	151	153	3	X	
Acetophenone	105	77	51	2	X	
Acrylamide	71	44	55	1		X

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
alpha,alpha-Dimethyl phenethylamine	58	91	42	2		X
alpha-Pinene	93	121		1		X
Aniline	93	66		1		X
Anthracene	178	176	179	4	X	
Aramite, Total	185	191	319	5		X
Aramite-1	185	191	319	5		X
Aramite-2	185	191	319	5		X
Atrazine	200	173	215	4	X	
Benzaldehyde	77	105		1	X	
Benzidine	184	92	185	4		X
Benzo[a]anthracene	22	229	226	5	X	
Benzo[a]pyrene	252	125	253	6	X	
Benzo[b]fluoranthene	252	253	125	6	X	
Benzo[g,h,i]perylene	276	277	138	6	X	
Benzo[k]fluoranthene	252	253	125	6	X	
Benzoic acid	105	122		2	X	
Benzyl alcohol	108	79	77	1	X	
Bis(2-chloroethoxy)methane	93	123	95	2	X	
Bis(2-chloroethyl)ether	63	93	95	1	X	
Bis(2-ethylhexyl) phthalate	149	167	279	5	X	
Butyl benzyl phthalate	149	91	206	5	X	
Caprolactam	113	55		2	X	
Carbazole	167			4	X	
Catechol	110	64		1		X
Chlorobenzilate	139	251	111	5	X	
Chrysene	228	226	229	5	X	
Di(2-ethylhexyl)adipate	129	57		3	X	
Diallate	86	43		4		X
Dibenz(ah) acridine	279	280	278	6	X	
Dibenz(aj)acridine	279	280	278	6	X	
Dibenz(a,h)anthracene	287	139	279	6	X	
Dibenzofuran	168	139		3	X	
Diethyl phthalate	149	177	150	3	X	
Dimethoate	87	93	125	4		X
Dimethyl phthalate	163	194	164	3	X	
Dimethyl terephthalate	194	135		2		X
Di-n-butyl phthalate	149	150	104	4	X	
Di-n-octyl phthalate	149	43		5	X	
Dinoseb	211	163	147	4	X	
Disulfoton	88	60		4		X
Ethyl methanesulfonate	79	109	97	1		X
Ethyl Parathion	109	97		4		X

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
Famphur	218	93	125	4		X
Fluoranthene	202	203	101	4	X	
Fluorene	166	165	167	3	X	
Hexachlorobenzene	284	142	249	4	X	
Hexachlorobutadiene	225	223	227	2	X	
Hexachlorocyclopentadiene	237	235	272	3	X	
Hexachloroethane	117	201	199	1	X	
Hexachlorophene	196	198		6		X
Hexachloropropene	213	211	215	2		X
Hexadecane	57	43	71	1	X	
Indene	57	43	71	1	X	
Indeno[1,2,3-cd]pyrene	276	138		5	X	
Isodrin	193	195	66	4	X	
Isophorone	82	95	138	2	X	
Isosafrole	162	104	131	2		X
Kepone	272	237	357	5		X
Methapyrilene	97	58	191	4		X
Methyl Benzoate	105	77	51	1		X
Methyl methanesulfonate	80	79	65	1		X
Methyl parathion	109	125		4		X
Methyl Phenols, Total	107	108		1	X	
Monomethyl Terephthalate	149	121		3		X
Naphthalene	128	129		2	X	
n-Decane	57	43	41	1	X	
Nitrobenzene	77	123	65	2	X	
N-Nitro-o-toluidine	152	77	106	3		X
N-Nitrosodiethylamine	102	42	44	1	X	
N-Nitrosodimethylamine	42	74		1		X
N-Nitrosodi-n-butylamine	84	57	41	2		X
N-Nitrosodi-n-propylamine	70	42		1	X	
N-Nitrosodiphenylamine	169	168	167	4	X	
N-Nitrosomethylethylamine	88	42	43	1		X
N-Nitrosomorpholine	56	86		1		X
N-Nitrosopiperidine	114	42	55	2		X
N-Nitrosopyrrolidine	100	41	42	2		X
N-Octadecane	57	43	41	1	X	
o,o',o''-Triethylphosphorothioate	65	97	93	2		X
p-Dimethylamino azobenzene	120	225	77	5		X
Pentachlorobenzene	250	248	252	3		X
Pentachloroethane	167	165	169	1		X
Pentachloronitrobenzene	237	295	142	4		X
Pentachlorophenol	266	264	268	4	X	

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
Phenacetin	108	109	179	3		X
Phenanthrene	178	176	179	4	X	
Phenol	94	66	65	1	X	
Phenyl ether	170	141		3		X
Phenylmercapatan	110	66	109	1		X
Phorate	75	121		4		X
p-Phenylene diamine	108	80	107	2		X
Pronamide	173	175	145	4		X
Pyrene	202	200	203	5	X	
Pyridine	79	52	51	1		X
Quinoline	129	102		1		X
Safrole, Total	162	104	135	2		X
Sulfotepp	97	65		4		X
Thionazin	107	96	97	4		X
Toluic acid	91	119	136	2		X
SURROGATES						
Nitrobenzene-d5[1]	82	128	54	2		
2-Fluorobiphenyl[1]	172	171		3		
Phenol-d5 [2]	99	71		1		
2-Fluorophenol [2]	112	64		1		
2,4,6-Tribromophenol [2]	330	332	144	3		
Terphenyl-d141	244	122	212	5		
OTP[2] (8270D_LL_PAH Only)	244	122	212	5		

[1]Base Surrogate
[2]Acid Surrogate

INTERNAL STANDARDS

1,4-Dichlorobenzene-d4	152	150	115	1		
Naphthalene-d8	136	68		2		
Acenaphthene-d10	164	162	160	3		
Phenanthrene-d10	188	94	80	4		
Chrysene-d12	240	236	120	5		
Perylene-d12	264	265	260	6		

For a complete list of target analytes for each method refer to the TALS Method Limit Groups (MLGs).

Attachment 8:
Standard Preparation Information

Purchased Standards

Vendor Name	Standard Description	Part Number	Concentration (ug/mL)
Supelco	8270 Tuning Mix	47548-U	1000
Restek	8270 L1/S1	570666	1000
Restek	8270 L1/S1 SS	570666.sec	1000
Restek	8270 L1/S9	569730	2000
Restek	8270 L1/S9 SS	569730.SEC	2000
Restek	8270 L1/S10	569731	2000
Restek	8270 L1/S10 SS	569731.SEC	2000
Restek	8270 L1/S11	569732	2000
Restek	8270 L1/S11	569732.SEC	2000
Restek	8270 L2/S1	567678	1000
Restek	8270 L2/S1 SS	567678.sec	1000
Restek	8270 L2/S2	569733	1000
Restek	8270 L2/S2 SS	569733.SEC	1000
Restek	8270 L2/S3	567680	2000
Restek	8270 L2/S3 SS	567680.sec	2000
Restek	8270 L2/S4	567681	1000
Restek	8270 L2/S4 SS	567681.sec	1000
Restek	8270 L2/S5	567682	2000
Restek	8270 L2/S5 SS	567682.sec	2000
Restek	8270 L2/S7	568726	2000
Restek	8270 L2/S7 SS	568726.sec	2000
Restek	8270 L2/S8	568727	2000
Restek	8270 L2/S8 SS	568727.sec	2000
Restek	8270 L2/ADD	568396	2000
Restek	8270 L2/ADD SS	568396.sec	2000
Restek	8270 L3/S1	567683	2000
Restek	8270 L3/S1 SS	567683.sec	2000
Restek	Hexachlorophene	568395	5000
Restek	Hexachlorophene SS	568395.sec	5000
Restek	8270 SURR	567685	5000
Restek	o-Terphenyl	31066	2000
Restek	8270 Internal Standard	567684	2000

Expiration:
Un-opened: Manufacturer's expiration date
Opened: 6 months from date opened or manufacturer's expiration date, whichever is sooner

Storage:
Un-opened: Manufacturer's suggested
Opened: Refrigerated at 0-6°C.

Prepared Standards

Note:
The following conditions apply for all prepared standards:
Final Solvent = Methylene Chloride
Storage = 0-6°C
Expiration = 3 months from preparation date

EPA 625 / EPA 8270D / EPA 625.1 / EPA 8270E DFTPP
TALS Name = SMDFTPP050

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 Tuning Mix	1000	500	10	50

SM LIST 1A INT
TALS Name = SM-List1AInt

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1	1000	4000	10	400
8270 L1/S9	2000	2000		
8270 SURR	5000	1000		

SM LIST 1B INT
TALS Name = SM-List1BInt

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S11	2000	2000	10	400
8270 L1/S10	2000	2000		

SM LIST 2-AP9 INT
TALS Name = SM-L2-AP9INT

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/ADD	2000	1000	10	200
8270 L2/S1	1000	2000		
8270 L2/S2	1000	2000		
8270 L2/S3	2000	1000		
8270 L2/S5	2000	1000		

SM-OP-INT
TALS Name = SM-8270OPINTC

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S4	1000	2000	5	400
8270 L2/S7	2000	1000		
8270 L2/S8	2000	1000		

LIST 1A Second Source ICV
TALS Name = SM-LIST1A-ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1-SS	1000	160	2	80
8270 L1/S9-SS	2000	80		
8270 SURR	5000	32		
8270 ISTD	2000	40		

LIST 1B Second Source ICV
TALS Name = SM-LIST1B-ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S10-SS	2000	80	2	80
8270 L1/S11-SS	2000	80		
8270 ISTD	2000	40		

LIST 2 Second Source ICV
TALS Name = SM-LIST2-ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S1-SS	1000	160	2	80
8270 L2/S2-SS	1000	160		
8270 L2/S3-SS	2000	80		
8270 L2/S5-SS	2000	80		
8270 L2/ADD-SS	2000	80		

Hexachlorophene/Organophos Second Source ICV
TALS Name = SM-HEXAOPHICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
R-HEXA-SS	5000	320	2	80
8270 L2/S4-SS	1000	160		
8270 ISTD	2000	40		
8270L2/S8-SS	2000	80		
8270L2/S7-SS	2000	80		

Low-Level Internal Standard Solution
TALS Name = SM-LLISTD

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 Internal Standard Mix	2000	1000	10	200

8270LL Full Standard
TALS Name = SM-LL BNA INT

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1	1000	500	10	50
8270 L2/ S10	2000	250		
8270 L1/ S11	2000	250		
8270 L1/ S9	2000	250		
8270 SURR	5000	100		
OTP	2500	200		

8270LL AP-9 Intermediate Mix
TALS Name = SMLLA9INT(CR)

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S1	1000	500	10	50
8270L2/S2	1000	500		
8270 L2/S3	2000	250		
8270 L2/S5	2000	250		
8270 L2/ADD	2000	250		

8270LL Hexachlorophene/Organophos Intermediate Mix
TALS Name = SM-OP-INT

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S4	1000	500	10	50

8270L2/S7	2000	250		
8270 L2/S8	2000	250		

Low-Level LIST 1 Second Source ICV
TALS Name = SMLLBNAICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1-SS	1000	20	2	10
8270 L1/S10-SS	2000	10		
8270 L1/ S11-SS	2000	10		
8270 L1/ S9-SS	2000	10		
8270 SURR	5000	4		
OTP	2000	8		

Low-Level Appendix 9 Second Source ICV
TALS Name = SMLLAP9ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S1	1000	10	2	10
8270 L2/S2	2000	20		
8270 L2/S3	2000	20		
8270 L2/S5	2000	10		
8270 L2/ADD	5000	10		
LLISTD Solution	2000	20		

Low-Level Hexachlorophene/Organophos Second Source ICV
TALS Name = LLOPHhexICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
R-HEXA-SS	5000	200	2	10
8270 L2/S4-SS	1000	20		
LLISTD Solution	200	20		
8270 L2/S7-SS	2000	10		
8270 L2/S8-SS	2000	10		

SM List 1A Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LIST 1A-005	SM LIST 1A INT	200	25	40	2	5
LIST 1A-010	SM LIST 1A INT	200	50	40	2	10
LIST 1A-020	SM LIST 1A INT	200	100	40	2	20
LIST 1A-040	SM LIST 1A INT	200	200	40	2	40
LIST 1A-060	SM LIST 1A INT	200	300	40	2	60
LIST 1A-080	SM LIST 1A INT	200	400	40	2	80
LIST 1A-100	SM LIST 1A INT	200	500	40	2	100
LIST 1A-160	SM LIST 1A INT	200	800	40	2	160
LIST 1A-200	SM LIST 1A INT	200	1000	40	2	200

SM List 1B Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LIST 1B-005	SM LIST 1B INT	200	25	40	2	5
LIST 1B-010	SM LIST 1B INT	200	50	40	2	10
LIST 1B-020	SM LIST 1B INT	200	100	40	2	20
LIST 1B-040	SM LIST 1B INT	200	200	40	2	40
LIST 1B-060	SM LIST 1B INT	200	300	40	2	60
LIST 1B-080	SM LIST 1B INT	200	400	40	2	80
LIST 1B-100	SM LIST 1B INT	200	500	40	2	100
LIST 1B-160	SM LIST 1B INT	200	800	40	2	160
LIST 1B-200	SM LIST 1B INT	200	1000	40	2	200

SM List 2 Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LIST 2-005	SM-LIST 2-AP9 INT	200	50	40	2	5
LIST 2-010	SM-LIST 2-AP9 INT	200	100	40	2	10
LIST 2-020	SM-LIST 2-AP9 INT	200	200	40	2	20
LIST 2-050	SM-LIST 2-AP9 INT	200	500	40	2	50
LIST 2-080	SM-LIST 2-AP9 INT	200	800	40	2	80
LIST 2-100	SM-LIST 2-AP9 INT	200	1000	40	2	100
LIST 2-200	SM-LIST 2-AP9 INT	200	2000	40	2	200

Hexachlorophene/Ophos Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
HEXAOP-005	SMOPINT	1000	10	40	2	5
	R-HEXA	5000	20			50
HEXAOP-010	SMOPINT	1000	20	40	2	10
	R-HEXA	5000	40			100
HEXAOP-020	SMOPINT	1000	40	40	2	20
	R-HEXA	5000	80			200
HEXAOP-050	SMOPINT	1000	100	40	2	50
	R-HEXA	5000	200			500
HEXAOP-080	SMOPINT	1000	160	40	2	80
	R-HEXA	5000	320			800
HEXAOP-100	SMOPINT	1000	200	40	2	100
	R-HEXA	5000	400			1000
HEXAOP-200	SMOPINT	1000	400	40	2	200
	R-HEXA	5000	800			2000

SM 8270LL BNA Working Standards

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	LL ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LLBNA0.20	BNA 50	50	8	40	2	0.20
LLBNA 1.0	BNA 50	50	40	40	2	1.0
LLBNA 2.0	BNA 50	50	80	40	2	2.0
LLBNA 5.0	BNA 50	50	200	40	2	5.0
LLBNA 10	BNA 50	50	400	40	2	10
LLBNA 15	BNA 50	50	600	40	2	15
LLBNA 20	BNA 50	50	800	40	2	20

SM 8270LL BNA Working Standards

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	LL ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LLAP91.0	LLAP9 Intermediate	50	40	20	2	1.0
LLAP92.0	LLAP9 Intermediate	50	80	20	2	2.0
LLAP95.0	LLAP9 Intermediate	50	200	20	2	5.0
LLAP910.0	LLAP9 Intermediate	50	400	20	2	10
LLAP915.0	LLAP9 Intermediate	50	600	20	2	15
LLAP920.0	LLAP9 Intermediate	50	800	20	2	20

Low-Level Hexachlorophene/Ophos Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LLOphHex1.0	SM-OP-INT	50	40	20	2	1.0
	R-HEXA	5000	20			50
LLOphHex2.0	SM-OP-INT	50	80	20	2	2.0
	R-HEXA	5000	40			100
LLOphHex5.0	SM-OP-INT	50	200	20	2	5.0
	R-HEXA	5000	100			250
LLOphHex10.0	SM-OP-INT	50	400	20	2	10
	R-HEXA	5000	200			500
LLOphHex15.0	SM-OP-INT	50	600	20	2	15
	R-HEXA	5000	300			750
LLOphHex20.0	SM-OP-INT	50	800	20	2	20
	R-HEXA	5000	400			1000

Attachment 9:
Procedures for SIM Analyses

Analytical Procedures for SIM Analysis

The mass spectrometer (MS) may be used in the selected ion monitoring mode to increase the sensitivity of the GC/MS analysis. In SIM mode, the MS is set to monitor for only a few selected ions; therefore, more ions of the selected mass(es) can be filtered and counted, resulting in an increase of sensitivity of 10 to 100 fold over scan monitoring, depending on the compound. SIM GC/MS analysis sacrifices selectivity for sensitivity since only a few characteristic ions are monitored. A spectral match cannot be made against a reference library and tentatively identified compounds (TICs) cannot be determined from a SIM run. In general, the SIM analysis should be used only when a few compounds are required to be monitored. SIM can also be used to confirm the presence of a target compound determined by GC when the routine GC/MS scan analysis cannot provide confirmation.

The following procedures are based on the guidance in SW-846 Method 8270D/8270E. The analytical sequence is the same as is given in the associated analytical SOPs, with mass tune criteria and calibration verification every 12 or 24 hours.

- Analyze the 50 ug/mL DFTPP in the scan mode. Evaluate the DFTPP against the acceptance criteria given in previous sections in the SOP.

- Determine the approximate retention time of the target by analyzing the target analyte(s) by GC/MS scan.

- Set the MS to monitor for the characteristic ions (minimum of two ions) for the target analyte(s) and internal standard(s). The ion dwell time should be set to give at least six integrations across the peak. A dwell time of 50-100ms is common.

- Prepare and analyze a minimum of three calibration standards for the target compounds. The lowest standard should be at the required quantitation limit and the other two standards should define the working range of the GC/MS. The internal standard(s) should be at a concentration of approximately 4ug/mL for SVOC.

- Evaluate the resulting calibration curve according to the initial calibration procedures given in SOP SA-QA-016. If r^2 is >0.990, the calibration curve is acceptable.

  If the initial calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 24-hour clock for the DFTPP analysis in 625 SIM and the 12-hour clock for the DFTPP analysis in 8270 SIM.

SIM Analysis of Target Compounds

- Add an appropriate volume of internal standard to the extract or sample to give the same concentration as in the calibration standards. Analyze the extract or sample under the same conditions as the standard.

Note: It is advised that the extract be split before addition of ISTD solution. One split can be utilized for scan analysis and second split reserved for SIM analysis.

- Compare the retention time of the sample to the retention time of the standard.

  If a peak is detected at the retention time of the target compound containing the selected masses in the same ratio as the standard, the peak is confirmed as the target compound and the concentration is calculated. The relative intensities of the ions in the sample should agree within ±20% of the intensities of the ions in the standard.

  If a peak is not present at the appropriate retention time or if the ratios of the ions are not the same as the standard, the analyte is not confirmed

- If the concentration of the target compound exceeds the highest calibration standard, analyze a more dilute aliquot of the sample or extract, maintaining the internal standard concentration at the same level as the calibration standards.

Attachment 10:
Poor Responder Information

The following analytes have been identified, in the reference method and/or via historical data, to be poor and/or erratic performers:

1,2,4,5-Tetrachlorobenzene
1,3,5-Trinitrobenzene
1,3-Dinitrobenzene
1,4-Dinitrobenzene
1,4-dioxane
1,4-Naphthoquinone
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,3,5,6-Tetrachlorophenol
2,4-dinitrophenol
2,6-Dichlorophenol
2-Acetylaminofluorene
2-Naphthylamine
2-Picoline
2-secbutyl-4,6-dinitrophenol (Dinoseb)
3&4-methylphenol
3,3'-Dimethylbenzidine
3'3-Dichlorobenzidine
3-Methylcholanthrene
4,4''-Methylenebis(2-chloroaniline)
4,4-methylenebis (2-chloroaniline)
4,6-Dinitro-2-methyl- phenol,
4-Aminobiphenyl
4-Chloroaniline,
4-nitrophenol
4-Nitroquinoline-1-oxide
5-Nitro-o-toluidine
6-Methylchrysene
7,12-Dimethylbenz(a)anthracene
a,a-Dimethyl-phenethylamine
Acrylamide
Aniline
Aramite (#1)
Aramite (#2)
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Chlorobenzilate
Diallate (#1)
Diallate (#2)
Dibenz(a,h)acridine
Dibenz(a,j)acridine
Dimethoate
Diphenylamine
Disulfoton
Dodecanol
Ethyl methanesulfonate
Famphur
Hexachlorocyclopentadiene
Hexachloroethane
Hexachlorophene
Hexachloropropene
Hydroquinone
Isodrin
Isosafrole (#1)
Isosafrole (#2)
Kepone
Methapyrilene
Methyl methanesulfonate
Methyl Parathion
N-Nitrosodiethylamine
n-Nitrosodimethylamine
n-Nitrosodi-n-butylamine
N-Nitrosomethylethylamine
N-Nitrosomorpholine
N-Nitrosopiperidine
N-Nitrosopyrrolidine
o,o,o-Triethylphosphorothioate
o-Toluidine
p-(Dimethylamino)azobenzene
Parathion
Pentachlorobenzene
Pentachloroethane
Pentachloronitrobenzene
Pentachlorophenol
Phenacetin
Phenol
Phorate
p-Phenylenediamine
Pronamide
Pyridine
Quinoline
Safrole
Sulfotepp
Thionazin

These analytes are exempt from the LCS, MS, MSD, Sporadic Marginal Exceedance, ICV, and CCV criteria for EPA method 8270D and EPA 8270E as listed in this SOP.

Attachment 11:
EPA 8270D Minimum RRF Criteria

Analyte	Minimum RRF Criteria
1,2,4,5-Tetrachlorobenzene	0.010
1,1'-Biphenyl	0.010
2,3,4,6-Tetrachlorophenol	0.010
2,2'-Oxibis(1-chloroporopane)	0.010
2-Chloronaphthalene	0.800
2-Chlorophenol	0.800
2-Methylphenol	0.700
2-Methylnaphthalene	0.400
2-Nitroaniline	0.010
2-Nitrophenol	0.100
2,4-Dichlorophenol	0.200
2,4-Dimethylphenol	0.200
2,4-Dintirophenol	0.010
2,4-Dinitrotoluene	0.200
2,4,5-Trichlorophenol	0.200
2,4,6-Trichlorophenol	0.200
2,6-Dinitrotoluene	0.200
3-Nitroaniline	0.010
3,3'-Dichlorobenzidine	0.010
4-Bromophenyl phenyl ether	0.100
4-Chloro-3-methylphenol	0.200
4-Chloroaniline	0.010
4-Chlorophenyl phenyl ether	0.400
4-Methylphenol	0.600
4-Nitroaniline	0.010
4-Nitrophenol	0.010
4,6-Dinitro-2-methylphenol	0.010
Acenaphthene	0.900
Acenaphthylene	0.900
Acetophenone	0.010
Atrazine	0.010
Anthracene	0.700
Benzaldehyde	0.010
Benzo(a)anthracene	0.800
Benzo(a)pyrene	0.700
Benzo(b)fluoranthene	0.700
Benzo(g,h,i)perylene	0.500
Benzo(k)fluoranthene	0.700
Bis(2-choroethyl)ether	0.700
Bis (2-chloroethoxy)methane	0.300
Bis (2-ethylhexyl)phthalate	0.010
Butyl benzyl phthalate	0.010

Caprolactam	0.010
Carbazole	0.010
Analyte	**Minimum RRF Criteria**
Chrysene	0.700
Dibenz(a,h)anthracene	0.400
Dibenzofuran	0.800
Diethyl phthalate	0.010
Dimethyl phthalate	0.010
Di-n-butyl phthalate	0.010
Di-n-octyl phthalate	0.010
Fluoranthene	0.600
Fluorene	0.900
Hexachlorobenzene	0.100
Hexachlorobutadiene	0.010
Hexachlorocyclopentadiene	0.050
Hexachloroethane	0.300
Indeno(1,2,3-cd)pyrene	0.500
Isophorone	0.400
Naphthalene	0.700
Nitrobenzene	0.200
N-Nitroso-di-n-propylamine	0.500
N-Nitroso-di-phenylamine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Phenol	0.800
Pyrene	0.600

Attachment 12:
Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

SEMIVOLATILE GC/MS DEPARTMENT

1. Rinse glassware 3 times with methylene chloride
2. Place glassware, top-down, within storage rack and allow to air dry.
3. Store glassware in closed drawer.

FSM108:05.14.14:0

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

18.0 Revision History

Summary of Changes:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Incorporated references to EPA 8270E.
- Incorporated the following changes as per SC DHEC Technical Deficiency Letter dated 05/18/19:
  - Added note the SC DHEC prohibits the use of sporadic marginal exceedance. Section 9.1.1 and Section 16.2.4
  - Corrected spelling of decafluorotriphenyl phosphine. Section 9.2.1
  - Clarified calculation to reference waters. Section 11.2.3
  - Added requirement to evaluate %RSE. Section 9.2.2.1 and Attachment 3

Addendum to the Environmental Health and Safety Manual

TestAmerica Savannah
5102 LaRoche Avenue
Savannah, GA 31404
Phone: 912-354-7858
Fax: 912-352-0165
www.testamericainc.com

Facility Distribution No. 1 **Distributed To: TALS File System Shares**

TestAmerica Savannah Emergency Quick Reference Guide

Emergency Contacts:

- Bernard Kirkland – 912-339-0059
- Whitney Palefsky – 912-695-8788

Hazardous Waste Types:

- Corrosives – Acids
- Flammable Materials – Volatile Organics, Acetone, Hexane
- Toxic Materials – PCBs

Waste Codes:

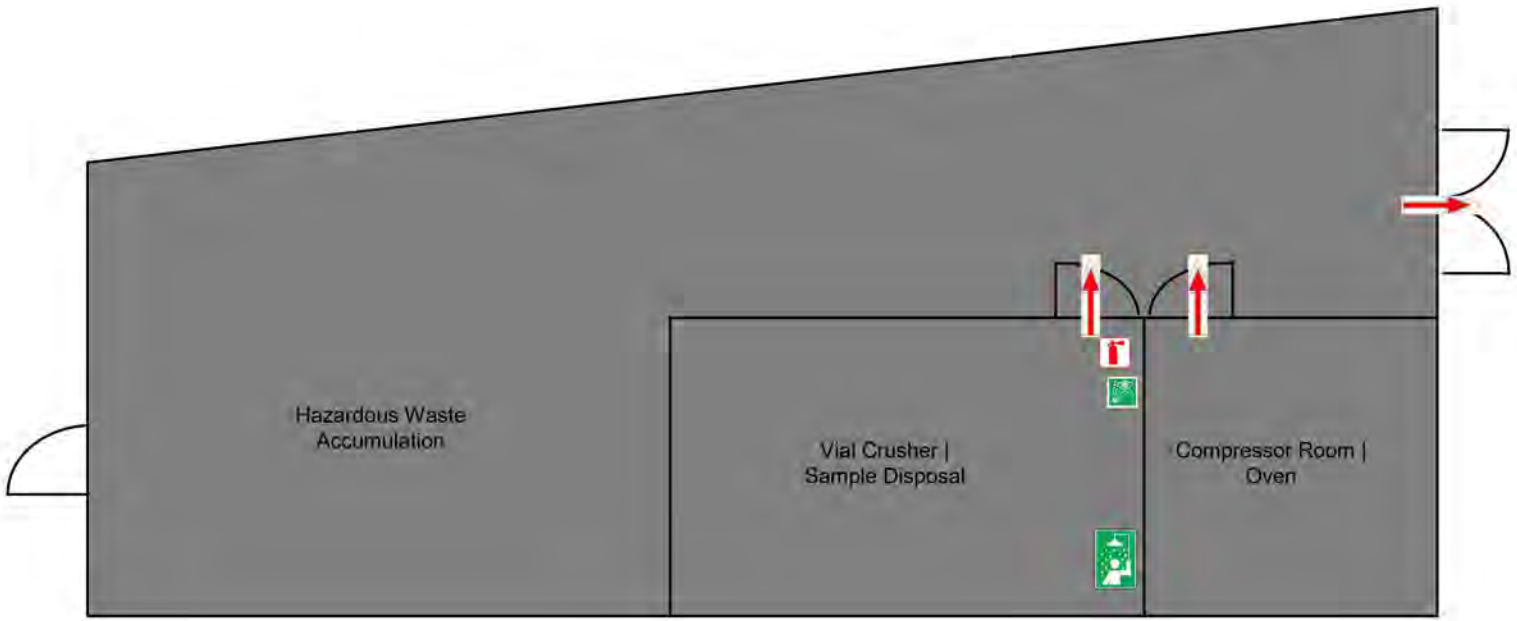
- NA3077, HAZARDOUS WASTE, SOLID, N.O.S.
- UN1208, WASTE HEXANES
- UN1230, WASTE METHANOL SOLUTION
- UN1282, WASTE PYRIDINE
- UN1593, WASTE DICHLOROMETHANE
- UN1760, WASTE CORROSIVE LIQUIDS, N.O.S.
- UN1993, WASTE FLAMMABLE LIQUIDS, N.O.S.
- UN2920, WASTE CORROSIVE LIQUIDS, FLAMMABLE, N.O.S.
- UN3264, WASTE CORROSIVE LIQUID, ACIDIC, INORGANIC, N.O.S.

Other Hazards:

- Pressurized Gases
- Bulk Chemical Storage – Indoor/Outdoor
    - Indoor – Dichloromethane – Toxic
    - Outdoor – Liquid Nitrogen – Cryogenic; Liquid Argon – Cryogenic

Attachments:

- Facility Floor Plans
- Site Map



Legend

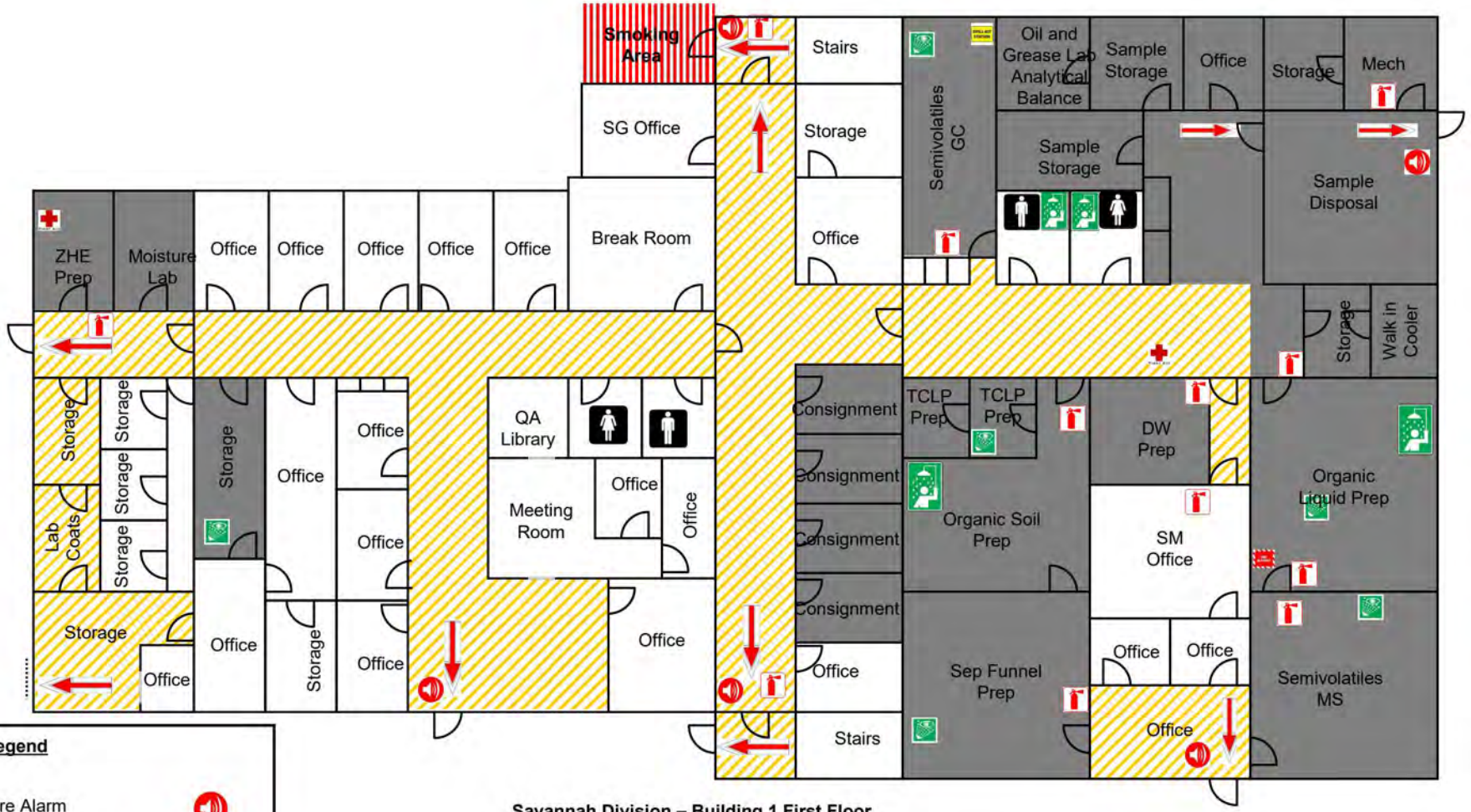
- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Savannah Division – Building 1 Waste Disposal

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA001:12.10.14:0



Savannah Division – Building 1 First Floor
16,600 Square Feet

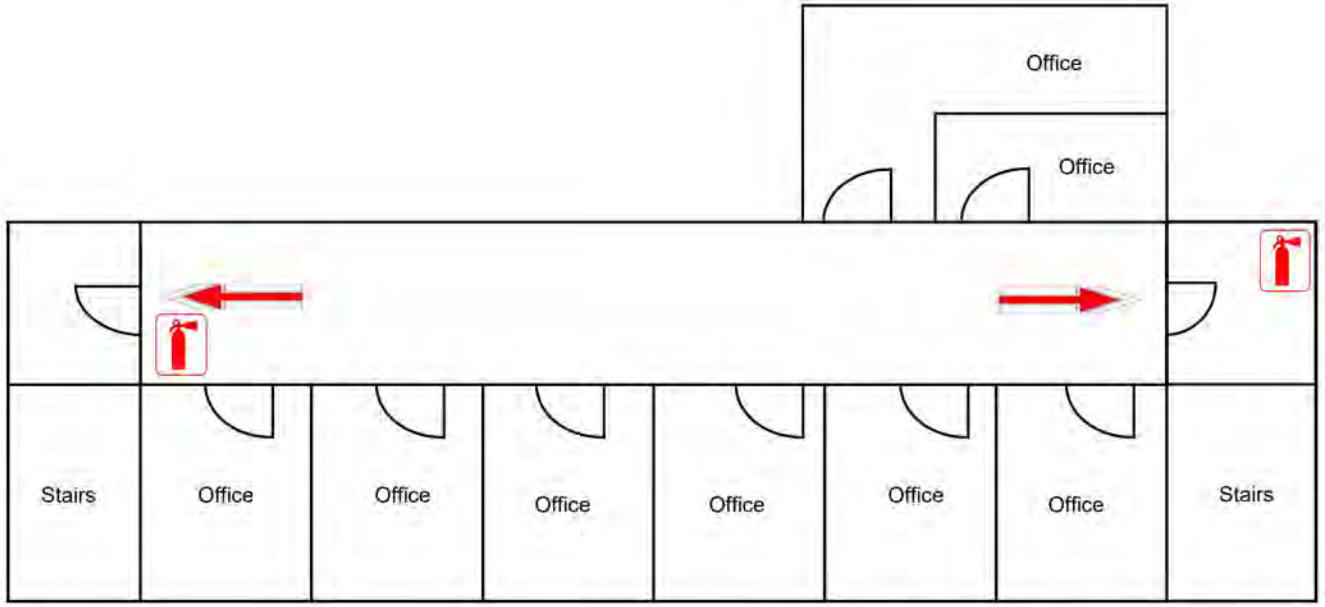
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA002:12.10.14:0



Savannah Division – Building 1 Second Floor
2,200 Square Feet

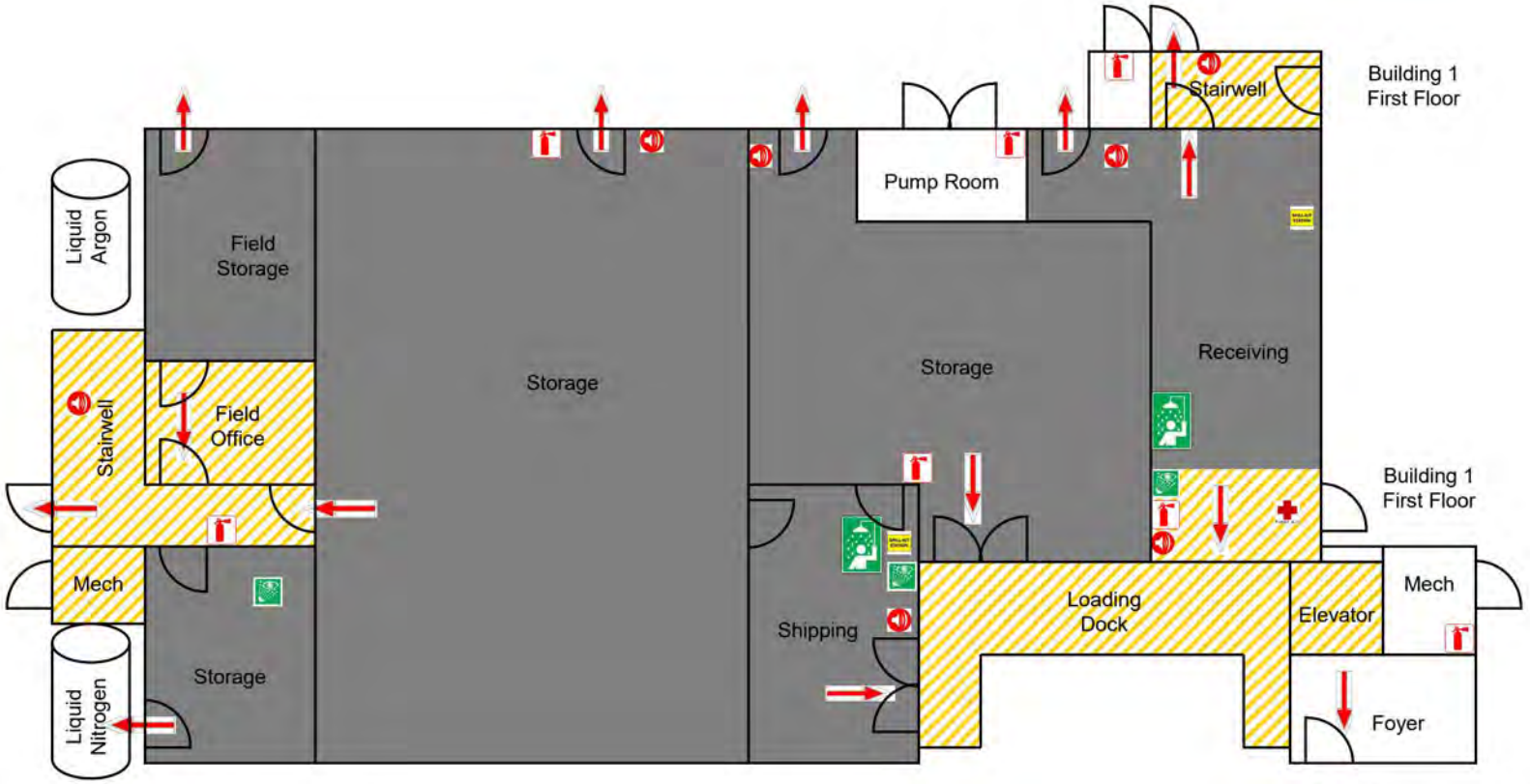
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA003:12.10.14:0



Savannah Division – Building 2 First Floor
8,400 Square Feet

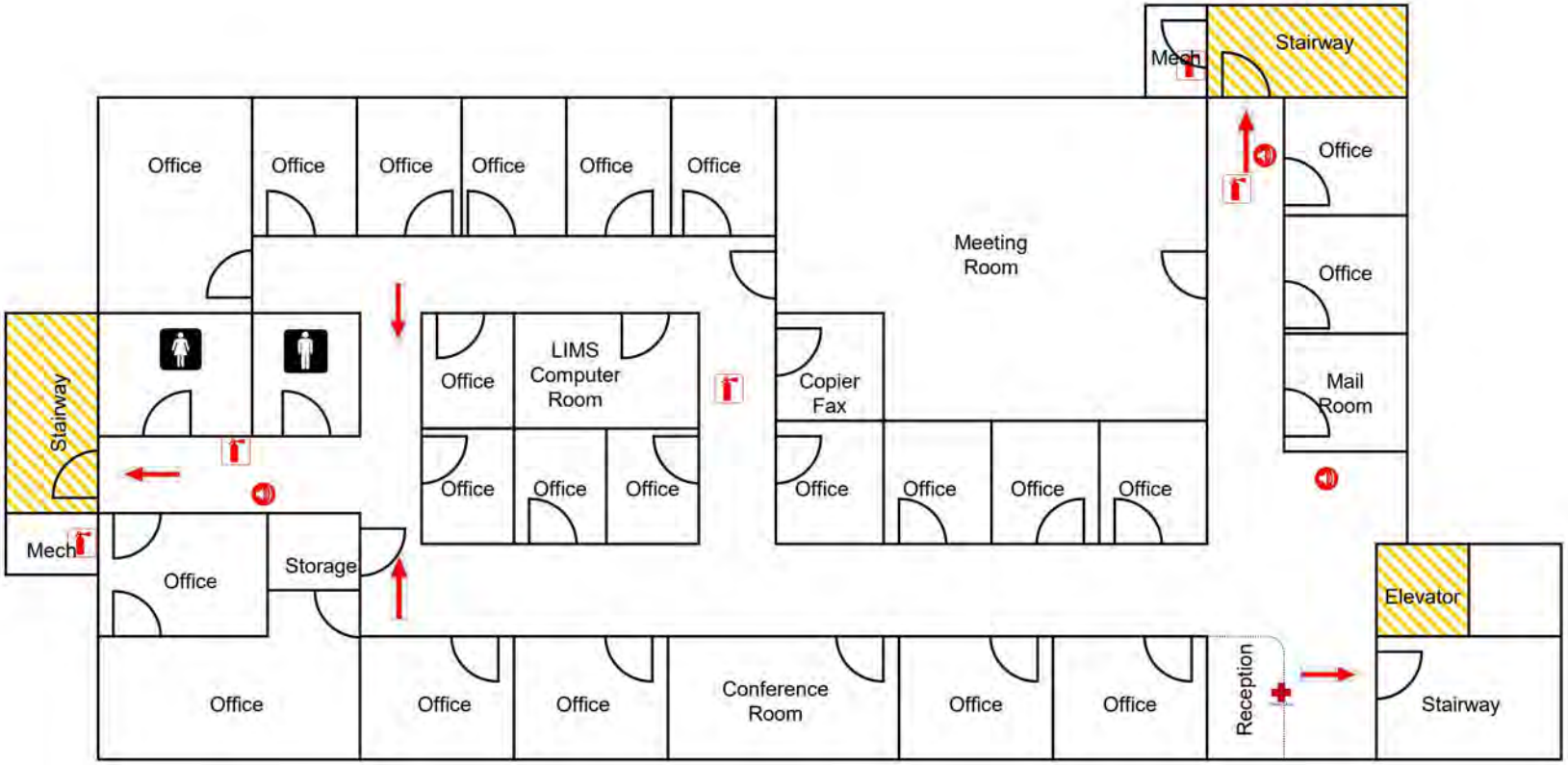
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA004:12.10.14:0



Savannah Division – Building 2 Second Floor
8,400 Square Feet

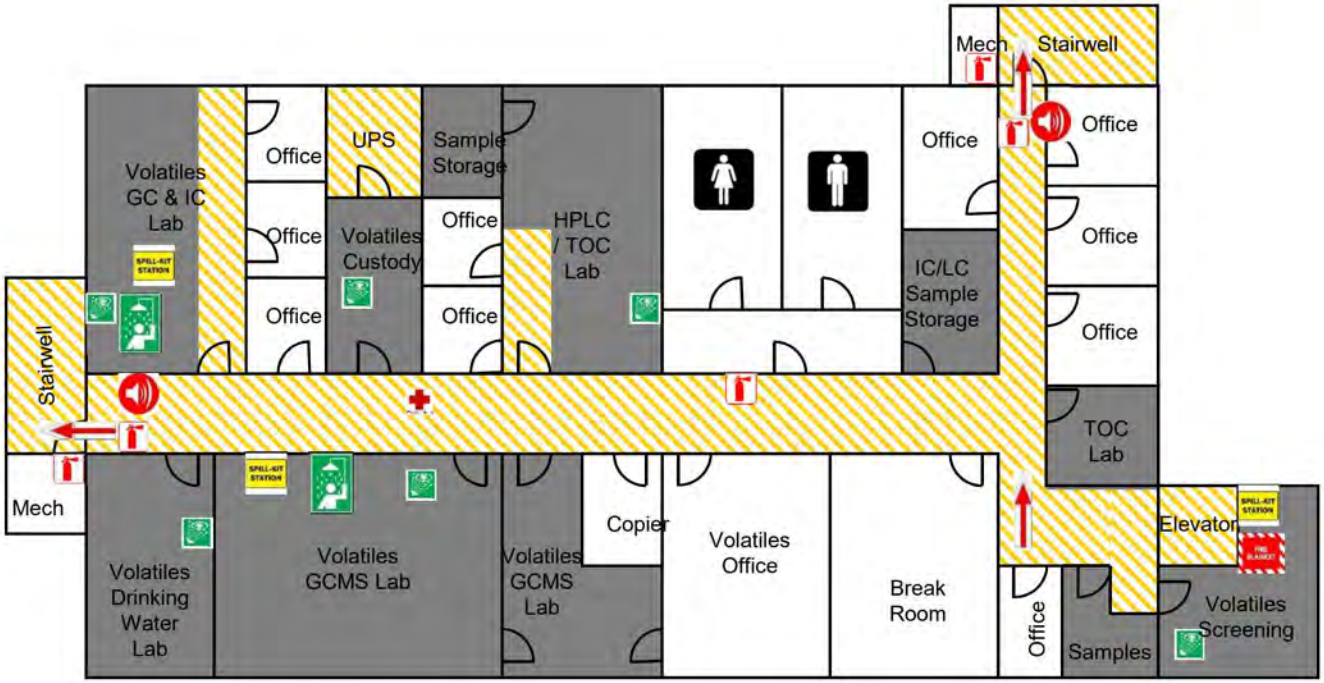
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA005:12.10.14:0



Savannah Division – Building 2 Third Floor
8,400 Square Feet

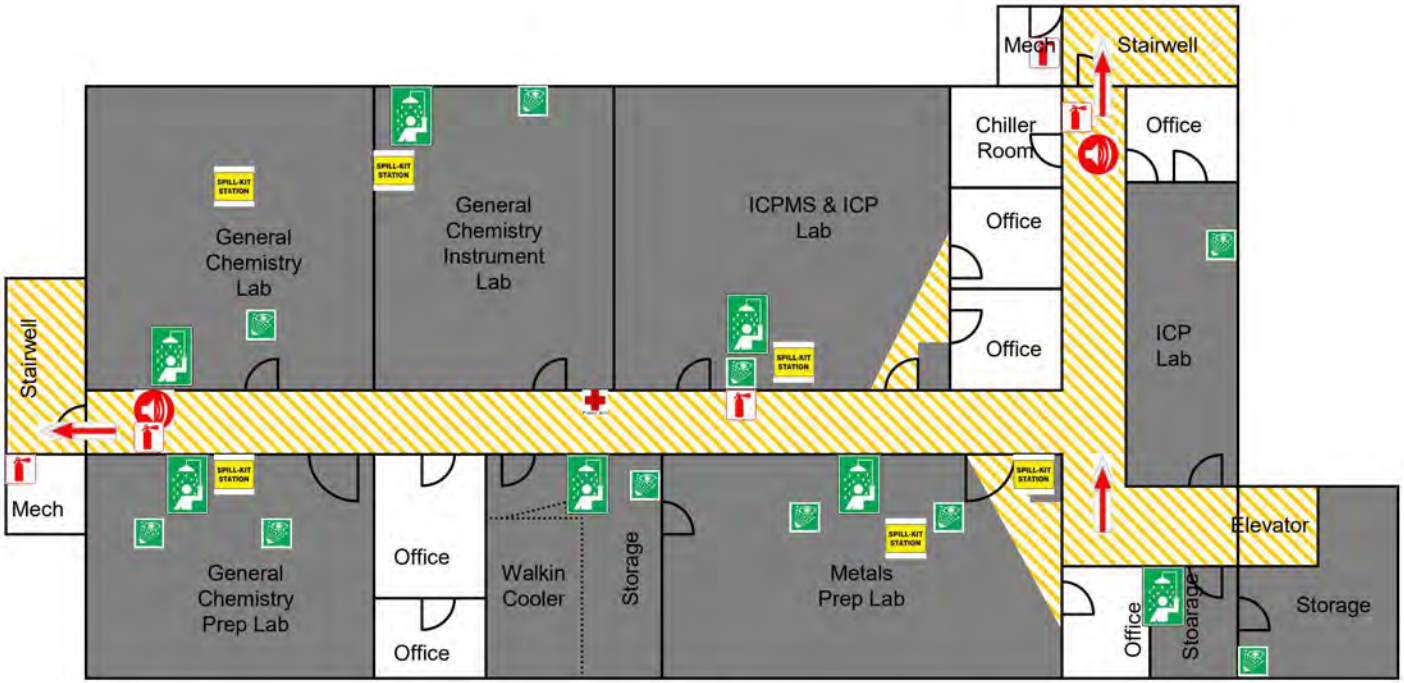
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA006:11.23.15:1



Savannah Division – Building 2 Fourth Floor
8,400 Square Feet

Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA007:12.10.14:0

Signature Page

Addendum to the Environmental Health and Safety Manual Approval Signatures

Approvals (Signature/Date):

Whitney Palefsky June 15, 2018
Whitney Palefsky Date
Environmental Health & Safety Coordinator
Quality Assurance Manager

Bernard Kirkland June 15, 2018
Bernard Kirkland Date
Laboratory Director

Addendum to the Environmental Health and Safety Manual

Table of Contents

Section 1: STOP WORK AUTHORITY

If circumstances occur on the premises that may endanger the health of the employee or others, any employee may stop the work that is being conducted. The employee must then notify the Environmental Health and Safety Coordinator (EHSC), QA Manager and/or Department Manager of the problem in order to initiate corrective action.

Examples of health risks that would warrant such an action are fume hood failures, fires, acid or solvent spills, mercury or other hazardous chemical spills, and identification of samples that could potentially pose an immediate, acute hazard or that will require special precautions during processing (e.g., noxious samples with very strong odor, "hot" samples with very high concentrations of target and/or non-target analytes, etc.).

Any instance in which work is stopped due to a safety issue requires formal investigation by the EHSC. The EHSC will complete the appropriate documentation of the incident. The EHSC is responsible for determining when and how to resume work after work has been formally stopped due to a safety hazard.

The following steps will be taken to address samples for which a Stop Work Order has been initiated:

- The Project Manager (PM) must immediately (within 2 hours) contact the EHSC, all Supervisors, and the Safety Committee to inform them about the sample(s) and the identified issue.
- Each Supervisor and Safety Committee Member must immediately notify each member of their department.
- Each Supervisor and Safety Committee Member must immediately locate all samples/containers pertaining to the Stop Work Order, within their department, and move these samples/containers to the appropriate "Stop Work Order" sample storage location, if possible.
    - Note: The sample storage location must be changed within TALS to reflect the current/correct location via the laboratory's routine Internal Chain of Custody (i.e., sample scanning) procedures.
- The PM and EHSC must follow up with each department (within 2 hours) to ensure all affected samples/containers have been re-located.
- The EHSC, PM, and Technical Management will determine how to proceed with processing the sample, if possible, or if sample processing needs to halt.
    - Client input may be required to assist with further sample classification, etc.., and to determine if special procedures are needed for sample disposal.

Section 2: FACILITY FLOOR PLANS

Refer to the attached floor plans for identification of facility areas, evacuation routes, and the location of emergency equipment.

Facility areas have been defined as follows:

Laboratory Areas:
Laboratory areas are areas where laboratory and/or sample receipt procedures take place. Due to the nature of the materials handled and/or stored in these areas (e.g., samples, extracts, digestates, standards, and reagents), PPE is required at all times.

Note: These areas are shaded in dark gray on the floor plan.

Dual Use Areas:
Dual Use areas are pathways/hallways that lead from non-laboratory areas to laboratory areas and vice versa. Employees may carry containers of food, drink, tobacco products, and/or cosmetics through these areas. These containers should be sealed while in these areas and must not be stored in these areas.

Additionally, employees may carry sealed laboratory materials through these areas. PPE is required in these situations.

Note: These areas are shaded with gray hashmarks on the floor plan.

Non-laboratory Areas:
Non-laboratory areas are areas outside the laboratory where only administrative and/or non-laboratory procedures take place (e.g., employee offices, break rooms, bathrooms, etc.). PPE is not permitted in these areas.

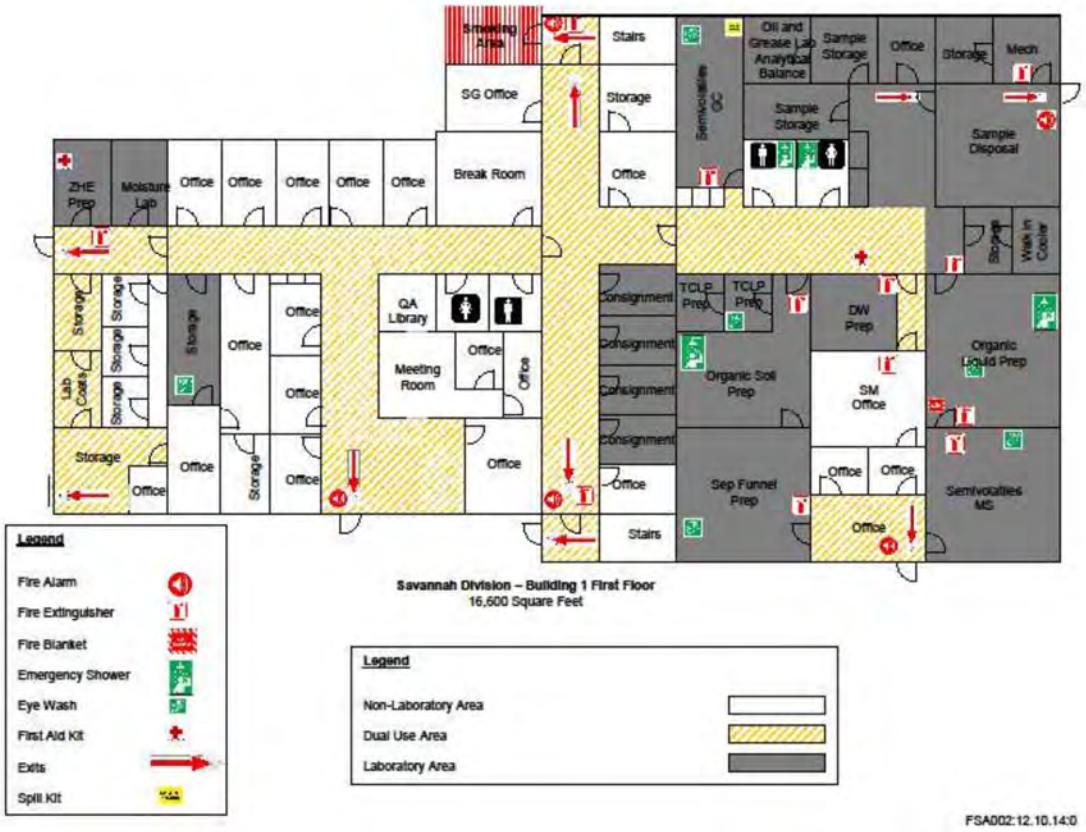
Note: These areas are unshaded (i.e., white) on the floor plan.

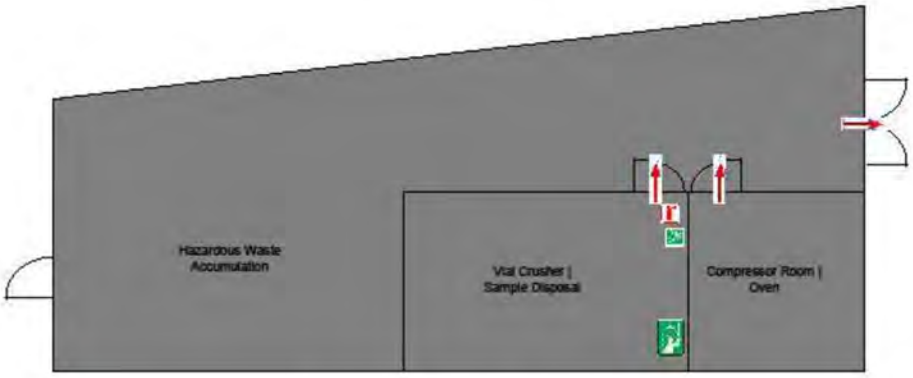
Note: The entire building has been designated as a smoke-free facility; therefore, smoking is not permitted within the building. **Smoking is only permitted on the concrete patio area behind the Building One break room.** Cigarette butts are to only be disposed of in the designated buckets. Cigarette butts require extinguishing prior to being placed in the receptacle. No trash of any kind may be disposed of in these buckets.

The following table summarizes the requirements for each area.

Area	Lab Coat[1]	Eye Protection	Gloves	Open-toe Shoes	Laboratory Materials	Food & Drink
Laboratory Area	Required at all times	Required at all times	Required when working with laboratory materials	Prohibited	Permitted	Prohibited
Dual Use Area	Required when working with or transporting laboratory materials	Required when working with or transporting laboratory materials	Required when working with or transporting laboratory materials	Prohibited when working with or transporting laboratory materials	Permitted[3]	Permitted[4]
Non-Laboratory Area	Prohibited[2]	Permitted	Prohibited	Permitted	Prohibited	Permitted

[1]Lab apron with lab-issued long sleeve shirt may be worn in lieu of lab coat.
[2]Blue (i.e., Visitor) lab coat is permitted in this area.
[3]Laboratory materials must be sealed and must not be stored in this area.
[4]Food and drink should be covered, and must not be consumed or stored in this area.


Smoking Area
Stairs
SG Office
Storage
Office
Break Room
ZHE Prep
Moisture Lab
Office
Office
Office
Office
Office
Semivolatiles GC
Oil and Grease Lab Analytical Balance
Sample Storage
Office
Storage
Mech
Sample Storage
Sample Disposal
Storage
Walk In Cooler
Consignment
Consignment
Consignment
Consignment
TCLP Prep
TCLP Prep
DW Prep
Organic Liquid Prep
Organic Soil Prep
SM Office
Sep Funnel Prep
Office
Office
Semivolatiles MS
Office
Stairs
QA Library
Meeting Room
Office
Office
Storage
Storage
Storage
Storage
Lab Coats
Storage
Office
Office
Office
Storage
Office
Storage
Office
Office
Legend
Fire Alarm
Fire Extinguisher
Fire Blanket
Emergency Shower
Eye Wash
First Aid Kit
Exits
Spill Kit
Savannah Division – Building 1 First Floor
16,600 Square Feet
Legend
Non-Laboratory Area
Dual Use Area
Laboratory Area
FSA002:12.10.14:0



Legend

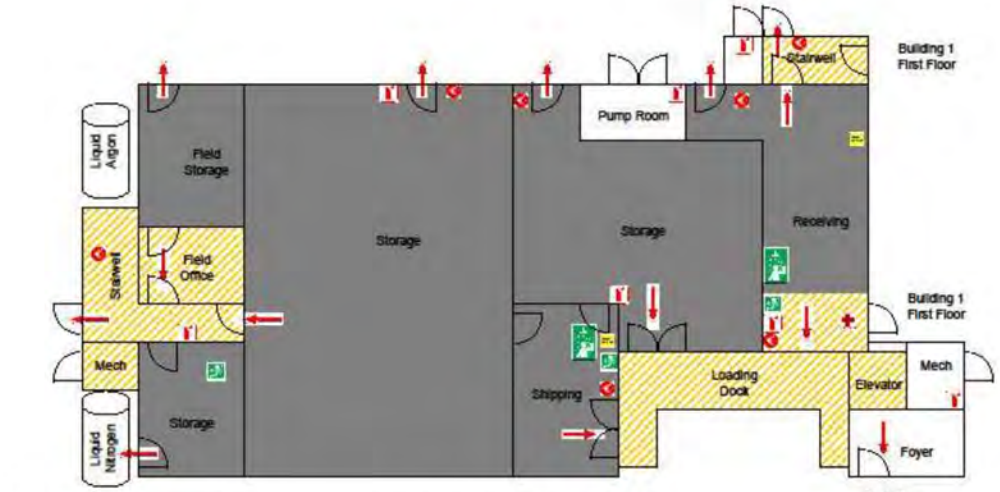
- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Savannah Division – Building 1 Waste Disposal

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA001:12.10.14:0



Savannah Division – Building 2 First Floor
8,400 Square Feet

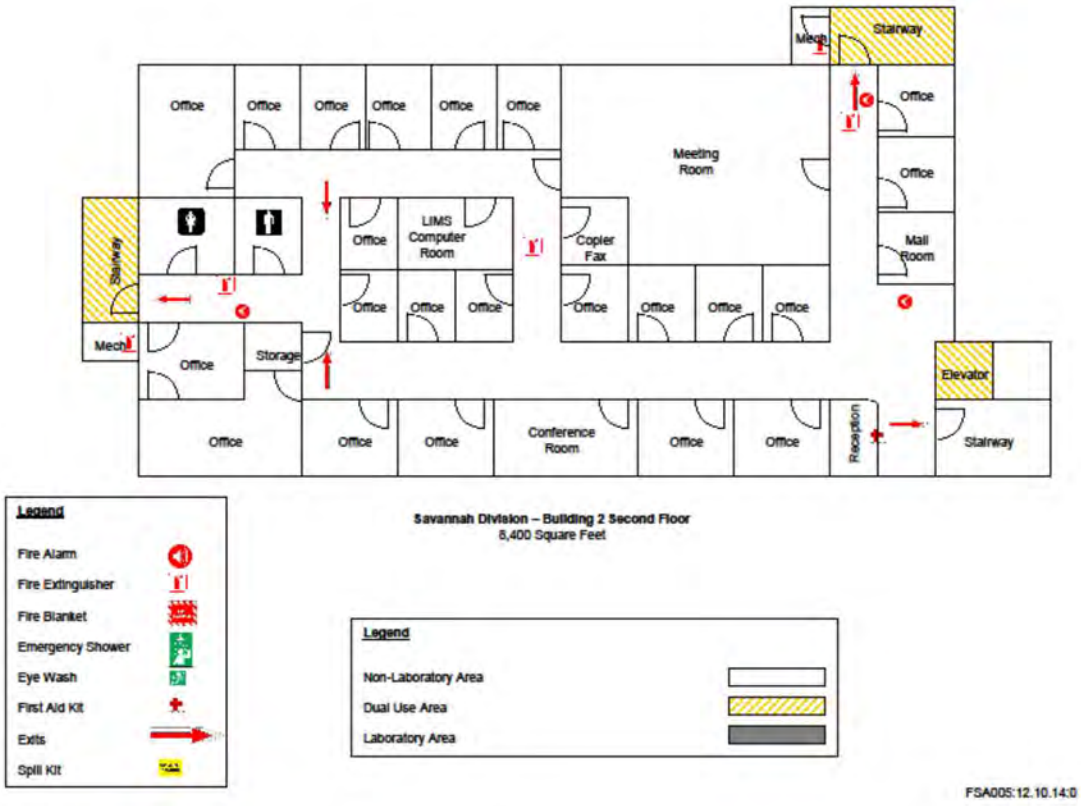
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA004:12.10.14:0


Mech
Stairway
Office
Office
Office
Office
Office
Office
Meeting Room
Office
Office
Mail Room
Stairway
Office
LIMS Computer Room
Copier Fax
Office
Office
Office
Office
Office
Office
Office
Mech
Office
Storage
Elevator
Office
Office
Office
Conference Room
Office
Office
Reception
Stairway
Legend
Fire Alarm
Fire Extinguisher
Fire Blanket
Emergency Shower
Eye Wash
First Aid Kit
Exits
Spill Kit
Savannah Division – Building 2 Second Floor
5,400 Square Feet
Legend
Non-Laboratory Area
Dual Use Area
Laboratory Area
FSA005:12.10.14:0

Savannah Division – Building 2 Third Floor
6,400 Square Feet

Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA006:11.23.15:1

Section 3: Emergency & Laboratory Closure Procedures

The following types of emergencies could occur at TestAmerica Savannah and could result in laboratory closure:

- Accidents, illnesses, and near misses
- Fires
- Exposures including inhalation, skin contact, eye contact, ingestion, and injection
- Ventilation failures and power outages
- Spills of hazardous materials
- Severe weather (e.g., thunderstorms, floods, tornados, tropical storms, and hurricanes)
- Violence and disturbances in the work place

Personnel may use the intercom system, the fire alarm pull boxes, or the Work Alone buttons to notify the other personnel in the building of an emergency. With the exception of the information listed in this section, employees will follow the procedures outlined in Section 7 of the Corporate Environmental Health and Safety Manual (EHSM).

Procedures for Inclement Weather

During a power outage, emergency lights will activate and indicate exit pathways. Employees should assemble in lighted areas (i.e., those near windows).

During a tornado warning, employees must stop work and proceed to a safe location. First floor, interior rooms are preferred. Doors should be closed for any offices with windows, and employees should avoid hallways leading to outside entrances. If possible, all employees should go to the designated assembly area (i.e., the first floor breakroom and first floor hallways) as this area is on the ground floor and away from windows.

Procedures for Laboratory Evacuation

Figure 1 (Section 3A) represents the parking lot and lists the assembly areas for each section of the laboratory. These assembly areas will be used for every evacuation of the building and every evacuation drill. The assembly areas in Figure 1 are approximate locations. The actual locations of the assembly areas are designated with the use of metal signs attached to the fence. Each person must assemble in the appropriate area and check in with their department's Safety Committee member. During an evacuation, the Visitor's Log is removed by designated personnel and the back gate is unlocked by employees in the sample receipt and shipping area to allow egress from the property in case the primary exit is blocked.

There is an "InfoBox" on the back fence (near the FD assembly area) which contains facility-specific information (e.g., Emergency Contact Listing, Employee List, etc.). The EHSC or designee will confirm, through the use of the Employee List and the Visitor's Log, that all employees and visitors are accounted for. This will ensure that the building is completely evacuated.

When the emergency or the drill is over, the Laboratory Director or designee will decide when employees may return to the building.

Procedures for Laboratory Closure

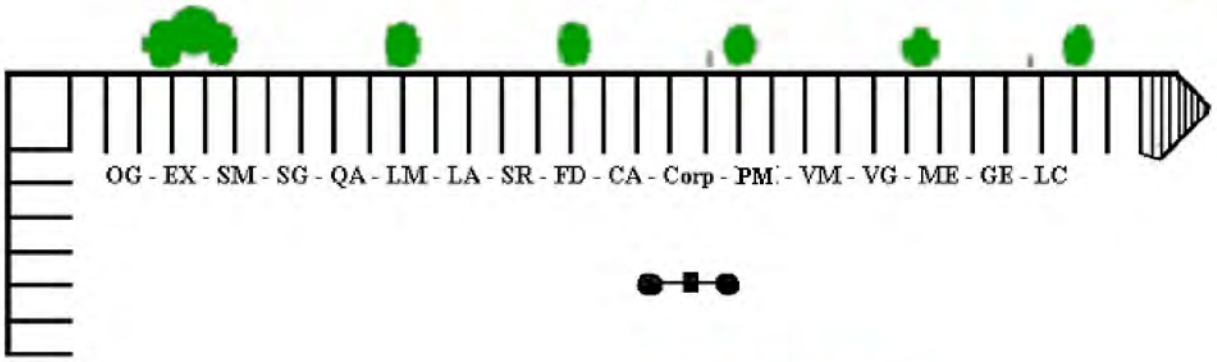
The laboratory should be closed during any mandatory local or statewide evacuations (ordered by the local or state Emergency Management Association), severe local flooding, or other reasons as determined by senior management. The laboratory facility will reopen as soon as is practical after the danger has passed and/or evacuation orders are lifted.

The Laboratory Director or designee will identify who is responsible for each action item on the Laboratory Closure Preparation Schedule (Section 3e) and assign a date and time when each action item should be completed. Additionally, a current list of emergency phone numbers (Section 3d) should be distributed to all employees as soon as a laboratory closure seems likely.

All employees are expected to stay in contact with TestAmerica during a laboratory closure and return to work as soon as is practical. Project Managers should stay in contact with clients by retrieving messages and returning calls daily.

Section 3a: LABORATORY EVACUATION ASSEMBLY AREAS

Figure 1



Section 3b: EMERGENCY CONTACT TELEPHONE NUMBER POSTING

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TESTAMERICA SAVANNAH EMERGENCY TELEPHONE NUMBERS
911 – Emergency, Fire, Ambulance
311 - Utilities

Provider	Telephone Number
Southside Fire Department (9 a.m – 5 p.m.)	(912) 354-1011
Savannah-Chatham Metropolitan Police	(912) 652-6500
NOVA Medical Center	(912) 231-7900
Hospital: Candler Hospital	(912) 819-6000
Hospital: Memorial Health University Med. Center	(912) 350-8000
Poison Control Center	1-800-222-1222
Atlanta Gas Light	(877) 427-4321
City of Savannah Utilities*	(912) 651-6460
Georgia Power**	(888) 660-5890
EH&S Director – Ray Frederici	(708) 925-2564
EHS Hotline	(877) 785-7233
Infotrac Emergency Response (Contract # 77722)	(800) 535-5053

*This is the Customer Service number. In the event of an emergency, call 311.
**There are 2 electric accounts associated with our laboratory: one for the old building (acct. # 61120-04016), and one for the new building (acct. # 26050-04011). If calling due to a *partial* power outage, you must specify which account is affected.

Laboratory Contacts	Telephone Number
Facilities Coordinator – Louie Evans*	(912) 272-4625
Laboratory Director – Bernard Kirkland*	(912) 339-0059
EH&SC - Whitney Palefsky*	(912) 695-8788
LC/IC/VO– Steven Proctor	(912) 944-9759
EX/CU – Kim Chamberlain	(912) 507-5885
Project Management – Kathy Smith	(732) 379-2505
QA – Whitney Palefsky	(912) 695-8788
SG/SM– Brad Mullis	(912) 547-1427
TA Atlanta Service Center	(678) 966-9991
TA Ft. Lauderdale Service Center	(954) 809-5580
TA Orlando Service Center	(407) 851-2560
TA Tallahassee Service Center	(850) 878-3994
TA Tampa	(813) 885-7427

*If this person is unavailable, leave a message and call the next person on the list.

FAN106:06.15.18:13

Section 3c: LABORATORY CLOSURE INSTRUCTIONS

All Staff:
Emergency phone numbers for daily instructions: Call the numbers, in the order listed, until you receive instructions:

912-353-1288 - (Employee Message)
813-885-7427 – TestAmerica Tampa (backup if above number is out of service)
850-474-1001 – TestAmerica Pensacola (backup if TestAmerica Tampa is closed)

Project Managers:
During a laboratory closure, call your voicemail daily and contact the designated back-up laboratory for messages from clients. TestAmerica Tampa is the designated back-up laboratory, unless they are also closed. TestAmerica Pensacola is the designated back-up laboratory, if TestAmerica Tampa is also closed.

All Department Managers and Supervisors:
Check voicemail daily. If voicemail is inoperable, call TestAmerica Tampa for messages. TestAmerica Pensacola is the designated back-up laboratory, if TestAmerica Tampa is also closed.

To Check Voice Mail from an Outside Line:
To call your voicemail from an outside phone, dial 912-354-7858. When the recording picks up, press the # key. The recording will ask for your extension. Enter your 4-digit extension. This takes you into the regular voice mail system. Retrieve messages using the normal procedure.

Section 3d: LABORATORY CLOSURE PREPARATION SCHEDULE

ADMINISTRATION

Responsible Party	Action
Bernard Kirkland Back-up: Kimberly Chamberlain	- Notify TestAmerica Tampa (or alternate back-up lab) of potential laboratory closure and forwarded calls. - Notify staff that TestAmerica Savannah may close. Inform staff to call 912-353-1288 for information. Distribute emergency numbers to staff. - Notify the webmaster to post a message on TestAmerica website about closing the lab.
Linda Bashlor Back-up: Norma Tyler	- Change switchboard messages. Client phone calls (912-354-7858): Change greeting to reflect current situation. For example: "TestAmerica Savannah has closed due to a hurricane evacuation. Please do not send samples to our facility while we are closed. If you are in need of immediate assistance you may contact our Tampa division at 813-885-7427." Staff phone calls (912-353-1288): Change greeting to reflect situation. For example: "TestAmerica Savannah has closed due to a hurricane evacuation. Contact your immediate supervisor for details and further instructions."
All PMs All PMAs	- Contact clients (via phone or email) to divert sample shipments to another lab. Provide alternate contact number (TestAmerica Tampa) and describe how PM will stay in contact (voicemail, email, TestAmerica Tampa, etc.).
All PMs	- Review and report all available in-house data. - Check voicemail (and email, if you can access internet) daily. Delete prior messages so disk space does not fill up.
Kimberly Chamberlain Back-up: Louie Evans	- Contact Fed-ex and UPS to coordinate cooler receipt and/or re-routing.
Louie Evans Back-up: Kimberly Chamberlain	- Perform facility preparations. This may include: 1. Securing outdoor drums and equipment. 2. Turning power off for non-essential areas. 3. Securing outside doors with plastic.

OPERATIONS

Responsible Party	Action
All Department Managers and Supervisors	- Notify operations staff of plan to close TestAmerica Savannah and to call 912-353-1288 for message. - Review backlogs and transfer any unanalyzed samples that will likely exceed critical rush TAT or HT to another division. - Perform laboratory preparations. This may include: 1. Elevating electronics and other valuables off floors in first-floor building. 2. Shutting down instruments that are not running samples.

Section 3e: PROCEDURES FOR POWER SHUTDOWN DURING LABORATORY CLOSURE

Laboratory Building

1. TURN OFF main switch in electrical room of old building.

2. TURN OFF both service disconnects in new building electrical room.

   - One is marked MAIN BREAKER
   - One is marked CHILLER MAIN

3. TURN OFF all breakers in the MAIN PANEL Uninterruptible Power Supply (UPS) room 3rd floor of the new building EXCEPT breaker marked U3B.

4. TURN OFF breakers marked 17, 19, and 21 ONLY in panel U3B that is located in the 3rd floor conference room.

Laboratory Instruments

All Departments:

Instrument: Computers
Shutdown Procedure:
1. Close all windows and applications
2. Click on START menu
3. Click on SHUT DOWN
4. Computer will automatically shut itself off

Department: General Chemistry I

Instrument:
Ovens
Shutdown Procedure:
- Turn off power.
- Unplug.

Instrument: Xplorer1
Shutdown Procedure:
-Set the instrument to Standby in the software.
-Shut down the instrument software.
-Power off instrument
-Power off computer.

Instrument: Spectrophotometers and pH meters
Shutdown Procedure:
- Shut down instrument by shutting off power.
- Shut down any computers that are connected to the instruments

Instrument: Mitsubishi Multi-X 2500
Shutdown Procedure:
- Switch off heater button on side of instrument
- Let cool approximately 20 minutes
- Switch off power button

Instrument: BOD AssayPlus
Shutdown Procedure:
- Exit the software
- Power off the computer and the Autosampler

Instrument: Konelab
Shutdown Procedure:
1. To Shutdown the computers from the Main Menu
a) F8
b) F3
c) F8
d) F3
e) Answer Questions
2. To shutdown the instrument: go to the back of the instrument (right side) and turn the knob to “off”.

Instrument: PCTitrate
Shutdown Procedure:
- Exit the software
- Power off the computer and the Autosampler

Department: General Chemistry II

Instrument: CLCK, CLCN, CLCO, CLCP, CLCQ and CLCR (HP)
Shutdown Procedure:
In the instrument control panel, on the bottom right-hand side, click the "Off" button – this will shut down the pump. Close the Chemstation software and type "ok" when prompted to turn off the pump, thermostat, and lamps. The components can now be shut down by pressing the green LED power button on the bottom left of each module. Note that CLCO and CLCP share a computer, and software sessions for each instrument must be shut down individually.

Instrument: ICG, ICH, ICL, ICN and ICK
Shutdown Procedure:
First shut down the computer program – ICG and ICL are controlled by one computer, while ICN, ICH and ICK are controlled by the second computer. In the bottom right-hand corner locate the Chromeleon Server icon, right click and select "Stop Server". Next, open "Chromeleon" (green tab at the bottom). Ctrl Tab to bring up instruments one at a time. In the top middle of the panel it says System – Startup – Shutdown. Press Shutdown. Ctrl Tab to the next instrument and do the same thing. Then, turn the instruments and autosamplers off for each instrument set. The power buttons are located on the back of each module and Autosampler.

Instrument: HP instruments
Shutdown Procedure:
Autosamplers are controlled via the controller box. The power switch is located on the front lower left of the controller box.

Instrument: TOC Shimadzu
Shutdown Procedure:
1. Shut down the instrument by first stopping the run
• If a run is in process, press the stop key
• When the run completes go to "Instrument"
• Then "Standby"
• "Shut Down Instrument"
• Press the "Standby" key
2. Power off the computer and the instrument
3. Shut down the compressed air.

Department: Metals

Instrument: ICP - Varian Software (2)
Shutdown Procedure:
1. Plasma off button (top of computer screen), loosen pump platens and tubing
2. Save and exit any open worksheets
3. Shut down Varian software
4. Computer Shutdown
5. Turn off power switch on front of instrument
6. Turn off chiller (button on front)
7. Shut off gas and water to both instruments
-Argon and water on wall next to elevator
-Nitrogen on back wall behind ICPD

Instrument: ICP/MS (2)
Shutdown Procedure:
1. Click on "abort" to stop instrument sample analysis
2. Select Instrument – Instrument Control. Once the control window opens, select "plasma off". Click "Yes" to Plasma off.
3. Select Vacuum – Vacuum off. Click "Yes" to shut off the vacuum. (It takes approx. 10 minutes to go from standby to shut down)
4. Exit the software and turn off the computer and printer (switches on front of computer and monitor; printer switch on right side of printer)
5. Turn off the instrument (Switch on the lower right front of the instrument)

Instrument: Leeman (Hg analyzer)
Shutdown Procedure:
1. F10 stops analysis
2. Turn power off to the computer (switch on front)
3. Turn power off to lamp and analyzer (two switches on the front of the analyzer)

Instrument: Prep - Ovens (2)/Water Baths (2)
Shutdown Procedure:
1. Turn power off (switches on front of ovens and water baths)

Instrument: Turbidimeters and pH meters
Shutdown Procedure:
- Shut down instrument by shutting off power.
- Shut down any computers that are connected to the instruments

Department: Volatiles

Instrument: GC/MS
Shutdown Procedure:
1. On the instrument computer, schedule the MS to vent. Once vent is complete, proceed to shutting down the components.
2. Shut down ChemStation software. Then power off the computer.
3. Turn off the Archon/Centurion Autosampler power switch located in the back of the instrument.
4. Turn off the Encon Concentrator power switch located in the back.
5. Turn off the GC power switch located in the front or side according to type.
6. Turn off the mass spec power switch located in the front or back according to type. Note: Avoid shutting off the mass spec until certain that the power will be off for an extended time, or the battery back-up is near being exhausted.

Instrument: GC
Shutdown Procedure:
1. On the instrument computer, shut down ChemStation software. Then power off the computer.
2. Turn off the Encon power switch located in the back.
3. Turn off the PID detector power switch located in front of the PID power supply.
4. Turn off the Archon power switch located in the back of the instrument
5. Turn off the GC power switch located on the front.
6. Turn off the Tekmar headspace autosampler on the back.

Department: Semivolatiles Department - GC

Instrument: ECD S, X, J, AA, AD, Z and Y, FID Q and AB
Shutdown Procedure:
Power switch location: Front panel Bottom Left Corner

Department: Semivolatiles Department – GC/MS

Instrument: 6890 Gas Chromatograph with 5973 or 5975 Mass Selective Detector
Shutdown Procedure:
1. . On the instrument computer, schedule the MS to vent. Once vent is complete, proceed to shutting down the components.
2. Power switch located on the bottom left of each instrument.
3. Press to power on or off..
4. Verify that the rough pump is also off..
5. If rough pump is not automatically turned off with the MSD, unplug from power source.

Section 3f: TALS DISASTER RECOVERY PROCEDURES

Implementation of Disaster Recovery Plan

The following individuals and TestAmerica divisions should be notified when the Disaster Recovery Plan is implemented. In addition to the locations and individuals listed below, efforts should be made to contact all decision makers/managers and other key laboratory personnel potentially impacted by the situation.

Bernard Kirkland
Laboratory Director
TestAmerica Savannah
912.354.7858

Todd Baumgartner
VP Operations
TestAmerica Savannah
1.850.556.2113

TestAmerica Pensacola
1.850.474.1001

TestAmerica Tampa
1.813.885.7427

TestAmerica Denver
1.303.736.0100

Disaster Recovery Procedures
The Savannah laboratory accesses most of the data files via CITRIX server housed in Denver. This includes all information within the TestAmerica Laboratory Information Management System (TALS), QA-Drive files, Public_QA-drive files, etc. In the event of a disaster in Savannah, little or no information should be lost.

Section 3g: ARRANGEMENTS WITH LOCAL AUTHORITIES

Letters were sent to the Chatham County Police, the Southside Fire Department, and Candler General Hospital informing them of our intent to use their facilities in the event of an emergency. These letters were dated on March 27th, 2002. Additionally, a copy of this document will be submitted to these entities upon finalization.

Section 4: LOCK OUT / TAG OUT PROCEDURES

An operation is regulated by the lock out / tag out policy when:

- Any employee (or contractor) is required to remove or bypass a guard or other safety device.
- Any employee (or contractor) is required to place any part of his body into the mechanism of a piece of equipment or path of hazardous energy unless the activity is routine, repetitive, and integral to the use of the equipment for production and the operator has been properly trained in the precautionary steps necessary to perform the activity safely or is provided other protection (guarding).

Lock Out Procedures

Equipment will only be locked or tagged out by authorized employees who have been trained in the company's procedure and who are familiar with the specific procedures for the equipment. A list of employees trained on this procedure is included in the Ancillary and Safety Training Matrix. Training must be completed annually.

All affected employees will be notified of the application of the lockout devices and/or tags at the beginning of the lockout procedures.

All energy sources will be identified and are to be locked out. (Energy sources include, but are not limited to, electrical, mechanical, hydraulic, pneumatic, thermal, and chemical sources.) Each employee involved with the operation will place his/her lock on each energy-isolating source. Multiple locks will be attached using hairpin or tong devices. The locks must be applied with a warning tag describing why the equipment is locked out, who placed the lock on the equipment, and the date the lock was installed.

Stored or residual energy must be relieved, disconnected, blanked off, restrained, and otherwise rendered safe. Energy sources subject to re-accumulation, such as capacitors, hydraulic reservoirs, air tanks, steam traps, etc., should be controlled by isolation and locking out. If there is a possibility of re-accumulation of stored energy to a hazardous level, verification of isolation shall be continued until the servicing or maintenance is complete.

When all steps involved with shutdown listed in the specific procedures for equipment have been completed, make sure that all personnel are clear, and attempt to start or activate the equipment to make sure that all energy sources have been locked out. Return controls to "off" position.

Cord- and plug-connected equipment does not require lock out / tag out if the following conditions exist:

- The authorized employee is within sight of the equipment
- Unplugging the equipment isolates the equipment from all energy sources
- The equipment has no stored energy

Tag Out Procedures

Do not use tags alone (without locks) in an energy-isolation procedure unless absolutely necessary. The only exception to this is when the equipment or process does not lend itself to being physically locked out. If this equipment is upgraded or modified so that it becomes possible to lock out the equipment, then the proper lockable switches, fittings, or valves will be added.

Tags are to be used with locks to identify the employee, the hazard, and the date.

Tags must be durable and able to withstand the environment in which they are used.

Tags are to be attached with pull ties and must be secure so that it is readily apparent what the tag is warning about. Alternate methods of attaching tags may be used as long as they are not easily removed or reusable and must withstand 50 pounds unlocking strength. (Rubber bands, wire ties, and string are not permissible means of attachment.)

Any employee, who removes, bypasses, ignores, or otherwise defeats a tag without permission of the authorized person responsible for it, or without proper management approval, may be subject to immediate dismissal. (See procedures for removal of locks and tags.)

Employees must be made aware that tags do not protect against the unexpected energization of the equipment, and that they should be extraordinarily alert around tagged out equipment and systems that are not also locked out.

Steps for Restoration of Equipment and Removal of Locks and Tags

The work area is to be inspected to ensure that all personnel, tools, loose parts, and non-essential items are in a safe position (this step is essential to eliminate the chance of anyone being exposed to unexpected release of hazardous energy) and that guarding is in place. If the equipment is to be brought on-line for set-up or adjustment temporarily without guarding, affected employees must be adequately protected.

All employees who would be affected by the start-up of the equipment must be notified of the removal of the lockout devices before they are removed.

To remove locks or tags from a piece of equipment without the individual who locked it out requires the approval of the laboratory manager or director. The lab manager or director must have verified that the individual who locked out the equipment is not at risk, as well as, the individual who inspected the equipment and determined that it is safe. This procedure is to be done only after every effort has been made to have the individual who locked out the equipment remove his/her lock and tag. Whenever a lock/tag has been removed by someone other than the person who applied it, the person who originally locked out the equipment must be notified before he/she returns to the work area.

Devices Requiring Lock Out/Tag Out

Branch circuits throughout building
Disconnects to chill water pumps
Disconnects to hot water pumps
Disconnects to ah-1 and ah-2
Chiller panel

Section 5: PERSONAL PROTECTIVE EQUIPMENT CLARIFICATION

Laboratory aprons, in conjunction with the long-sleeved laboratory-issued shirts, may be used in lieu of laboratory coats for certain designated procedures (e.g., while conducting sample receipt procedures) if approved by the EHSC and the Laboratory Director.

Section 6: LABORATORY CELL PHONE POLICY

Cell phones are to be used in laboratory areas in emergencies only. Cell phones in the laboratory can be a distraction, an annoyance to employees working in the area, and can cause safety problems. All cell phones should be stored outside of the laboratory areas and the ringers should be set to silence.

Phone calls from outside parties to laboratory personnel should be made through the (912) 353-1288 number.

Section 7: LABORATORY-SPECIFIC SAFETY TRAINING

Initial Safety Training:
All employees must complete the required safety training before being allowed to work in the laboratory. The Environmental Health and Safety Coordinator (EHSC) or designee reviews the items in the New Employee Training Checklist found in Appendix VII of the TestAmerica Environmental Health and Safety Manual (EHSM). The training topics include the EHSM; general safety and work practices; emergency procedures; alarm systems; emergency equipment; access to medical records; accident and near miss reporting; Department of Transportation (DOT) awareness training; detecting the presence or release of hazardous materials; review of the laboratory standard or the general industry hazard communication standard, as appropriate; personal protective equipment requirements; protective measures to prevent or minimize exposure to hazardous chemicals; waste storage, waste handling, and waste disposal procedures; and proper lifting procedures.

The EHSC issues the new employee the required personal protective equipment. This includes a lab coat and safety glasses and/or a prescription form for prescription safety glasses.

After the employee's safety questions and concerns have been addressed, the employee is escorted to the assigned laboratory department. The employee is assigned a mentor to be available to aid in the adjustment to the safety program. The mentor will usually be the Safety Committee member for the department to which the new employee has been assigned.

Continuing Safety Training:
The laboratory has ongoing safety training on a monthly basis. The training covers one to two subjects and may be covered via E-training or during all-staff meetings. Laboratory employees are required to complete all training topics. Administrative employees are required to complete a subset of the training topics. Training is documented using the all-staff roster and/or quizzes or via E-training replies. All training is tracked by the QA Department.

E-Training System and Documentation:
If an employee is unable to attend a monthly training session, the training is accomplished via the E-Training system.

The E-Training system works as follows:
- The QA Department sends an email to all affected employee for each outstanding training topic.
- The email contains the location of the training presentation and quiz and voting buttons.
- The employee must view the training presentation and complete the associated quiz.
- Once the employee has completed the training, he must click on the *"I have viewed the XXXXX presentation"* voting button in the email and forward a copy of the associated quiz to the QA Department.

The acceptance email notice generated by selecting this voting button serves as documentation of the training and is filed in the appropriate training file.

Department Managers are responsible for ensuring their staff has completed necessary safety training prior to the due date.

Training documentation is housed electronically within employee- and/or topic-specific training files. A Training Matrix has been developed to indicate training status as Complete or Overdue and is housed on the Public_QA drive.

Section 7a: LIST OF QUALIFIED SAFETY TRAINERS

Name	Training Topics	Qualifications
Environmental Health and Safety Coordinator	Initial safety training, Refresher training	More than 15 years experience as an environmental scientist. Trained on corporate courses including Hazardous Waste Management and DOT HAZMAT Management.
Waste Coordinator	Initial safety training, Refresher training	Trained on corporate courses including Hazardous Waste Management and DOT HAZMAT Management.
Quality Assurance Manager	Initial safety training, Refresher training	More than 15 years experience as an environmental scientist. Trained on corporate courses including Hazardous Waste Management and DOT HAZMAT Management.
Department Managers / Supervisors / Lead Analysts	New Laboratory Employee Mentoring	The Department Managers, Supervisors, and lead analysts have completed the monthly training courses and are familiar with the procedures and associated hazards the new employee may encounter.
Safety Committee Members	New Administrative Employee Mentoring	The safety committee members have completed the monthly training courses and are familiar with the procedures and associated hazards the new employee may encounter.

Section 8: PROCEDURES FOR WORKING ALONE

TestAmerica Savannah maintains 2 tiers of activities with regards to working alone procedures:

1) Restricted Activities

Restricted Activities are those that could pose a moderate risk to employee safety. Restricted Activities include:

- Metals Digestions
- TKN Digestions
- Cyanide Digestions
- Sulfide Digestions
- Solvent Evaporation and Exchange (i.e., EX Preparations and Concentrations)
- Field Activities
- Sulfuric Acid Clean-ups
- Waste Disposal

Employees must employ additional safety measures (i.e., use of the buddy system) when performing Restricted Activities. Buddies must be formally assigned and must adhere to a strict schedule of checking in with each other (i.e., at least once every half hour).

3) General Activities

General Activities are those that pose a minimal risk to employee safety. General Activities are typically administrative activities (such as preparing reports and reviewing data) and all other laboratory activities excluding those defined above. Employees are permitted to work alone when performing General Activities.

The laboratory has been divided into six zones to classify whether an employee is considered to be working alone. These zones are as follows:

Zone	Building	Floor	Department	Activity Tiers
1	Old	1 & 2	Corp	General[1]
2	Old	1	EX/SG/SM	General[1] Restricted[2]
3	New	1 & 2	PM/SR/Admin	General[1]
4	New	3	VO	General[1]
5	New	4	ME & GE	General[1] Restricted[2]
6	N/A	N/A	Field	Restricted[3]

[1]Employees may work alone when performing these activities.
[2]Employees may work alone when performing these activities, if another employee is working in their zone and the buddy system is employed.
[3]Employees may work alone when performing these activities, if another employee is working in their zone and/or the buddy system is employed. See below for more information on field-specific procedures.

Field personnel:
Field personnel are equipped with a cell phone to maintain communication with the home laboratory. The laboratory keeps a copy of the sampling schedule. If changes are needed, the sampling crew can be reached by the use of a cell phone.

When the field personnel go to a site that has a site safety plan, they must comply with the requirements of that plan. If the site does not have a site-specific safety plan or the site-specific plan does not address evacuations, then the personnel must, at a minimum, familiarize themselves with the proper emergency evacuation routes.

In Case of Emergency (ICE) Buttons

In addition to following the Activity Tier and Work Zone requirements outlined above, all employees working non-routine business hours and/or working alone are required to enter the building through the main entrance on the first floor.

The employee must obtain an In Case Of Emergency (ICE) Button - also called a Work Alone Button. Employees must write their name beside the appropriate spot on the sign-out board, and move the associated magnet on the floor plan map to the area where they will be working. The ICE button is tied to the laboratory's alarm system and should be activated in case of an emergency.

When an ICE button is activated, an audible alarm will sound throughout the building, yellow strobes will flash, and TYCO is automatically notified. TYCO is responsible for notifying the police department who will use the sign-out board/floor plan to help identify where in the building the emergency may be located.

Note: The audible alarm and the visual strobe of the ICE system are different than the alarm and the strobe of the fire alarm system. If you are also in the building when another employee activates the ICE alarm, you should make your way to the front entrance to determine who else is in the building. You should then try to determine if you can help the employee in any way.

False Alarms:

If a button is inadvertently activated, TYCO will need to be notified of the false alarm. In order to notify TYCO of a false alarm, call TYCO at 1-800-289-2647. The TYCO operator may request the following information: account phone number (912-354-7858); the name and address of the company (TestAmerica Savannah, 5102 LaRoche Avenue); the customer number (109211223); and the cancellation code (1975). Once the false alarm has been reported, the alarm system must be reset.

To Reset the Alarm

Go to the alarm keypad, type in the code "972105", press "Reset" then "Alarms". Wait to see if the alarms reset. Press "Continue" then "Quit". This procedure will reset all of the alarms points in the building. If the keypad is locked up you will have to contact TYCO to get them to reset the keypad. Alarm keypads are located on the first floor (old building) at the main entrance and the second floor (new building) at the reception desk.

Section 9: CARCINOGENS

Most areas of the laboratory contain standards or reagents that at some concentration are considered carcinogens. Section 10.7 of the Environmental Health and Safety Manual contains information on carcinogens, reproductive toxins, and other highly toxic substances. Appendix XII contains a list of the carcinogens that are likely to be present at a TestAmerica laboratory location.

Most of the carcinogenic compounds that are present in the laboratory are in the form of standards that contain 10,000ppm or less of the substance in question. These standards are then diluted to concentrations well below the OSHA threshold limits. These materials should not pose a health hazard at the diluted levels as long as they are properly handled.

There are, however, known carcinogens that are present at percentage levels and even pure product. Some of the substances listed in Appendix VII that are present in higher concentrations at the Savannah location are:

Arsenic Compounds
Chloroform
Hexavalent Chromium
Lead Compounds
Methylene Chloride
Mineral Oils
Nickel Compounds
Phenolphthalein
Potassium Dichromate
Silica, Crystalline (silica gel)
Sulfuric Acid

It is important to remember that carcinogens do not cause cancer in every case, all of the time. Different substances have different levels of cancer causing potential. The type of exposure, frequency of exposure, and length and intensity of exposure are all factors in determining the cancer risk. This is another example of why it is important to be familiar with the information contained in the respective SOPs and Safety Data Sheets. It is important to know the risks and to know how to safely handle the required materials.

The specific laboratory areas in the Savannah facility where carcinogens (in standards or reagents) are handled are as follows:

GC and GC/MS Volatile Laboratory
GC and GC/MS Semivolatile Laboratory
Metals Instrument Laboratories – ICP, ICPMS/Hg, and Digestion areas
Organic Extractions Laboratory (Liquid and Solids)
Wet Chemistry Laboratories – Prep, Instrument, and BOD/TOX/Solids

It should be noted that all TestAmerica employees receive carcinogen awareness and safe handling training on an annual basis.

Section 10: Walkthroughs and Inspections

10.1 Quarterly Walkthrough

At a minimum frequency of once per quarter the EHSC or designee will perform a walkthrough of all laboratory areas, waste areas, storage areas, and exterior areas of the Savannah location. This inspection is designed to test compliance with all aspects of the safety program. Figure 10.1 is an example of a checklist used during the walkthrough. When the inspection is complete the final report is sent to the Laboratory Director for acknowledgment and approval. An electronic acknowledgement is stored when the Laboratory Director has completed his review. The report is sent to the Department Managers, Maintenance personnel, and any other personnel that are involved with the corrective actions.

10.2 Weekly Inspections

On a weekly basis, all eyewashes and safety showers will be activated to test that they are performing acceptably and to assure that fresh water is flushed through the system.

10.3 Monthly Inspections

On a monthly basis, all fire extinguishers are checked to ensure that they are properly charged and that they are in good condition.

Figure 10.1

Room	Dept	Location	Auditor	Date Audited	Item / Area	Eval	Score	Possible Points	Comments	Required Corrective Action	Corrective Action Assigned To	Date Corrective Action Due	CAR Status	CAR Verified
1	GE1	BOD Lab			Entrances, Exits, Aisles, Hallways Clear									
1	GE1	BOD Lab			Fire Extinguishers / Inspection Current	N/A	N/A							
1	GE1	BOD Lab			Fume Hoods Operating / Inspection Current									
1	GE1	BOD Lab			General Safety (eye protection, lab coats, etc.)									
1	GE1	BOD Lab			Hazardous Chemicals: Labeled Correctly									
1	GE1	BOD Lab			Hazardous Chemicals: Stored Correctly									
1	GE1	BOD Lab			Housekeeping: All Containers Labeled Properly									
1	GE1	BOD Lab			Housekeeping: Floors Clean and Uncluttered									
1	GE1	BOD Lab			Housekeeping: No Chipped or Cracked Glassware in Use									
1	GE1	BOD Lab			Housekeeping: No Containers Left Open									
1	GE1	BOD Lab			Housekeeping: No Electrical Cords Exposed on Floors									
1	GE1	BOD Lab			Housekeeping: Safety Showers/Eyewash stations have Floor Clearance									
1	GE1	BOD Lab			Other	N/A	N/A							
1	GE1	BOD Lab			Other	N/A	N/A							
1	GE1	BOD Lab			Other	N/A	N/A							
1	GE1	BOD Lab			Other	N/A	N/A							
1	GE1	BOD Lab			Pressurized Cylinders: Chained or Strapped									
1	GE1	BOD Lab			Showers / Eye Wash Inspection Current									
1	GE1	BOD Lab			Spill kits									

Section 11: REPORT GENERATION AND RETENTION – Document Location Matrix

Category	Documents	Media	Location
OSHA/Safety	EHSM	E	Public_QA Drive QA-Drive / Safety / EHS Manual
OSHA/Safety	EHSM Addendum	E	Public_QA Drive QA-Drive / Safety / EHS Manual Addendum
OSHA/Safety	Area Safety Analyses	E	QA-Drive / Safety / Area Safety Analyses
OSHA/Safety	Contractor Communication Forms	E	QA-Drive / Safety / Contractor Communication Forms
OSHA/Safety	Standard Operating Procedures	E	Public_QA Drive QA-Drive / Document Management / SOPs
Incident Data	Incident Reports	E	Velocity System QA-Drive / Safety / Accidents
Incident Data	Supporting Documentation	E	Velocity System QA-Drive / Safety / Accidents
Incident Data	Doctor's Reports	E	Velocity System QA-Drive / Safety / Accidents
Incident Data	Monitoring Data	E	Velocity System QA-Drive / Safety / Accidents
Incident Data	Corrective Action Documentation	E	Velocity System QA-Drive / Safety / Accidents
Training: EHSM Section 4.7	Orientation Training Forms	E	QA-Drive/Training/Training Documentation Files/Last name, First name/Safety Training
Training: EHSM Section 4.7	Orientation Follow-up Exams	E	QA-Drive/Training/Training Documentation Files/Last name, First name/Safety Training
Training: EHSM Section 4.7	Documentation demonstrating that monthly training was carried out each month.	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	The annual review of key elements of the EHSM not covered during other training sessions	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Changes to EHSM	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Hazard Communication / Laboratory Standard	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Emergency Procedures	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Fire Extinguishers	E	QA-Drive / Safety / Training / Local Training Documentation

Category	Documents	Media	Location
Training: EHSM Section 4.7	Hazardous Material Use & Storage	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	PPE Requirements	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Handling Glassware	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Lifting and Moving Materials	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Ergonomics Training	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Procedures for Managing waste at SAAs	E	QA-Drive / Safety / Training / Local Training Documentation
Training: EHSM Section 4.7	Annual Ergonomics Assessments	E	QA-Drive / Safety / Training / Local Training Documentation
Training: Specialized Topics	Incident investigation training	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	Area Safety Analysis Training	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	HAZWOP/ Emergency Responder training	E	QA-Drive / Safety / Training / Corporate Training
Training:Specialized Topics	Training for individuals who are authorized to process and ship hazardous waste	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	Training for individuals who are authorized to ship dangerous goods under the DOT regulations	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	30-Hour OSHA certifications	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	First Aid/CPR	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	Electrical Safety	E	QA-Drive / Safety / Training / Corporate Training
Training: Specialized Topics	Defensive Driving	E	QA-Drive / Safety / Training / Corporate Training
Inspections	Quarterly EHSC Inspections	E	QA-Drive / Safety / Audits
Inspections	Inspection forms prepared by the management team when they visit operations	E	EHSC Office – Safety File drawer – folder labeled “Management Inspections”
Inspections	Regulatory inspections	E	QA-Drive / Safety / Audits
Inspections	Safety committee minutes	E	Public_QA Drive / Safety / Safety Committee Minutes
Monitoring Results	Air monitoring results	E	QA-Drive / Safety / Air Monitoring
Monitoring Results	Wipe tests of work surfaces for non radioactive materials	NA	NA
Monitoring Results	ECD Wipe Tests	E	QA-Drive / Safety / ECD Wipe Tests
Monitoring Results	Noise Measurements	E	QA-Drive / Safety / Noise Testing

Category	Documents	Media	Location
Monitoring Results	Medical Monitoring Data	E	QA-Drive / Safety / Air Monitoring
Monitoring Results	Fume Hood Inspections	HC	Facility Manager's Office
Emergency Equipment	Fire Alarm and Alarm System	HC	Facility Manager's Office
Emergency Equipment	Fire Extinguisher Inspections	HC	On each extinguisher (Archived labels in Facility Manager's Office)
Emergency Equipment	Eyewash Inspections	HC	On each eyewash (Archived labels in Facility Manager's Office)
Emergency Equipment	Safety Shower Inspections	HC	On each safety shower (Archived labels in Facility Manager's Office)
Emergency Equipment	First Aid Kit Inspections	E	QA-Drive / Safety / Audits
Emergency Equipment	Emergency drill records	E	QA-Drive / Safety / Evacuation Drill
Environmental Compliance Records	Air emission records	NA	NA
Environmental Compliance Records	Water discharge permits and test records showing compliance with the permits	NA	NA
Environmental Compliance Records	Any other permits and any associated tests	NA	NA
Environmental Compliance Records	Waste training records	E	QA-Drive / Safety / Training / Corporate Training
Environmental Compliance Records	EPA waste generator numbers	E	EHSM addendum
Environmental Compliance Records	Waste Manifests	HC E	QA Library QA-Drive / Safety / Waste Disposal
Environmental Compliance Records	Land Disposal Restriction Notifications	HC E	QA Library QA-Drive / Safety / Waste Disposal
Environmental Compliance Records	Certificates of destruction for waste sent off site for disposal	HC E	QA Library QA-Drive / Safety / Waste Disposal
Environmental Compliance Records	Tracking logs for waste shipped off site or treated on site	HC E	QA Library QA-Drive / Safety / Waste Disposal
Environmental Compliance Records	Inspection records for the main waste accumulation areas	HC	Data Storage Room

Category	Documents	Media	Location
Environmental Compliance Records	Biennial or other reports required by state authorities	E	QA-Drive / Safety / Biennial Reports
Environmental Compliance Records	Procedures for processing waste	E	EHSM Addendum
Environmental Compliance Records	Waste minimization plans	E	EHSM Addendum
Environmental Compliance Records	Training records for employees who are authorized to package and ship dangerous materials.	E	QA-Drive / Safety / Training / Corporate Training
Environmental Compliance Records	DOT security plans	E	EHSM
Environmental Compliance Records	Shipping records for dangerous goods	NA	NA
Environmental Compliance Records	Procedures for preparing and shipping sample kits under the exempt small quantity rule	E	SOP SA-CU-015
Environmental Compliance Records	Test results showing coolers that are packed comply with exempt small quantity rules	E	QA-Drive / Safety / Cooler Drop Tests
NRC Regulations	Licenses	NA	NA
NRC Regulations	Monitoring results for employees who might be exposed to radioactive materials	NA	NA
NRC Regulations	Wipe test results including ECD tests	E	QA-Drive / Safety / ECD Wipe Tests
Other Potential Records	Internal audit reports and related documents.	E	QA-Drive / Safety / Audits
Other Potential Records	External audits	E	QA-Drive / Safety / Audits
Other Potential Records	Records for visits from outside regulatory agencies including any reports, citations and fines.	E	QA-Drive / Safety / Audits

Section 12: RESTRICTED FOREIGN AND DOMESTIC SOILS

12.1 Overview

12.1.1 The United States Department of Agriculture (USDA) has imposed restrictions on the movement of foreign soil into the United States to prevent the human-assisted spread of agricultural organisms and pests such as imported fire ant, golden nematode, and Mexican fruit fly. To perform analyses on soil samples originating outside the United States, a laboratory representative must sign the USDA Animal and Plant Health Inspection Service (APHIS) Plant Protection Quarantine (PPQ) Compliance Agreement and obtain a permit to receive foreign soil.

12.1.2 The USDA also regulates domestic soil from some regulated (quarantined) areas of the United States to other unregulated domestic areas. Savannah is located within a regulated area; therefore, shipments of domestic soils into Savannah should not be affected. It should be noted, however, that shipments of soil outside of Savannah (to a subcontract lab, for instance) could fall under these regulations.

12.1.3 TestAmerica Savannah currently maintains a permit from the USDA to receive soil samples from foreign countries or territories outside the fifty U.S. states. A copy of the USDA Soil Permit and Compliance Agreement is located on the laboratory's public access drive (i.e., the Public_QA-Drive). These licenses are valid for a period of seven years. These licenses are issued to a single person and are non-transferable. Copies of these documents are housed in the Certification Folder on the laboratory's public access drive (i.e., the Public_QA-Drive).

Note: If the person designated on the permit as the responsible party leaves TestAmerica's Savannah laboratory, then the local USDA office must be notified promptly, and the Soil Permit and Compliance Agreement must be re-issued.

12.2 Pre-Arrival and Container Preparation Procedures

The laboratory must inform shippers to address the soil shipment exactly as it is written on the Soil Permit and the Compliance Agreement. All shipments of foreign soils must be shipped by bonded courier from the port of arrival to the laboratory. All foreign soil must be shipped in sturdy, leak proof containers, which preclude spillage or pest escape during transit or processing.

A copy of the laboratory's USDA Soil Permit and copies (1 per container) of the PPQ Form 550 (i.e., the Foreign Soil/Restricted Entry label, Attachment 1) must be included in all outgoing shipments of containers (i.e., coolers) to foreign countries.

Special instructions for the sampling crew must be included in outgoing shipments to foreign countries. This form includes instructions to clearly label the shipping containers (i.e., coolers) with the PPQ 550 Form prior to shipment back to the laboratory.

12.3 Sample Receipt Procedures

Upon receipt of the samples, the laboratory must adhere a fluorescent green sticker stating "FOREIGN SOIL – STERILIZE BEFORE DISPOSAL" on each sample bottle/container to distinguish these types of samples. This sticker will serve as a warning to the laboratory personnel that special handling and sterilization procedures are needed prior to sample disposal.

The used shipping containers (i.e., coolers) must be sterilized upon receipt to the laboratory. The coolers used to ship soil samples must be generously sprayed with an approved disinfectant such as bleach, quaternary ammonia, or 70% alcohol solution to the point of runoff; allowed to drain into a municipal water system; and air dried.

The water residue (effluent) from the processing of soil samples, i.e., the melted ice water accompanying soil cooler shipments and the sample packaging must be treated with one of the approved disinfectants listed above to prevent a hazard of pest spread.

Upon log-in into the LIMS, the laboratory Project Manager must include a worksheet note that states: "Foreign soil samples. Handle/dispose according to EHSM Addendum."

Foreign soil samples must not be received from or re-shipped to other laboratories unless the receiving laboratory has a valid USDA Soil Permit and Compliance Agreement to handle restricted foreign soil. Prior written approval must be obtained from the PPQ office to ship foreign samples to another laboratory.

12.4 Sample Handling Procedures

The employee must take all precautions to ensure there is no contact with the soils. Proper personal protective equipment such as gloves, lab coat, and safety glasses must be utilized when handling samples.

Foreign soil must be stored in a secure storage area, separated from other domestic samples. The laboratory utilizes separate, labeled bins within each regular storage area to accomplish this. These bins are labeled "Foreign Soils", and only foreign soil samples may be placed inside.

After sample analyses are complete, the sample disposal coordinators from each department will verify the waste stream required for each sample.

12.4.1 Non-Hazardous Samples

Route samples to the Foreign Soil Holding Area in the disposal shed for off-site heat treatment. Heat treatment is performed by a licensed waste disposal company, Clean Harbors. Clean Harbors picks up the samples, incinerates, and forwards records of disposal back to the laboratory. The waste profile for non-hazardous wastes is CH268115.

12.4.2 RCRA Hazardous Samples

Route samples to the Foreign Soil Holding Area in the disposal shed for off-site heat treatment. Heat treatment is performed by a licensed waste disposal company, Clean Harbors. Clean Harbors picks up the samples, incinerates, and forwards records of disposal back to the laboratory. The waste profile for RCRA hazardous wastes is

CH591328.

Foreign soils with PCB concentrations exceeding 50ppm are disposed with non-foreign soil as PCB waste under waste profile CH591328.

12.5 Documentation

Documentation of the disposal of foreign soils includes: 1) internal chain-of-custody records; 2) laboratory waste disposal records; 3) manifests from waste disposal vendor; and 4) record of disposal (incineration) from waste disposal vendor.

12.6 Training

All analysts that perform work on restricted soils must be trained on this procedure. In addition, annual refresher training must be performed.

12.7 Responsibilities

12.7.1 It is the responsibility of the Department Manager/Supervisor to ensure this procedure is performed by an employee who has been properly trained in its use and has the required experience.

12.7.2 It is the responsibility of the Shipping and Receiving Department to include the necessary documentation (forms and stickers) in all outgoing shipping containers bound for foreign sampling sites as outlined in this procedure. It is the responsibility of the Shipping and Receiving Department to label all sample containers/bottles with the fluorescent green Foreign Soil sticker to trigger the laboratory to handle the samples according to this procedure. It is the responsibility of the Shipping and Receiving Department to decontaminate used shipping containers and melted ice in coolers in accordance with this procedure.

12.7.3 It is the responsibility of the Project Manager to notify the Shipping and Receiving Department of outgoing shipping containers that are being sent outside the United States so that the appropriate USDA documentation can be initiated in accordance with this procedure. It is the responsibility of the Project Manager to include a LIMS Worksheet Note upon log-in of foreign soil samples that states “Foreign soil samples. Handle/dispose according to Section 12 of EHSM Addendum.”

12.7.4 It is the responsibility of the laboratory staff to handle all foreign soil samples in accordance with this procedure. This includes segregated sample storage and heat treatment.

12.7.5 It is the responsibility of each department’s sample disposal coordinator to route all foreign soils to the area designated for foreign soil sterilization/heat treatment. It is the responsibility of the soil disposal coordinators to maintain records of all sterilized samples in accordance with this procedure.

12.7.6 It is the responsibility of the QA Manager to maintain the laboratory’s U.S. Department of Agriculture permit. It is the responsibility of the QA Department to maintain training

records of these procedures.

Attachment 12.1: FOREIGN SOIL COOLER LABEL

U.S. DEPARTMENT OF AGRICULTURE
ANIMAL AND PLANT HEALTH INSPECTION SERVICE
PLANT PROTECTION AND QUARANTINE
HYATTSVILLE, MARYLAND 20782

SOIL SAMPLES
RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550
(JAN 83) *Edition of 12/77 may be used.*
***U.S.GPO:1990-(719-601)**

Attachment 12.2: PPQ FORM 550 NOTE

NOTE TO THE SAMPLING CREW:

PPQ Form 550s (i.e., the Soil Samples/Restricted Entry label) have been provided with this shipment.

Prior to shipment of samples back to the laboratory, a PPQ Form 550 must be taped to the outside of each cooler so that it will be in plain view of Customs officials.

FAN054:08.01.07:3

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Section 13: WASTE MANAGEMENT

As an environmental laboratory, TestAmerica Savannah generates waste that must be stored and subsequently disposed of in accordance with mandated requirements. The requirements set forth by Federal and State regulations apply to TestAmerica Savannah based on the monthly volume of hazardous waste generated by each facility.

TestAmerica Savannah's EPA Generator ID Number is: **GAD984303206.**

13.1 General Safety Concerns and Requirements

Sample Handling
All samples must be treated as if they are hazardous. The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must wear protective clothing (lab coat or apron), eye protection (face shield and/or glasses), and disposable gloves when handling samples.

Waste Handling
Proper lifting techniques must be used when moving containers of waste. All of the manufacturer's safety procedures must be followed when using a pallet jack to move waste drums.

Employees must wear a lab coat, gloves that are resistant to the materials being disposed of, and safety glasses while working in the waste disposal areas. If the employee is performing a task that could generate a splash hazard (such as bulking liquid waste), a face shield must be worn in addition to safety glasses.

Safety Equipment
Eye wash stations and safety showers are in the waste disposal area as well as outside next to the storage building and well pump. There are heat detectors and fire alarms/strobes inside of the waste disposal area. The fire alarm pull box is located inside of the door of the main building. Fire extinguishers are located in the waste disposal area and inside of the main building by the exit door. These features should be used in the event of an emergency.

A grounding cable/clamp will be used when bulking flammable liquids. All waste drums are stored on spill containment pallets.

Respiratory protection is not required under normal circumstances in the waste disposal area. Respiratory protection is not allowed unless all of the regulatory requirements have been met and the EHSC and the Laboratory Director approve the use.

Access and Signage
The unauthorized entry of persons into areas where hazardous wastes are handled and stored should be prevented to the extent practical through building security, property protection, and reasonable surveillance. If hazardous wastes are stored in areas where such protection cannot reasonably be afforded, the facility should provide reasonable physical security; for example, fencing and locking on gates or doors when unattended.

An area in which hazardous wastes are stored should be clearly identified to reasonably warn employees or other persons of potential hazards in the area. The sign should identify the area as a hazardous waste storage area and limit access to authorized personnel only.

13.2 Generator Status

The laboratory is currently classified as a Large Quantity Generator (LQG) as the routine volume of waste generated is typically less than 2200 pounds; however, there are some months when greater than 2200 pounds is generated. This classification allows for storage of waste on-site for up to 90 days. The laboratory must determine the volume of hazardous waste and acutely hazardous waste it generates each month and report these values to the Corporate Environmental Health and Safety Officer (EHSO).

13.3 Waste Characterization

TestAmerica Savannah is required to identify and characterize wastes to determine if they are hazardous. Federal regulations define a waste as any material including solid, liquid, semi-solid, or contained gaseous material which is or will be discarded; has served its intended purpose; or is garbage, refuse, or sludge.

A waste is hazardous if:

1) it is not excluded from the definition of waste or hazardous waste and it is a listed hazardous waste;
2) if it exhibits any one or more of the characteristics of a hazardous waste (ignitability, corrosivity, reactivity, and toxicity);
3) if it is a mixture of a listed hazardous waste;
4) if it is used oil which contains over 1000ppm of total halogens; or
5) if it contains 50ppm or more of polychlorinated biphenyls (PCB).

The term “listed hazardous waste”, used above, refers to the EPA-established lists of hazardous wastes based on the material's properties including toxicity to humans, persistence or bioaccumulation in the environment, or other environmental or physical harm, which may result from the waste material.

Any waste that is mixed with a listed hazardous waste is classified as a hazardous waste. This rule discourages dilution as a means of avoiding hazardous waste regulatory requirements.

The laboratory’s Environmental Health and Safety Coordinator (EHSC) is responsible for ensuring that waste characterization and disposal are performed according to applicable regulatory and company requirements. The EHSC is also responsible for ensuring that the documents and manifests used to track the process are properly completed and maintained. The EHSC may perform the tasks directly or may delegate the tasks to trained laboratory personnel.

13.4 Exclusions to Waste Characterization Requirements

13.4.1 Sewer System / Wastewater Discharge

Domestic sewage and any mixture of domestic sewage and other wastes that pass through a sewer system to a publicly owned treatment works (POTW) for treatment are excluded from the definition of a solid waste. Domestic sewage means untreated sanitary wastes that pass through a sewer system.

Certain listed hazardous wastes are not regulated as hazardous wastes if they are a mixture of wastewater and one or more of the following listed hazardous wastes:

- Certain solvents being collected in a facility sewer system as long as the concentration does not exceed specified limits.

- De minimis losses of commercial chemicals due to ordinary handling, leaks and spillage, and rinsing of containers.

- Wastewaters from laboratory operations being collected in the laboratory sewer system, which contain listed hazardous wastes due to toxicity as long as the concentration of such wastes does not exceed 1ppm at the headworks of the POTW.

13.4.2 Laboratory Samples

Laboratory samples are excluded from being considered hazardous wastes when they are being:

- Transported to the laboratory for testing
- Transported back to the sample collector after testing
- Stored by the collector before transportation to the lab
- Stored by the lab prior to testing
- Stored by the lab after testing, but before being returned to the sample collector
- Stored temporarily in the lab after testing for a specific purpose (evidentiary use or retesting)

This exemption does not apply if the laboratory determines that the waste is hazardous or the laboratory is no longer meeting any of the conditions stated above. Shipping of laboratory samples must comply with Department of Transportation (DOT) or Postal Service requirements.

13.4.3 Empty Containers

Empty containers, which may have residues of hazardous wastes, are exempt from regulation as a hazardous waste. Hazardous waste containers are considered empty if no more than one inch of residue remains or no more than three percent of the total volume of the container remains. The term container is a broad term to include reagent containers, sample bottles, scintillation tubes and vials, glassware, and broken glassware, etc.

For containers, which previously held an acute hazardous waste, the container is considered empty after the following conditions have been met (see acute hazardous waste listed in Figure 3):

- The container has been triple-rinsed using an applicable solvent
- The container has been cleaned by another method proven to achieve equivalent removal
- If an inner liner prevented contact with the container, removal of the inner liner renders the container empty

13.5 Timing

In general, a material becomes a waste and must be characterized when the generator deems the material to be a waste.

A listed hazardous waste becomes a hazardous waste when a waste first meets the listing description.

A mixture of solid waste and one or more listed hazardous wastes becomes a hazardous waste when a listed waste is first added.

Any waste or waste mixture becomes a hazardous waste when it exhibits any of the characteristics of a hazardous waste.

13.6 Laboratory Waste Streams

Once characterized, a waste is segregated into a waste stream for disposal. A waste stream is established for each type of waste that is generated by the laboratory and a suitable container, if needed, is used to store that waste prior to disposal.

Types of waste streams include flammable waste, chlorinated solvents, pyridine, COD, inorganic waste, discarded water samples, discarded soil samples, other discarded samples, acid waste, Lab pack materials, extracted soils, sodium sulfate and filter paper.

Waste streams are collected into 5-gallon or 15-gallon carboys in each Satellite Accumulation Area and in 55-gallon drums in the Waste Storage Area.

Each 30 or 55-gallon drum must contain a unique identifier. The laboratory uses a unique alpha character code and the date the drum was opened.

The following codes are used:

Abbreviations	Waste
MECL2	Methylene Chloride
PCBSoils	Polychlorinated Biphenyls Contaminated Soils
FLAM	Flammable Solvents
TKN	TKN Waste
PYR	Pyridine Waste
IGN	Ignitability Waste
VIALS	Hexane/$MeCl_2$ Sample Extracts in Vials
RCRA Soils	RCRA Contaminated Soils
SoilVials	Plastic and Glass Soil Vials
RCRA Water	RCRA Aqueous Waste

For example if a pyridine waste drum was opened on 07/08/08 the drum would be labeled PYR070808.

Drums for any waste stream must be free of leaks. Waste streams containing liquids exhibiting extreme pH conditions (<2 or >11) can cause damage to metal drums. In order to help prevent this from happening, each container that contains a liquid layer is to have its liquid layer pH checked with wide range pH paper prior to the container being emptied into a drum. It is much easier to adjust the pH of a small container than a 55-gallon drum. No drums will be accepted for disposal with a pH <2 or >13. These drums will be rejected at the disposal facility and sent back to the laboratory. A handling fee will be assessed and the drum will have to be neutralized prior to any future disposal.

Addition of liquids to 55-gallon drums is to be performed with a drum funnel. Using a drum funnel will protect both the drum and the person adding liquids to a drum. After addition of any waste to a drum, the drum is to be wiped down and the area thoroughly cleaned. This will allow any leaks to be easily identified during weekly waste storage area inspections.

A waste stream profile form must be filled out for each waste stream and maintained on-file. The Hazardous Waste Management and Regulator Data Form is a non-regulatory form that documents regulatory information on a specific waste stream. The laboratory's Hazardous Waste Manager should prepare a form for each hazardous waste generated and maintain the document for three years.

13.6.1 Empty Containers, Reagent Bottles, and Sample Container Waste Stream

Empty containers are not hazardous if emptied and managed pursuant to 40 CFR 261.7.

13.6.2 Laboratory Refuse Waste Stream

All laboratory trash must be managed as non-hazardous waste and disposed of via landfill or incinerator.

13.6.3 Solvent Waste Streams

TestAmerica Savannah has two solvent waste streams. These include halogenated solvents (methylene chloride, chloroform, and freon) and flammable solvents (acetone, hexane, ether, methanol and any other solvents listed with a primary hazard of flammability). It is imperative that these two waste streams be kept separate. Halogenated solvents and flammable solvents have the potential to react very violently when mixed. These two solvents also have the potential to react with one another and create an acidic medium.

The integrity of each waste stream profile must be kept. It is also important that no solvent waste stream ever have more than 5% water after the total addition of waste to a drum. Violation of these criteria will result in the drum being returned to the division of origin. This new waste stream will be approximately three times the cost of the normal disposal fee. In addition, a handling and return transportation fee will be assessed.

13.6.4 Wastewater from Organic Extractions

Wastewater from organic extractions is treated as non-hazardous waste. The wastewater, which may be acidic due to laboratory pH adjustment, is neutralized by passing the sample though limestone rocks (calcium carbonate) in the laboratory and is discarded through the plumbing. The neutralized water then passes into the City of Savannah's sewer system.

The laboratory also utilizes a neutralization pit to adjust water pH originating from the new building. The pH of the neutralization pit is monitored monthly by designated personnel.

13.6.5 Acidic and Alkaline Wastewater

Acidic and alkaline wastewater can be disposed of via the sewer if neutralized prior to release into the sewer system.

13.6.6 Inorganic Waste Streams

The inorganic/metals waste stream is comprised of metals standards and mixes that contain metals on the TCLP list at concentrations above the regulatory limit.

Aqueous metals wastes containing metals on the TCLP list above the regulatory level are to be separated into two waste streams. Mercury waste is to be isolated into its own waste stream. Any mixed solutions that contain mercury above the regulatory level are to be treated as mercury waste. Due to the small volume of waste that is generated for both mercury and metals waste, these waste streams are sent out as lab packs.

Prior to the aqueous metals waste being added to a drum, the pH is to be checked and adjusted, if necessary, to >2 but <11. The same rules of pH apply to metals waste as

they do to solvent waste. Drums not falling within the proper pH range will be returned and a handling and transportation fee will be assessed.

13.6.7 Neat and Primary Standards

Most neat standards do not have an expiration date. Those compounds that do have an expiration date generally have a fairly lengthy shelf life. When it becomes necessary to dispose of neat standards, the most feasible way to dispose of these standards is to return them to the vendor that they were purchased from.

Primary standard solutions, whether they were purchased as such or made up internally, are disposed of in the solvent waste stream the standard was made up with (example: standards made up in methylene chloride should be disposed of as a halogenated solvent).

Any standards containing PCBs greater than the regulatory limit of 50ppm must be characterized as PCB waste and disposed of as such. Any empty standard containers (vials, ampules, etc.) containing PCBs at a concentration greater than 500ppm must be disposed of as a hazardous waste. Those empty containers are to be placed into a solid PCB waste stream and disposed of accordingly.

13.6.8 Sample Extracts and Digests

The disposal of sample extracts and sample digests should be performed at the time that the actual samples are disposed (usually 30 days after results submission). If it is not possible to dispose of the digests and extracts on the same day as their associated samples, the extracts and digests should be disposed of in a reasonable time frame after the disposal of their associated samples.

Sample digests should be neutralized and disposed of as laboratory wastewater effluent in accordance with 40 CFR 261.3(E). The containers should be destroyed and disposed of in a sanitary landfill. TestAmerica Savannah has deemed these containers to be empty in accordance with 40 CFR 261.7.

Sample extracts should be disposed of in the waste stream associated with the final solvent that they were prepared in. For ECD work the final solvent would be hexane; therefore, the extracts should be disposed of as a flammable solvent. Extracts with a final solvent of methylene chloride should be disposed of as halogenated waste. Sample extracts should be disposed of at a satellite accumulation area in the appropriately labeled solvent waste carboy. All procedures for disposal of solvent waste should be followed as listed in this SOP.

13.6.9 Excess Samples

There are three separate waste streams for excess samples: non-hazardous samples, hazardous samples, and samples returned to client. Sample waste streams are determined by LIMS, which generates reports identifying how excess samples are to be disposed.

13.6.10 Non-Hazardous Aqueous Samples

Aqueous samples that appear on the non-hazardous sample disposal sheets are to be neutralized and disposed of via the public sewer system.

As samples appear on the non-hazardous sample disposal sheets, they are checked out of the department and moved into the sample disposal area. The samples are neutralized before disposal in the sanitary sewer system.

13.6.11 Soil Samples

Excess soil samples are to be characterized after showing up on non-hazardous sheets and checked out of the department. If excess soils are deemed non-hazardous they may be disposed of in a landfill or incinerator. If, by characterization, soil samples are deemed hazardous, they must be placed in a drum and disposed of in accordance with Section 13 of the EHSM Addendum.

13.6.12 Oil Samples

Oil samples that appear on the non-hazardous sample disposal sheets are to be disposed of in one of two ways. If the facility has a small volume of waste oil samples to dispose of, less than one gallon per month, then the waste oil can become part of the flammable liquid waste stream. If the facility has a large volume of waste oil, then a separate waste stream for waste oils should be created.

Oil samples that are deemed to be hazardous should be handled as part of the facility's flammable liquid waste stream, unless the sample has greater than 50ppm of PCBs. If the oil sample is hazardous and has greater than 50ppm of PCBs, it must be treated as PCB waste.

13.6.13 Hazardous Samples

Hazardous samples are samples that have been flagged as exhibiting hazardous characteristics, samples that require disposal via a hazardous waste disposal company per the clients request, and/or the composite samples collected from the in-house solid waste stream that have failed for hazardous waste characterization.

A list should be compiled listing all parameters for which the samples in question have failed the test for hazardous waste characterization and any other analytical data supporting the contents of other compounds found in the samples.

13.6.14 Samples to be Returned to the Client

Samples are excluded from regulation as hazardous waste as long as they are regarded as useful by the laboratory, the sampler, or the originator of the sample. Consequentially, samples may be returned as samples to allow the customer to make the judgment of how to dispose of the waste. If practical, this approach will reduce the waste generated by the lab and minimize liability. From the customer's perspective the sample is but a small part of a larger whole, which should not be an additional cost

burden. However, if sample return to the client is not possible, the laboratory must characterize the waste sample as to whether or not it is hazardous.

As discussed previously, laboratory samples are excluded from the definition of hazardous waste if they are being sent back to the sample collector. However, when these samples are returned to the collector/client **all DOT and IATA shipping regulations must be followed.** Environmental samples do not have to be transported back to the collector/client as hazardous waste. If the samples exhibit any of the hazardous material classifications listed below, however, they must be shipped back to the collector/client as a hazardous material. The hazardous material classifications as recognized by DOT and IATA are:

1. Explosives
2. Flammable gas
3. Flammable solids
4. Reactive substances
5. Oxidizers
6. Poisons
7. Radioactive Materials
8. Corrosive Materials

There are times when the sample collector and/or client may request that their sample be returned to them or to another location for further testing or disposal. Those samples that do not exhibit any hazardous characteristics as defined in 49 CFR HM-181 may be shipped back by any means acceptable to the client. If the client wants the samples to be shipped back via air transport, the IATA regulations must be reviewed to ensure that none of their regulations are being violated.

It is the project manager's responsibility to contact their client and obtain the necessary information needed to fill out the TestAmerica shipping bill. The shipping bill and all shipping labels are to then be given to the sample custody section. This form will address the following information:

1. Client's name
2. Client's address to which samples are to be shipped
3. Mode of shipment i.e. UPS, Federal Express, ground transport.
4. Type of containers to be used i.e. cooler, drum, etc.
5. Need for wet ice preservation
6. Any special client request

The sample custody group will then locate and pack the samples as instructed on the shipping bill. The Hazardous Waste Manager will then review the shipping bill and check all packages for proper packaging and labeling requirements. When all of the proper packaging and labeling requirements are met, the Compliance Officer will release the package(s) for shipment. When the samples are picked up by the transporter, the transporter is to initial the shipping bill, relinquishing the samples to his custody. A copy of this shipping bill will remain at the laboratory. Upon receipt of the samples, the client is to return a signed copy of the shipping bill verifying that the shipment has been received.

Samples that have been determined by the laboratory as not being potentially hazardous or that may contain hazardous or dangerous material except in quantities as defined in 49 CFR HM-181 and the IATA are to be returned to the client by the most efficient and economically feasible means.

Samples being shipped back as non-hazardous may be packaged for return to the collector/client in coolers and regular TestAmerica shipping boxes.

13.7 Waste Stream Management and Storage

Once the lab has identified the wastes it generates, it must consider what it will do to manage the wastes. Waste management techniques include keeping non-hazardous wastes segregated from hazardous wastes, recycling of wastes, and sewering of wastes when appropriate. To reduce the volume of hazardous wastes generated and requiring management, the laboratory should make efforts to utilize processes that generate non-hazardous waste whenever practical.

The Satellite Accumulation Areas consist of 5-gallon carboys placed in secondary spill containers located throughout each laboratory department that generates the waste. The waste streams generated in each section are as follows:

- Extractions Liquid-Liquid Lab – Flammable Wastes & Chlorinated Solvents
- Extractions Soil Lab – Flammable Wastes & Chlorinated Solvents
- HPLC Lab – Flammable Wastes & Chlorinated Solvents
- Volatiles Lab – Flammable Wastes
- General Chemistry Lab – Flammable Wastes, Pyridine Wastes, and COD Wastes

Wastes are ultimately stored in 30 or 55-gallon drums. The drums are stored in the Main Waste Accumulation Area. Each drum is labeled with the date the first waste was accumulated and the appropriate hazardous designation for the waste contained within.

13.7.1 Use and Management of Containers

Proper selection and use of containers for hazardous waste must ensure effective containment, avoiding incompatibility of wastes and container, careful handling and keeping the container fully closed except when adding or removing wastes. As stated before, containers of hazardous waste must be labeled as hazardous waste and the labels marked with an accumulation start date. The EHSM contains a list of the containers that are allowed in section 13.7.3.

13.7.2 Inspection

Inspection of hazardous waste container storage areas is required on a weekly basis for Small Quantity Generators (SQG) only. It does not apply to satellite accumulation areas, but only to storage areas *(see Appendix A - Inspection Checklist Figure 9.1)*. Storage

areas must not be crowded, and adequate aisle space must be afforded for inspections and spill response actions. A thorough inspection should include the following:

- Condition of containers
- Proper container labeling including accumulation start date
- Adequate supply of spill response material – spill cleanup materials are stored in the extractions laboratory areas.
- Safety equipment should be in proper working order
- Secondary containment for drums – the pallets that the drums sit on are spill containment pallets.

13.7.3 Waste Processing

The Hazardous Waste Coordinator collects the waste containers from the Satellite Accumulation Areas as needed. These 5-gallon waste containers are emptied into the appropriate 30 or 55-gallon drums in the Main Waste Storage Area. Once the drums are filled to capacity (allowing a minimum of a 5% headspace) the Hazardous Waste Coordinator schedules a pick-up time with the hazardous waste company. Note: Methylene chloride drums require a minimum of 6 inches of headspace.

All laboratory departments must notify the Hazardous Waste Coordinator of any hazardous waste they have accumulated. The Hazardous Waste Coordinator determines which waste can be brought for proper disposal. An inventory sheet must accompany all waste that is brought to the Hazardous Waste Coordinator.

Waste Tracking and Inspection Forms are completed by the Hazardous Waste Coordinator. A copy of the TestAmerica Savannah Waste Tracking log is included (Figure 2). The Drum ID is generated in accordance with Section 13.6 by using the waste type or abbreviation and the start date. For Example: methylene chloride waste that the drum was started on January 4th, 2005 would be **MECL2010405**; flammable waste started on December 17th, 2004 would be FL121704. The waste tracking log is attached and stored with the waste manifest. Manifests, Land Disposal Restrictions (LDRs), and regulatory reports are maintained on-file by the Hazardous Waste Manager.

13.8 Initial Requirements and Time Limits

13.8.1 Packaging Wastes

Once hazardous wastes are generated they must be immediately packaged and labeled. The label must be marked with the words *hazardous waste*, the classification of the hazardous waste, and the accumulation start date. Containers used for hazardous waste storage must be kept securely closed or covered except to add, remove, or inspect waste. Containers should be in good condition and should be compatible with the waste that will be stored in them. Different hazardous wastes should not be mixed in a container unless the wastes are known to be compatible and are being co-mingled to improve waste management efficiency.

13.8.2 Accumulation and Storage Time Limits

Hazardous waste storage time limits depend on the generator status. The time limits begin on the date of first accumulation (when hazardous wastes are first placed in the container and an accumulation start date marked on the label), unless the satellite rule applies to the container.

13.8.3 90 Day Limit for Large Quantity Generators

For LQGs the time limit for on-site storage is 90 days.

13.8.4 Satellite Rule

The satellite rule (40 CFR 262.34(c)) allows substantial accumulation of hazardous waste at the point of generation before the time clock begins to run. A satellite accumulation area must be at or near the point of generation, must be under the control of the operator generating the waste, and must be in containers. Up to 55 gallons can be stored in the satellite accumulation area for each separate waste stream. A label indicating hazardous waste and a description of the waste must be affixed to the container. Within three days of reaching the volume limit (55 gallons), the container must be moved to the regular hazardous waste storage area.

Note: Waste containers in the satellite accumulation areas are not required to be labeled with the start date per 40 CFR 262.34(c).

13.9 Waste Minimization

The laboratory should attempt to reduce the amounts of wastes generated at the source through changes in procedures, processes, equipment, and raw materials. The following operating practices can help minimize wastes generated:

- Improved material handling and inventory practices to reduce loss of hazardous materials due to mishandling, expired shelf-life of materials, and improper storage conditions
- Improved housekeeping practices
- Leak/spill prevention
- Waste segregation
- Managing samples which are potentially hazardous in a manner which facilitates return to the customer before the sample becomes a hazardous waste
- Sewering of excluded hazardous wastes mixed with domestic sewage from the lab may be permissible because the wastewater is excluded from the definition of solid wastes.

13.10 Recycling

The laboratory should reuse and recycle wastes on-site as a replacement for new materials when feasible. The most common example of recycling is the recovery and rescue of extraction solvents. This type of recycling is considered to be reclamation due to the application of heat during the distillation process. Wastes that are reclaimed count toward the volume of wastes generated by the laboratory and must be managed as

hazardous waste until recycled. At this time the Savannah location is not recycling any wastes on site.

13.11 Waste Disposal and Discharge

13.11.1 On-Site Treatment of Laboratory Hazardous Wastes

Bench top treatment of hazardous wastes to render chemical wastes and wastewaters suitable for sewer discharge, make the waste less hazardous, or to reduce its volume prior to other types of disposal is a historical and appropriate practice for laboratories. The RCRA regulations state that a permit is required for the treatment of hazardous wastes unless specifically exempted (40CFR270.1) The specific exemptions from permitting are:

- Any treatment of material excluded from the definition of waste
- Treatment by elementary neutralization
- Rinsing of empty containers
- Treatment in an emergency.

13.11.2 Sewering of Laboratory Wastes

There are several provisions in the RCRA regulations, which allow the sewering of hazardous waste including those from laboratories. These exemptions include:

- General exclusion from the definition of solid wastes for mixtures of domestic sewage and other wastes, which pass through a sewer system.
- Specific regulatory provisions, which exclude certain types of laboratory solvent wastes mixed with wastewater from the definition of hazardous waste
- Other rationales for discharge to the sewer system such as demonstrating that the wastewater in question is non-hazardous and pretreatment to reduce to eliminate a hazardous constituent from wastewater

13.11.3 Prohibited Hazardous Waste Discharge

An excluded hazardous waste referred to as a pollutant in Clean Water Act regulations may not be discharged to a sewer system on Publicly Owned Treatment Works (POTW) if the waste is ignitable, corrosive with a pH of less than 5, contains oil and grease, would produce toxic gases or fumes sufficient to cause worker health problems at the POTW.

Laboratories should be aware of any local property discharge limits (see Figure 8). It is the duty of the EHSC, to keep the laboratory in compliance with local discharge regulations and limits.

13.11.4 Reporting Requirements and EPA ID Numbers

Large quantity generators are required to submit bi-annual reports, and must have an EPA ID number. All TestAmerica facilities have an EPA Generator ID number. This number is needed to complete the Uniform Hazardous Waste Manifest necessary to

transport hazardous wastes off-site. The laboratory should be aware that state and local governments might require reports or notifications not required by the federal regulations for SQG. Some states might require filings for fee assessment, permit issuance, or other taxing purposes.

13.11.5 Off-Site Disposal and Transportation of Hazardous Wastes

Before TestAmerica Savannah can contract for the transportation of hazardous wastes off-site for disposal, the laboratory must package and label the hazardous wastes in accordance with DOT and hazardous waste regulations.

13.11.6 DOT Shipping Information

To satisfy DOT shipping requirements, the shipper needs to consult the DOT Hazardous Materials Table, 49 CFR 172.101. After locating the chemical name of the waste the lab is shipping, the lab should determine from the table the Hazard Class (column 3), United Nations (U.N.) Number (column 4), Packaging Group (column 5), and Labeling and Placarding Requirements (column 6). If a shipment can fit into more than one hazard class, the DOT requires the shipper to use the class that has the lowest class number.

13.11.7 Labeling

Each container of 110 gallons or less must be labeled with the following information:

- Clear designation that the container holds hazardous waste with the following words displayed:
  Hazardous Waste - Federal Law Prohibits Improper Disposal. If found, contact the nearest police, public safety authority, or the U.S. Environmental Protection Agency.
- The name and address of the facility generating the waste
- The Uniform Hazardous Waste Manifest document number
- Proper DOT shipping name and U.N. number
- EPA hazardous waste number
- The generator's EPA ID number

13.11.8 Uniform Hazardous Waste Manifest

All shipments of hazardous waste for disposal must have a completed Hazardous Waste Manifest. The Hazardous Waste Manifest is a document designed to facilitate cradle-to-grave tracking of hazardous wastes that the regulatory agencies need to monitor the generation and eventual disposal of such wastes. The manifest also satisfies the DOT's requirements for shipping papers for hazardous materials transportation. The lab will need the following information to complete the manifest:

- The laboratory's EPA ID number
- Transporter Information and ID number
- Destination Facility Information and ID number
- Hazardous Waste Information (proper shipping name, hazard class, volume of waste)

The laboratory must complete items 1 - 16 on the manifest (Figure 10). The transporter signs item 17 upon pickup, leaves two copies (one for the lab and one that the generator must send to the EPA regional office within 30 days) and keeps the remaining multi-copy set. The transporter then delivers the waste to a disposal facility, which becomes the destination facility. The destination facility signs item 20 and sends the original acknowledgment copy back to the lab. Upon receipt of the acknowledged copy, the lab should compare this document versus the original for discrepancies. Any discrepancies should be investigated by contacting the destination facility. The lab is responsible for its waste from cradle-to-grave; therefore, if the lab has not received an acknowledged copy within 35 days, the destination facility should be contacted. Both laboratory copies of the manifest must be retained for three years as a legal record indicating that the lab complied with hazardous waste regulations and that the waste arrived at its specified destination.

13.12 Training Requirements

13.12.1 Hazardous Waste Management Training

Laboratory employees who handle hazardous wastes must undergo the Hazardous Waste Management initial 8-hour training course. This training is currently handled through the Corporate EH&S Program.

A 4-hour refresher training must be completed every year which, at a minimum, reviews the information covered in initial training.

13.12.2 Hazardous Materials Management (DOT) Training

Any employee who engages in activities involving the packaging, shipping, and labeling of hazardous materials needs to have Hazardous Materials Management training. This requirement is only necessary for employees shipping hazardous materials. This does not include custody personnel that ship samples and sample containers. This training is currently handled through the Corporate EH&S Program.

A 4-hour refresher training must be completed every 3 years which, at a minimum, reviews the information covered in initial training.

13.12.3 OSHA HAZWOPER Training

Any laboratory which generates hazardous waste must comply with the OSHA Hazardous Waste Operations and Emergency Response Standard commonly referred to as HAZWOPER (29 CFR1910.120). Every facility should have at least one employee who has completed a 24-hour HAZWOPER course. This training is currently handled through the Corporate EH&S Program.

Note: The 40-hour HAZWOPER training is required for any field personnel who collect samples at HAZWOPER sites. This training must be performed by an outside agency.

A 4-hour refresher training must be completed every year which, at a minimum, reviews the information covered in initial training.

13.13 REFERENCES

40 CFR Transportation Part 260, Waste Management

13.14 APPENDICES

Appendix A
Figure 1 – Waste Stream Abbreviations
Figure 2 – TestAmerica Savannah Hazardous Waste Tracking Form
Figure 3 – Acutely Hazardous Chemicals Used at TestAmerica Savannah
Figure 4 – Determination of whether a RCRA Solid Waste is a Hazardous Waste
Figure 5 – Waste Profile Form
Figure 6 – Hazardous Waste Management and Regulatory Data Form
Figure 7 – Regulatory Levels
Figure 8 – Discharge Limits
    Figure 8.1 – Application of Satellite Rule to Points of Generation
Figure 9 – Inspection Forms and Instructions for Use
    Figure 9.1 – Main Waste Storage Areas Weekly Inspection Form
Figure 10 – Example of Uniform Hazardous Waste Manifest and Instructions

Appendix B
TestAmerica Savannah Sample Disposal Tracking System

Appendix A
Figure 1: WASTE STREAM ABBREVIATIONS

Abbreviation	Waste Type	Dept	Waste Profile #
MECL2	Methylene Chloride	EX, SG, SM	CH196325
PCBSoils	Polychlorinated Biphenyls Contaminated Soils	EX, SG, SM	CH196304
TKN	TKN Waste	GE	CH315347
PYR	Pyridine Waste	GE	CH196327
VIALS	Hexane/$MeCl_2$ Sample Extracts in Vials	SG, SM	CH129436
MIX	Mixed Solvent Waste	EX, SG, SM, VM, VG, EX, FD	CH196328
RCRASoil	Plastic and Glass Soil Vials	All	CH609534
RCRAWater	Aqueous RCRA Waste	All	CH401668
CAD	LACHAT Cadmium Columns	GE	
COD	COD Waste	GE	Return to vendor
Ignite	Used Oils/Flammable Samples	All	CH705078

Example naming convention for methylene chloride drum opened on January 1, 2009 would be MECL2010109.
Example naming convention for pcb contaminated soil waste drum opened on March 17, 2009 would be PCBSoils031709.

Figure 2: TESTAMERICA SAVANNAH HAZARDOUS WASTE TRACKING FORM

TestAmerica Savannah Hazardous Waste Drum Tracking Log

	Drum ID			
	Waste Type			
	Date	Initials	Waste	Amount Added
Date Started				

Manifest ID: _____ Date Shipped _____

	Drum ID			
	Waste Type			
	Date	Initials	Waste	Amount Added
Date Started				

Manifest ID: _____ Date Shipped _____

FAN102:02.18.14:6

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Figure 3: ACUTELY HAZARDOUS CHEMICALS USED AT TESTAMERICA SAVANNAH

Note: These containers must be triple rinsed before disposing of the container.

Chemical (Neat Form Only)	Triple Rinse With	Disposal of Waste Rinse
Acrolein	Acetone	Flammable Waste
Aldicarb	Acetone	Flammable Waste
Aldrin	Acetone	Flammable Waste
Butanone-2	Acetone	Flammable Waste
Butanone-2 Oxime	Acetone	Flammable Waste
Carbon Disulfide	Acetone	Flammable Waste
Dieldrin	Acetone	Flammable Waste
Dinoseb	Acetone	Flammable Waste
2,4-Dinitrophenol	Acetone	Flammable Waste
Dimethoate	Acetone	Flammable Waste
Endosulfan	Acetone	Flammable Waste
Endothall	Acetone	Flammable Waste
Endrin	Acetone	Flammable Waste
Famphur	Acetone	Flammable Waste
Heptachlor	Acetone	Flammable Waste
2-Methyl-4,6-dinitro phenol	Acetone	Flammable Waste
Methyl Parathion	Acetone	Flammable Waste
Potassium Cyanide	Water	Sewer
Pyridine	Methanol	Pyridine Waste
Sodium Cyanide	Water	Sewer
Thiophenol	Acetone	Flammable Waste
Toxaphene	Acetone	Flammable Waste

Figure 4: DETERMINATION OF WHETHER A RCRA SOLID WASTE IS A HAZARDOUS WASTE

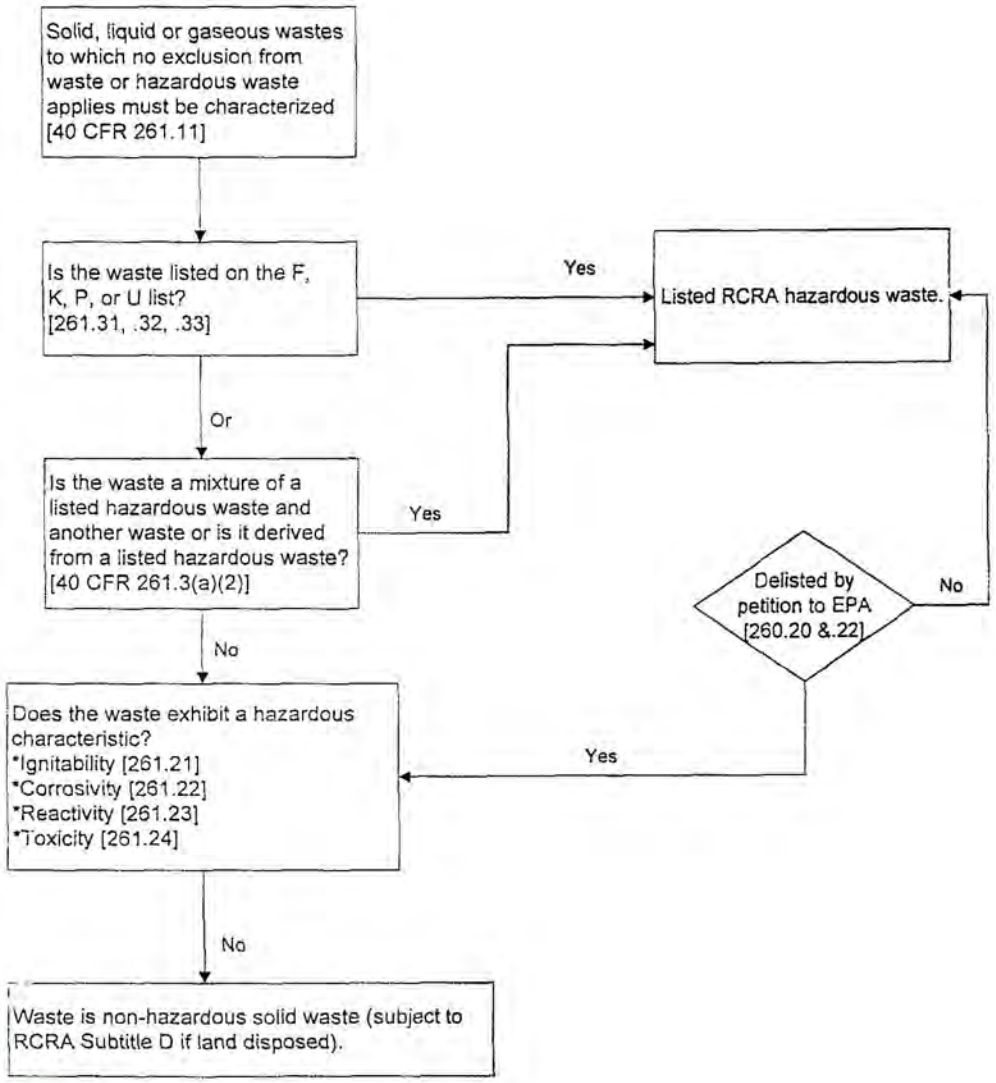


Figure 5: WASTE PROFILE FORM

A generator of waste must handle the waste as if it is hazardous, unless by knowledge and/or testing the waste can be demonstrated to be non-hazardous pursuant to 40 CFR 261 requirements. Therefore, a hazardous waste must be profiled or appropriately described in terms of its chemical and physical properties and how such properties relate to regulatory requirements to assure correct characterization. This is turn will assure proper transportation and treatment or disposal of the waste and completion of hazardous waste management documentation (labels, manifests and other paperwork). Generators are required to maintain documentation on hazardous waste generated for three years.

The **Waste Profile Form** is a non-regulatory form that incorporates source information, chemical and physical data relevant to hazardous waste identification and regulatory information. Compiling this information accurately is essential to proper hazardous waste identification and management, ***or***, if the waste is non-hazardous, to competently demonstrate this determination.

As the generator will be held strictly liable for errors in waste characterization, care in use and completion of this form must be exercised.

Tips on Accurate Waste Profiling

> ***Regulations*** on hazardous waste identification [40 CFR 261] should be consulted for a precise and thorough description of the applicable characteristic or regulatory threshold for the property in question.
>
> ***Samples*** of the waste to be profiled must be ***representative samples.*** This requirement means that if the waste is variable, a sufficient number of samples must be taken to assure that the waste stream as a whole is being profiled accurately. In general, if the results of testing indicate a close fit either above or below a certain criterion, more sampling and retesting should be conducted to assure correctness of the result. The ultimate authority on representative sampling and methodology is EPA reference document ***SW-846.***
>
> ***Knowledge or testing*** can be the basis for the generator's characterization of its waste. For example, determinations of whether the waste is a listed hazardous waste are virtually always made on the basis of knowledge of the source of the waste or its composition by knowing the process which generated the waste or the composition of the raw materials which became wastes from a Material Safety Data Sheet (MSDS) or other information. In some cases, hazardous waste characteristics (like corrosivity, ignitability, and reactivity) can be identified by knowledge only, for example, based on MSDS data; however, the generator should assure its determination is representative of the waste generated. Furthermore, all hazardous waste characteristics must be considered to assure full profiling and proper management, not only the one characteristic that clearly makes the waste hazardous. Finally, if the objective is to characterize the waste as non-hazardous, there must be some testing performed because characterization based on knowledge alone puts the generator at significant risk and disposal facilities usually require documentation if the waste is not clearly inert.

There is no requirement for EPA approval of the generator's characterization of its wastes unless the generator intends to delist a listed hazardous waste.

Instructions for Using the Hazardous Waste Profile Form

1. **Source and Nature of Waste.** This section catalogues the descriptive information on the generator, the generating process and the waste in question. This section covers information essential to characterize the waste through knowledge: source process and/or composition. It also covers necessary information to assure an appropriate testing procedure is selected and the basis to consider a sample to be representative.

2. **Lists of Hazardous Waste.** This section requires the generator to review the EPA lists of hazardous wastes to determine if based on the source of the waste or its composition it is a listed hazardous waste. Users of this form should be aware of the mixture and derived from interpretation of the EPA regulations which require a generator to consider a waste with a component which is a listed hazardous waste to be a hazardous waste generally irrespective of concentration. Identification of the hazardous waste number is important for completing additional documentation and for reference purposes.

3. **Characterization of Samples for Physical Hazardous Properties.** This section documents information derived on the basis of knowledge or testing on the physical hazard properties (ignitability, corrosivity, and reactivity, which are hazardous waste characteristics) of a waste, which would make it a hazardous waste if the applicable thresholds are exceeded. The form allows for documentation of testing as a source of the result with appropriate reference to the lab report and date ***or*** knowledge and source of this information.

4. **Characterization of Sample for Toxicity Characteristics.** This section documents information derived on the basis of knowledge or testing on the toxicity characteristic. The test for the toxicity characteristic is Toxicity Characteristic Leaching Procedure (TCLP). This section may be completed on the basis of testing or knowledge, although appropriate testing is recommended if the generator is attempting to demonstrate its wastes are non-hazardous. For each toxicity parameter the form provides for documentation of the testing and/or the source of information.

5. **Restricted Wastes: Hazardous Constituents and Treatment Standards.** This section allows the generator to document the results of hazardous constituent analyses and compliance with treatment standards required for restricted hazardous wastes.

6. **Conclusion.** This section is used to summarize the waste characterization conclusion and to indicate additional information with respect to the type of hazardous waste. This latter information is derived directly from the information documented on this form and will assist the generator in meeting additional regulatory requirements for the hazardous waste in question. For further references on the different types of hazardous waste, see the noted section.

Recordkeeping Requirements

The information on this form must be maintained for at least three years from any regulated activity involving the waste. If the hazardous wastes are restricted, information must be maintained for five years. It should be kept current by updating and periodic review, especially if the properties or composition of the waste changes.

Figure 6: HAZARDOUS WASTE MANAGEMENT AND REGULATORY DATA FORM

Instructions for Completion and Use of Hazardous Waste Management and Regulatory Data Form

1. **Generator Information/Description of Hazardous Waste.** This section documents generator information and basic hazardous waste identification data. The generator information includes the facility's EPA ID number and any applicable state information. The description of the hazardous waste should be derived from Figure 5 Waste Profile Form.

2. **Initial Packaging and Accumulation System.** This section indicates the initial containment, labeling requirements for on-site accumulation and the system the generator will use for accumulation (i.e., satellite rule). It also indicates whether any compatible wastes can be co-packaged.

3. **On-Site Waste Management.** This section indicates whether the waste may be sewered or treated on-site. Due to the important regulatory restraints on these activities, the form should indicate the regulatory basis for these methods and any limitations that apply.

4. **On-Site Accumulation/Storage Time.** This section identifies the applicable storage time limitation based on the generator's status and how the generator plans to comply with respect to this waste stream.

5. **Shipping Label Information**. This section provides the additional shipping label information for a hazardous waste being transported. The following are the information requirements for transportation labels. ***Note:*** Many generators use the transportation label as an on-site label. This is permissible as long as the additional information is added prior to shipment.

   The words Hazardous Waste
   Name and address of the generator
   Physical state (solid, liquid, or compressed gas)
   Hazardous properties
   Accumulation start date (the date when wastes are first placed in the container)
   Proper DOT shipping name
   Hazard class
   UN or NA number (Use UN number unless one is not assigned.)
   EPA hazardous waste number
   Generator's EPA ID number
   Legend on the label which prohibits improper disposal and requires the contacting of EPA and local law enforcement in the event of an emergency

   The hazardous waste transportation information including proper DOT shipping name, hazard class, UN/NA numbers, and other shipping information is contained in DOT regulations at 49 CFR, especially 172.101 (hazardous material table) and explanatory regulations. These federal regulations apply to all transportation of hazardous wastes. Compliance must be meticulous.

6. **Manifest Information.** This section covers the additional information needed to properly complete a uniform hazardous waste manifest. This information overlaps the labeling data requirements. The required information includes waste-specific data, waste description, proper shipping name, and other information necessary to fully and accurately complete the manifest form. To complete the special handling and safety equipment aspects of the manifest, the user should consult the Material Safety Data Sheet (MSDS) for the raw material components of the hazardous waste or similar information from chemical safety guides. This information is non-regulatory. Consequently, strict precision is not essential. However, concern for the safety of handlers requires appropriate advice.

7. **Recordkeeping**. The information on the Hazardous Waste Management and Regulatory Data Form is not regulatory, and therefore is not subject to a specific recordkeeping requirement. However, the generator must be able to document actions taken to comply with regulatory requirements and the validity of the information represented. Therefore, this form is an efficient means of supporting manifests, labels and other regulatory requirements. This form should be kept current and maintained for at least three years to document hazardous waste management decisions.

Figure 7: REGULATORY LEVELS

PCBs: >50ppm
Ignitability: ≤140 Degrees F
Corrosivity: ≤2.0 or ≥12.5
Reactivity: Cyanide: 250ppm Sulfide: 500ppm

Maximum Concentration of Contaminants for the Toxicity Characteristic			
EPA HW No.[1]	Contaminant	CASNo.[2]	Regulatory Level (mg/L)
D004	Arsenic	7440-38-2	5.0
D005	Barium	7440-39-3	100.0
D018	Benzene	71-43-2	0.5
D006	Cadmium	7440-43-9	1.0
D019	Carbon tetrachloride	56-23-5	0.5
D020	Chlordane	57-74-9	0.03
D021	Chlorobenzene	108-90-7	100.0
D022	Chloroform	67-66-3	6.0
D007	Chromium	7440-47-3	5.0
D023	o-Cresol	95-48-7	200.0[4]
D024	m-Cresol	108-39-4	200.0[4]
D025	p-Cresol	106-44-5	200.0[4]
D026	Cresol		200.0[4]
D016	2,4-D	94-75-7	10.0
D027	1,4-Dichlorobenzene	106-46-7	7.5
D028	1,2-Dichloroethane	107-06-2	0.5
D029	1,1-Dichloroethylene	75-35-4	0.7
D030	2,4-Dinitrotoluene	121-14-2	0.13[3]
D012	Endrin	72-20-8	0.02
D031	Heptachlor (and its epoxide)	76-44-8	0.008
D032	Hexachlorobenzene	118-74-1	0.13[3]
D033	Hexachlorobutadiene	87-68-3	0.5
D034	Hexachloroethane	67-72-1	3.0
D008	Lead	7439-92-1	5.0
D013	Lindane	58-89-9	0.4
D009	Mercury	7439-97-6	0.2
D014	Methoxychlor	72-43-5	10.0
D035	Methyl ethyl ketone	78-93-3	200.0
D036	Nitrobenzene	98-95-3	2.0
D037	Pentachlorophenol	87-86-5	100.0
D038	Pyridine	110-86-1	5.0[4]
D010	Selenium	7782-49-2	1.0
D011	Silver	7440-22-4	5.0
D039	Tetrachloroethylene	127-18-4	0.7
D015	Toxaphene	8001-35-2	0.5
D040	Trichloroethylene	79-01-6	0.5
D041	2,4,5-Trichlorophenol	95-95-4	400.0
D042	2,4,6-Trichlorophenol	88-06-2	2.0
D017	2,4,5-TP (Silvex)	93-72-1	1.0
D043	Vinyl chloride	75-01-4	0.2

[1] Hazardous waste number.
[2] Chemical abstracts service number.
[3] Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.
[4] If o-,m-, and p-Cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level of total cresol is 200 mg/L [6-1-90].

Figure 8: DISCHARGE LIMITS

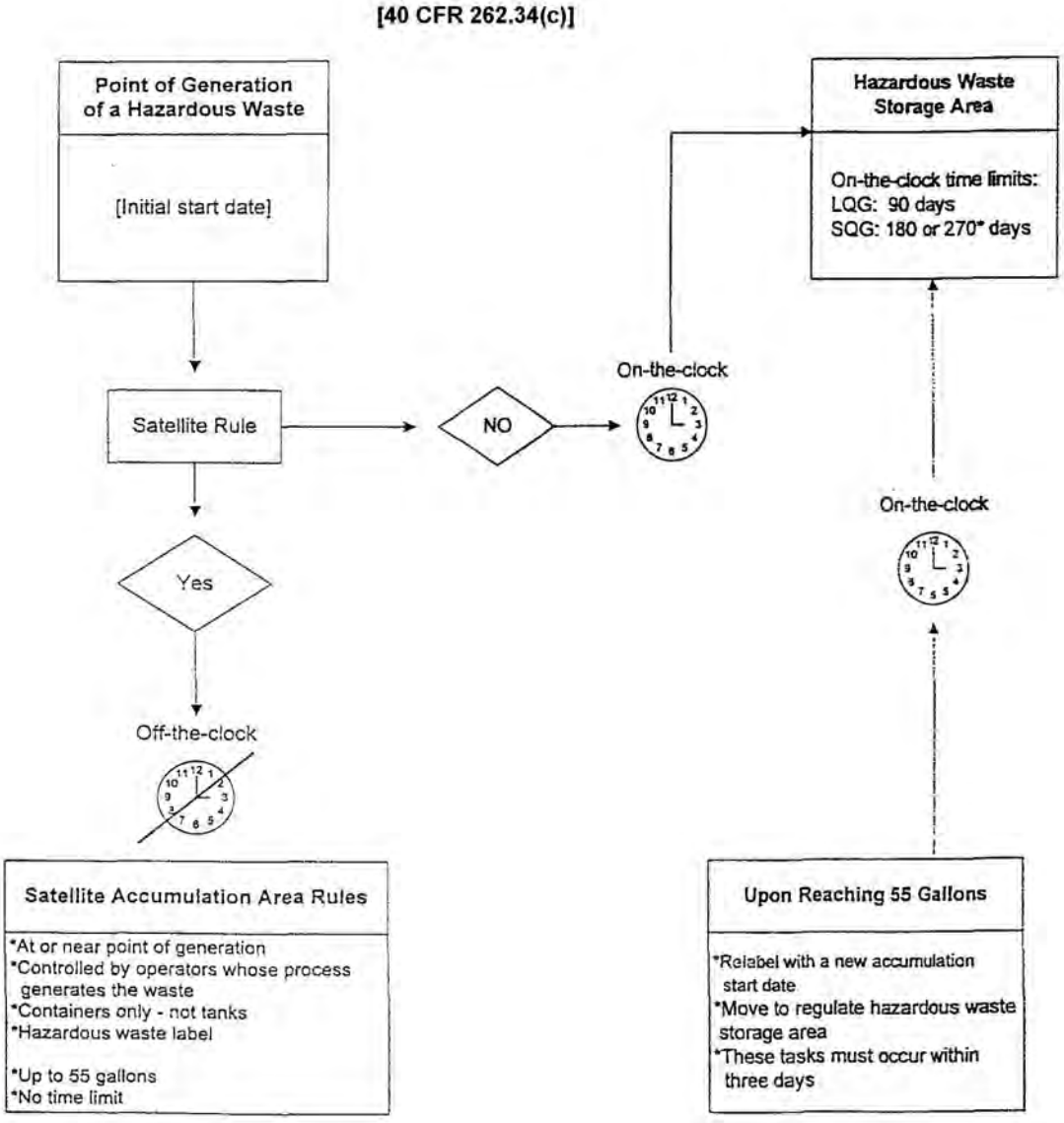
The following specific chemical and physical property discharge limits are typical of limitations imposed on users of the wastewater system:

Constituent	Discharge Limits (mg/L or ppm)
Arsenic	1.0
Cadmium	0.8
Total Chromium	2.5
Copper	2.0
Lead	0.6
Mercury	0.01
Nickel	2.0
Silver	0.5
Zinc	5.0
Cyanide (total)	2.5
Selenium	0.1
Phenols	1.0
Total Identifiable Chlorinated Hydrocarbons	0.05
Boron	1.0
Fluoride	3.0

Restrictions also usually include the following prohibitions on discharges:

- Having a temperature higher than 150°F (65°C).
- Containing more that 25mg/L of oil and grease or petroleum or mineral origin, or 175mg/L of oil and grease of animal or vegetable origin.
- Having a pH of less than 6 or greater than 11.5.
- Containing suspended solids greater than 500mg/L.
- Having a standard five-day biochemical oxygen demand more than 600mg/L.
- Containing a dissolved sulfite content higher than 1.0mg/L.
- Containing a TDS (total dissolved solids) content that exceeds 1000mg/L or the potable water supply TDS by more than 250mg/L, whichever is greater.
- Containing a chloride content that exceeds 200mg/L or the potable water supply by more than 50mg/L, whichever is greater.

Figure 8.1: APPLICATION OF SATELLITE RULE TO POINTS OF GENERATION



*270 days if waste must be transported 200 miles or more for treatment or disposal.

Figure 9: INSPECTION FORMS AND INSTRUCTIONS FOR USE

1. Inspection Log. Figure 9.1

Use of the Inspection Log

- The log should be kept in a secure central location or at a secure location near the storage area.
- The person responsible for the inspection should assure all deficiencies are corrected and properly documented.
- Inspections must be performed weekly. Note: Weekly means every seven days, not once per week irrespective of the day.

Recordkeeping

Documentation of inspections of hazardous waste storage areas must be maintained for at least three years from the date of the inspection. This recordkeeping requirement applies to the log and the checklists used to conduct the inspection.

Figure 9.1: TESTAMERICA SAVANNAH MAIN WASTE STORAGE AREAS WEEKLY INSPECTION FORM

TestAmerica Savannah Main Waste Storage Areas
Weekly Inspection Form

Name of Inspector (Print):		**Signature of Inspector:**	
Date:		**Time:**	
Number of open drums of waste:		**Number of closed drums of waste:**	**Number of total drums of waste:**

Yes	No	
		Are the waste containers in good condition?
		Are the containers closed?
		Are incompatible materials stored separately?
		Are the containers properly labeled?
		Are the labels clearly visible?
		Is the accumulation start date clearly indicated?
		Is the waste tracking log up to date?
		Is there adequate aisle space?
		Is the waste area in acceptable condition?
		Is there sufficient waste to schedule a pick-up?

Comments:

Corrective Actions:

FAN149:11.03.11:2

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Figure 10: EXAMPLE OF UNIFORM HAZARDOUS WASTE MANIFEST

Please print or type. *(Form designed for use on elite (12 - pitch) typewriter)*

Form Approved. OMB No. 2050 - 0039 Expires 9 - 30 - 91

UNIFORM HAZARDOUS WASTE MANIFEST

1 Generator's US EPA ID No. | Manifest Document No. | 2. Page 1 of | Information in the shaded areas is not required by Federal law

3. Generator's Name and Mailing Address | A. State Manifest Document Number

B. State Generator's ID

4. Generator's Phone ( )

5. Transporter 1 Company Name | 6. US EPA ID Number | C. State Transporter's ID | D. Transporter's Phone

7. Transporter 2 Company Name | 8. US EPA ID Number | E. State Transporter's ID | F. Transporter's Phone

9. Designated Facility Name and Site Address | 10. US EPA ID Number | G. State Facility's ID | H. Facility's Phone

11. US DOT Description *(Including Proper Shipping Name, Hazard Class, and ID Number)*	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. Waste No.
GENERATOR a.					
b.					
c.					
d.					

J. Additional Descriptions for Materials Listed Above | K. Handling Codes for Wastes Listed Above

15. Special Handling Instructions and Additional Information

16. **GENERATOR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations.

If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; **OR,** if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

Printed/Typed Name | Signature | *Month Day Year*

TRANSPORTER

17. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | *Month Day Year*

18. Transporter 2 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | *Month Day Year*

FACILITY

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in item 19.

Printed/Typed Name | Signature | *Month Day Year*

EPA Form 8700 - 22 (Rev. 9 - 88) Previous editions are obsolete.

APPENDIX B: TESTAMERICA SAVANNAH SAMPLE DISPOSAL TRACKING SYSTEM

TestAmerica Savannah's Sample Disposal Tracking System is integrated with the TALS sample login to ensure cradle-to-grave custody of samples. The TALS notifies departments, sample custodians and project managers when samples are ready to be disposed of and ensures the proper client specified disposal method is used. TestAmerica offers the following disposal methods:

1. Disposal with normal laboratory waste stream (default)
2. Return to client
3. Disposal at hazardous waste disposal facility

Thirty days after the invoice date samples will show up on sample disposal sheets. All samples are sorted by disposal method on disposal sheets. Sample ID's remain on disposal sheets until department personnel or sample custodian record that samples have been properly disposed. The TALS gives the laboratory the ability to look up the disposal history of a particular log number and sample ID.

Project Managers should discuss disposal options with clients prior to the start of all projects. It is important that the correct sample disposal code is entered in the TALS at login. The TestAmerica default disposal code is HW-RTC. Disposal codes may be modified after login. All samples associated with a given log number will automatically update if disposal code is modified after login. Sample disposal personnel may only use TALS generated disposal instructions. Verbal communication to disposal personnel concerning disposal status is forbidden. It is the project manger's responsibility to keep disposal codes up-to-date in the TALS.

All results for samples analyzed by a particular method are checked against corresponding regulatory limits by the TALS. If any result for a particular sample exceeds regulatory limits, the sample will be tagged as HAZ (Hazardous) by the TALS. If all results for a sample are less than the regulatory limits then the sample will be tagged as UNCONFIRMED in the TALS.

NON-HAZARDOUS SHEETS *(Laboratory Waste Stream Disposal)*
All samples which appear on these sheets should be disposed of in the regular laboratory waste stream. Liquids must be neutralized if preserved with acid and sent down sanitary sewer. Soils will be composited for testing and sent to a landfill or incinerator if deemed non-hazardous by testing. The criteria for samples on non-hazardous sheets are as follows:

1. Sample must have a status of UNCONFIRMED code.
2. Sample disposal code must not contain a code of Hazardous or HOLD.
3. Sample matrix or description must not be QC related.
4. Invoice date must be 30 days or greater.
5. Sample must not be 100% consumed by analysis.

SPECIAL DISPOSITION SHEETS
All samples appearing on special disposition sheets may not be disposed in regular laboratory waste stream. Samples, depending on the disposal code, will be held, returned to client, or set aside for licensed waste hauler to pick up. The criteria for samples on special disposition sheets are as follows:

1. Samples which have a status of "HAZ" or have a disposal code of HOLD or All-RTC.
2. Sample matrix or description is not QC related.
3. Invoice date must be 30 days or greater.

4. Sample must not be 100% consumed by analysis.

DEPARTMENTAL, REMOTE & CUSTODIAL SHEETS

Once the special disposition and non-hazardous list have been created by the TALS, individual department disposal sheets are created and printed. Currently, Metals, Volatiles, Semivolatiles, and General Departments receive both non-hazardous and special disposition sheets. Remote sheets will also print by department to show samples from other divisions ready for disposal. Finally, master custodian sheets (special disposition sheets only) will print to ensure all containers from a particular sample-ID were disposed by all departments.

Section 14: BLOODBORNE PATHOGENS PROGRAM

Summary of Program

Bloodborne pathogens (BBPs) are infectious materials in blood that can cause disease in humans including hepatitis B and C and human immunodeficiency virus (HIV). Workers exposed to these pathogens risk serious illness or death. TestAmerica Savannah does not process human tissue or fluid samples; therefore, employee exposure to BBPs via this route of transmission does not exist.

Field services personnel, however, may work at sites located far away from medical facilities. As such field service personnel are required to maintain first aid and CPR training. For this reason, this BBP program has been developed by the laboratory.

Reference Documents and Regulations

This program is supplemented by Section 19 of the TestAmerica Environmental Health and Safety Manual (EHSM) and is based on 29CFR 1910.1030.

Exposure Control Plan

Affected Positions

Employees associated with the Field Department are the only employees assigned to Savannah who are required to maintain First Aid and CPR certification.

Affected Procedures

The laboratory does not handle any samples that could contain bloodborne pathogens. The bloodborne pathogen training is only necessary for the field staff due to the sampling contracts requiring them to maintain First Aid and CPR certification and not due to any sampling of bloodborne pathogen contaminated samples.

Affected Employees

The Savannah Training Matrix lists all employees required to have First Aid and CPR certification.

Supplemental Information

Refer to Section 19 of the EHSM for information of the following required topics:

- Methods of Compliance (Section 19.3)
- Hepatitis B Vaccination and Post Exposure Evaluation Follow-Up (Section 19.4)
- Communication of Hazards to Employee (Section 19.5)
- Training (Section 19.6)
- Recordkeeping (Section 19.7)
- Sharps Injury Log (Section 19.8)
  Note: A Sharps Injury Log is not applicable to the BBP Program employed at TestAmerica Savannah.
- Medical Declination (Section 19.9)

Section 15: HEARING PROTECTION PROGRAM

Exposure to high levels of noise can be detrimental to a person's hearing. As such, OSHA has established requirements for employee hearing protection as outlined in 29 CFR 1910.95. This regulation establishes a noise threshold that employees can be exposed to without implementation of hearing protection. A Hearing Protection Program must be established, if it is determined that an area or piece of equipment exceeds 50% of the noise threshold.

The laboratory's Hearing Protection Program is outlined in this section and is designed to meet the requirements of 29 CFR 1910.95 and Section 8.2.5 of the TestAmerica Corporate Environmental Health and Safety Manual (EHSM).

Summary of Program
In the Hearing Protection Program, the laboratory conducts noise studies to identify equipment that could exceed the exposure limits defined in the OSHA standard. If the level of exposure equals or exceeds an 8-hour Time Weighted Average (TWA) of 85 decibels, or a single dose of 50% (i.e., 42.5 decibels), employees must wear ANSI-approved hearing protection devices (i.e., ear plugs or muffs). This level is referred to as the Action Limit.

Employees who work in these identified areas must undergo baseline hearing exams (i.e., audiometric testing) and annual hearing exams, at no cost to the employee. If the results of the annual audiogram indicate a shift in hearing threshold, greater than 10dB, the employee must be notified within 21 days. If it is determined that this threshold shift is related to workplace exposure, then additional administrative and/or engineering controls must be taken (e.g., retraining; re-fitting of hearing protectors; implementation of more robust hearing protectors; etc.).

> Note: Testing to establish a baseline audiogram shall be preceded by at least 14 hours without exposure to workplace noise. Conversely, ear plugs/muffs may be used as a substitute, prior to the test, to reduce workplace noise.

Other considerations:
- Noise testing must be performed using a calibrated noise meter. Monitoring shall be repeated whenever a change in production, process, equipment or controls increases noise exposures. This testing can be performed in conjunction with the Area Safety Analysis.
- If the Action Limit is exceeded, employees must be notified of the results of the testing,
- Employees may elect to observe the testing.
- Annual training must be conducted on this program. This training must include the list of affected equipment and/or areas; the effects of noise on hearing; the purpose of hearing protectors; instructions on the use, care, and fit of hearing protectors; the purpose of the audiometric testing; and an explanation of the test procedures.
- A copy of the OSHA cited standard must be made available to employees during this training. (Note: A copy of the OSHA standard (i.e., 29 CFR 1910.95) is available on the internet.)

Affected Areas
The laboratory has not identified any areas that exceed the Action Limit.

Section 16: REVISION HISTORY

Summary of Changes from Previous Revision:

- Minor grammatical, editorial, and formatting changes made throughout document, including update of personnel titles.
- Included Quick Reference Guide
- Updated emergency contact list. Section 3b
- Revised Document Location Matrix. Section 11

Eurofins Test America SOPs

Seattle

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Laboratory Waste Management and Disposal

Approvals:

Signatures on File

Manjit Nijjar Date
Health & Safety Manager / Coordinator

Dennis Bean Date
Laboratory Director

Manjit Nijjar Date
Quality Assurance Assistant
For Terri Torres
Quality Assurance Manager

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 PURPOSE

This Standard Operating Procedure (SOP) provides the necessary information required for compliance with TestAmerica's Environmental Health and Safety Manual. The SOP lists disposal procedures for client samples and chemical waste, safety concerns and the documentation needed for these disposal activities at the Seattle/Tacoma location.

2.0 SCOPE

2.1 This procedure is directed to employees trained in the handling of hazardous waste.

2.2 TestAmerica Seattle generates waste from sample analysis, unused sample(s), and out-of-date / off-specification chemicals, reagents and standards.

2.3 A concerted effort shall be given to determine if a waste generated by the laboratory (refer to Section 4.10) is a listed waste.

2.4 The laboratory is not a hazardous waste transporter, storage, or disposal facility (TSD). Nor will the laboratory treat, store, or transport hazardous waste without a permit, unless allowed by federal, state, and local laws and regulations.

2.5 All waste being packaged and shipped for disposal shall only be conducted by trained personnel (according to 29 CFR 1910.120.

2.6 Hazardous waste shall be sent to an approved TSD. Approval is given by federal and state agencies as well as internal company approval.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

3.1 All federal, state, and local regulations shall be followed at all times.

3.2 Any employee working in an atmosphere that has chemical contaminates above the permissible exposure limits is required to wear an approved respirator (air purifying or supplied air).

3.3 All employees will be trained in the use of a respirator if required to wear a respirator.

3.4 All respirators will be maintained according to 29 CFR 1910.134.

3.5 Any chemical release that cannot be controlled by the hazardous waste workers will be handled in the following manner (as necessary):

3.5.1 Immediate evacuation of the facility.

3.5.2 Call 911

3.5.3 Contact PSC Emergency Spill Services at 1-877-577-2669

3.5.4 Give first aid to all injured employees or / and transport employee(s) to the appropriate medical facility.

3.5.5 Notify your local EHSC and the corporate EHSO about the incident and the actions that have been taken to rectify the situation.

3.5.6 If the hazardous material was released to the environment at or near reportable quantity, notify the National Response Center. 1-800-424-8802

3.5.7 Keep employees away from the building until the fire department deems the building safe to reenter.

3.5.8 Submit a written report of the incident to corporate health and safety and to the fire department.

3.5.9 Notify OSHA within 48 hours when 5 or more personnel become ill due to exposure or within 8 hours if a fatality is involved.

3.6 OSHA Requirements

3.6.1 The hazardous waste manager will have 24 hours of training in accordance with 29 CFR 1910.120. This employee will also be fit tested (if applicable) and certified to wear a respirator (air purifying or supplied air).

3.6.2 All hazardous waste workers will be given at least 24 hours of training in accordance with 29 CFR 1910.120.

3.7 Respiratory Protection

3.7.1 The waste technician will utilize the hoods specifically set up for bulking hazardous waste into drums. This system will also be utilized when neutralizing acids or bases.

3.7.2 If the hood fails during bulking or neutralizing of waste, the waste technician will stop all bulking immediately and close all drums. The facility coordinator will be immediately notified so repairs can be made.

3.7.3 A respirator must be worn while dumping waste into the following drums, or the drums must be under a hood, or a carboy with a spigot must be used so that the employee can move away from the drum during the transfer.

- Acid Waste with Metals
- Flammable/Chlorinated or non Chlorinated Solvents/Contaminated solids
- Pyridine/Phenol
- Volatile and Semi Volatile Organic Compounds
- Loose pack Vials – Poison
- Loose pack Vials – Flammable/Corrosive Liquids

3.7.4 A self contained breathing apparatus must be worn when bulking Methylene Chloride or the bulking must take place under a laboratory hood meeting the minimum requirements for hood flow at 80 ft/min.

3.7.5 Samples may contain high concentrations of hazardous and or toxic compounds. Analytical lab waste and expired chemicals will generally have physical and health hazardous characteristics. i.e. flammable, carcinogen.

3.7.6 Store liquid incompatible wastes on separate spill containment pads.

3.7.7 Heat producing, sparking, or open flamed materials or equipment are prohibited in the flammable liquid waste area.

3.7.8 Stacking full plastic 20 gallon barrels more than three high or any stacking of full 55 gallon barrels is prohibited.

3.7.9 Adequate ventilation or proper respiratory protection will be used when working with waste (such as lab packing or bulking solvents).

3.7.10 Grounding of flammable liquid drums is accomplished by placing the metal drums on the metal grates, which are connected to grounding wires.

3.7.11 Keep adequate space between barrels for leak checking and safe movement.

3.7.12 Keep a 30 inch clearance space around the perimeter of the waste barrels and the 90 day spill containment pad.

3.7.13 Ensure all safety equipment is functional and accessible before engaging in waste activities.

3.7.14 Lab coats, safety glasses or goggles, and specific chemical resistant gloves are required while handling or disposing any waste chemicals or samples designated for disposal. Respirators, lab aprons, and earplugs may also be required. Steel toed boots are required for moving 55 gallon barrels.

3.7.15 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the operations in different areas of the laboratory, and take appropriate precautions and wear appropriate attire and safety glasses.

3.7.16 Waste personnel are never allowed to work alone without a means to communicate the need for help.

3.7.17 The door between the waste disposal area and the back lab must be kept open at all times the waste technician is disposing of samples or chemicals. This is to provide unimpeded access to the safety shower.

3.8 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acid Waste with Metals	CORROSIVE		Acids cause burns upon contact or inhalation. Vapors are irritant to eyes. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
Flammable/ Chlorinated or non Chlorinated Solvents	Flammable, Poison		Symptoms of overexposure may include headache, drowsiness and dizziness. Solvents maybe a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
Pyridine/ Phenol	Poison, Flammable liquid and vapor		Symptoms of overexposure may include headache, drowsiness, dizziness, and nausea. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
Volatile and Semi Volatile Organic Compounds	Flammable, Corrosive, poison		Symptoms of overexposure may include headache, drowsiness and dizziness. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
Loose pack Vials – Poison	Poison, Flammable liquid and vapor		Symptoms of overexposure may include headache, drowsiness and dizziness. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
Loose pack Vials – Flammable/ Corrosive Liquids	Corrosive, poison		Symptoms of overexposure may include headache, drowsiness and dizziness. Solvents maybe a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
Contaminated Solids	Poison		Symptoms of overexposure may include headache, drowsiness, dizziness, and nausea. Do not allow contact with skin, mucus membranes, or eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 DEFINITIONS

4.1 **Liquid** - is a material that has a vertical flow of over two inches within a three minute period, or a material having one gram or more liquid separation, when determined in accordance with the procedures specified in ASTM D 4359-84, "Standard Test Method for Determining Whether a Material is a liquid or Solid," 1984 Edition. (49 CFR).

4.2 **Solid** - is a material that has a vertical flow two inches or less within a three minute period, or a material having one gram or less liquid separation, when determined in accordance with the procedures specified in ASTM D 4359-84, "Standard Test Method for Determining Whether a Material is a Liquid or Solid," 1984 Edition. (49 CFR).

4.3 **Organics** - (For this document) are materials that are primarily composed of carbon based compounds. Carbonates are an exception for this definition.

4.4 **Inorganics** - (For this document) are materials that are primarily composed of non-carbon based compounds. Carbonates are an exception for this definition.

4.5 **Acids** - (For this document) are substances that have a pH of 4 or less.

4.6 **Bases** - (For this document) are substances that have a pH of 10 or greater.

4.7 **TCLP (Toxicity Characteristic Leaching Procedure)** - is a simulated leaching procedure from 40 CFR 261 Appendix II to determine the toxicity characteristics of a liquid, solid, or multi-phased waste.

4.8 **Hazardous Waste** - See the definition in 40 CFR 261.3.

4.9 **Regulated Wastes** - are wastes that are governed by federal, state, or local regulations.

4.10 **Dangerous wastes** included federal hazardous wastes and wastes regulated only by Washington State.

4.11 **Listed Wastes** - are wastes that are specified in 40 CFR 261 Subpart C by a specific name (such as carbon disulfide; P022) or by a process (such as bottom stream from an acetonitrile column in the production of acrylonitrile; K013). There are five lists for waste: D list, characteristic waste; F list, wastes from non-specific sources; K list, wastes from specific sources; U list (toxic) and P list (acutely toxic), discarded commercial chemical products, off-specification species, container residues, and spill residues thereof.

4.12 **Lab Pack** - are small containers of hazardous waste placed into a larger container (over pack).

4.13 **Waste Streams** - are collected hazardous wastes from one or more sources to create a single generic source of hazardous waste (such as chlorinated solvent wastes from organic extractions, wet chemistry, and GC/VOA analysis will be combined to form a laboratory chlorinated solvent waste stream).

4.14 **TSD Facility** - is a treatment, storage, and disposal facility. A facility that treats, stores, and/or disposes of hazardous waste, that is permitted by the EPA (federal and/or state), and follows the criteria written in 40 CFR 264, 265, 266, 267, and 268.

4.15 **Waste Profile** - is the composition of a waste stream from chemical analysis or generator knowledge. The components of the waste stream will be shown as a maximum and minimum concentration since the components may vary.

4.16 **Satellite Accumulation Point** - is where waste can be accumulated (at the point of generation) in smaller containers, later to be brought to a designated waste area [as stated in 40 CFR 262.34(c)(1)]. Containers must still contain the words "hazardous waste" or the contents of the container.

4.17 **Elementary Neutralization Unit (ENU)** - is a unit that neutralizes wastes that are hazardous only because they exhibit the characteristic of corrosivity (D002).

4.18 **Uniform Hazardous Waste Manifest (UHWM)** - is the shipping document that must accompany all hazardous waste shipments to TSD facilities. (EPA form 8700-22 (REV 3-05)))

4.19 **Aqueous liquids** - are liquids that are water based.

4.20 **USDA –** US DEPARTMENT OF AGRICULTURE

4.21 **APHIS –** ANIMAL AND PLANT HEALTH INSPECTION SERVICE

4.22 **PPQ** – PLANT PROTECTION AND QUARANTINE

4.23 **PSS** – PERMIT SERVICES STAFF

5.0 PROCEDURE

5.1 Disposal Log

5.1.1 Each sample must be reviewed for proper disposal. This information can be found in the laboratory LIMs system, TALS, under the sample management section. TALS contains information critical to the tracking of the samples as they pass from sample storage to disposal. TALS contains information on which samples have been rotated and when.

5.1.2 All refrigerated client samples are to be kept in the laboratory refrigerators for a minimum of fourteen days. Ideally if space allows, the samples should remain in refrigeration until disposal. The status of every sample must be verified completely before disposal.

5.1.3 Before any samples can be disposed of, these two considerations must be made:

- Client samples must be kept for a minimum of thirty days from completion/invoicing of the final report.
- The sample's status must be such that we no longer need the sample.

5.2 Pulling Samples and Documentation

- A query against the TALS database will be used to determine the samples that can be rotated out of sample storage.
- It is important to check and remove samples regularly from the various refrigerators to free up space for incoming samples and reduce disorder especially during peak sampling periods.
- Take the list of locations with invoiced containers, a waste disposal cart, and a pen and proceed to the sample storage area in need of rotation.
- Take the sample-filled cart to the waste disposal warehouse for segregation for further storage, disposal or archiving.
- If the samples that are being rotated are less then 30 days after the invoice date, they will not be eligible for disposal.
- Place these samples in boxes (or leave them in the storage container) marked with the date they were pulled from sample storage and place them on the sample processing shelves.

PLEASE NOTE:

At this point, it is important to note in the midst of any sample refrigerator, there are samples labeled "**ARCHIVE**". Some archived samples must remain in cold storage. Archive samples have their own disposal timeline and disposal procedures that must be followed. If you come across any archive samples, they must be put aside to prevent accidental disposal.

5.3 Foreign Samples

Foreign samples are kept in a locked refrigerator separate from other samples. Foreign samples must be disposed of in accordance with TA-QA-0531

5.4 Sample Disposal and Documentation

Proper disposal of all samples must be performed to minimize environmental hazards and personal health risks.

Samples that have been stored for 30 days past the invoice of the project may be eligible for disposal.

Collect a group of samples that have been sorted by matrix. i.e. a group of water samples that if they are eligible for disposal and are non hazardous will be bulked into the neutralization tank, neutralized and then released into the public sewer system.

Separate out all samples that do not fit into the waste stream. Mark the sample with its hazardous constituent, i.e. Hg or PCB's.

Scan out all the samples using the internal chain of custody (ICOC) program in the LIMS system. See Appendix A for directions on how to use the ICOC.

Remove any samples from the group that cannot be disposed of. Refer to Appendix B for instructions on what to do with these samples.

The remaining samples can them be bulked into their appropriate waste stream.

5.4.1 Soil samples After segregating any samples that should not be disposed of into the "toxic solids waste stream, place the remaining samples in the cubic yard box for toxic solids.

5.4.2 Water Samples: Waste waters, drinking waters, ground waters and surface waters are all disposed of via the sanitary sewer system. Aqueous samples that have a pH below 6 or above 9 must be neutralized before disposal into the local sanitary sewer system. The containers are rinsed and sent out for recycle.

With the exception of the following:

- Any water known or suspected to contain organic materials in concentrations above permitted limits (according to the Pierce County Sewer Limits and / or RCRA).
- Any water known or suspected to contain heavy metals in concentrations above permitted limits (according to the Pierce County Sewer Limits and / or RCRA).
- Any water with only the characteristic of corrosivity unless the liquid is neutralized in an elementary neutralization unit.
- Any water known or suspected to contain solids not permitted by the Pierce County Sewer Limits, unless the solids are filtered.
- Any other contaminates in water that would prohibit disposal in a sanitary sewer by the Pierce County Sewer Limits

- Water samples marked with specific hazardous contaminates are bulked into separate drums specific to its contaminate.

5.5 Solids (Soils, Sludges and Solids)

5.5.1 All solid samples (and their containers) will be placed into the bulk waste stream "*soil samples and debris*" with the following exceptions:

- All solids known or suspected to contain PCBs at or above 50 ppm are segregated from the "waste solids and soils" bulk waste stream, and placed in the PCB waste stream.
- Any solid that cannot be treated by incineration.
- Any solid known to be a P listed waste.
- Any solid known or suspected to contain dioxins or dioxin precursors (such samples will be returned to the client).
- Any solid with mercury above 0.2 ppm.

5.5.2 All solids prohibited from the "*soil samples and debris*"bulk stream will be segregated by the solid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

5.5.3 **Soil samples** After segregating any samples that should not be disposed of into the “toxic solids waste stream, place the remaining samples in the cubic yard box for toxic solids **Other Wet and Other Dry** These samples may not fit into our bulk waste streams and will have to be lab packed for disposal.

5.5.4 **Waste Profiles** A copy of Seattle/Tacoma’s current waste file may be found in the file cabinet in the inorganic department manager’s office.

5.6 Removing Foreign and Regulated Domestic Soil Samples and Water Residues from Storage

- Only authorized personnel are permitted access to foreign and regulated domestic soil samples in the locked refrigerator.
- All foreign and regulated domestic soil samples must be heat treated (autoclaved) or sent out for incineration to a permitted facility.

5.7 Archive Sample Disposal

Archive samples are samples that require extended storage beyond the thirty days from completion, as requested by Project Managers or clients.

You must have written or electronic approval from the Project Manager to dispose of any archived samples.

5.7.1 Other Wet and Other Dry Sample Matrices Disposal

Samples with these matrices may need further review to determine how they will be disposed. This additional information can be obtained by looking at the chain of custody and by speaking with the project manager. The project manager often knows what the client is analyzing and can identify the matrix. Lastly, the client may be queried as to the contents of their sample. Project management’s authorization is required before the waste disposal department contacts a client.

5.8 Bulking Chemical Waste and Samples with Hazardous Constituents

5.8.1 Accumulation of Bulk Waste

5.8.1.1 Waste is accumulated in a designated waste area. This area must meet the requirements of 40 CFR 262 (Standards Applicable to Generators of Hazardous Waste). Washington Rules and Regulations.

5.8.1.2 Since waste is generated in operation areas outside the designated waste area, satellite accumulation points may be set up in the laboratory using containers less than one quart for acutely toxic waste or a maximum of 55 gallons for other waste. All laboratories will maintain satellite accumulation points that meet or exceed the requirements of 40 CFR 262.34.

5.8.1.3 Individuals using satellite accumulation points will be trained in the proper use and function of the satellite accumulation point.

5.8.1.4 Satellite Waste containers will be emptied no more than 72 hours after a container is filled.

5.8.1.5 Waste containers will be labeled with the words Hazardous Waste or their contents, the date the container reached 1 quart for acutely hazardous, 55 gallons for hazardous, or when any size container is brought back to the disposal warehouse. All liquid waste shall be in secondary containment or placed on the spill containment pad.

5.8.1.6 Non compatible wastes will be stored in separate secondary containment units.

5.8.1.7 Some Satellite waste containers are taken to the designated waste area, and bulked into larger drums and the empty container returned to the satellite accumulation point. Some satellite waste containers are sent out for disposal without transfer to a bulk drum. When this is the case a new satellite waste container must be placed where the old one was removed from.

5.8.2 Collect waste from satellite locations.

5.8.2.1 Segregate chemical waste, client samples and expired chemicals according to compatibility.

5.8.2.2 Standard waste streams for the laboratory are:

- High Metal waste contains liquids that have above regulatory levels of metals in them. The matrix is usually acidified water with a ph level less than two. The acidity is due to the acid preservative, usually hydrochloric acid or nitric acid. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors.
- High VOC waste contains liquids that have one or more of the following characteristics: flammable, volatile, toxic, carcinogenic, and acidic (pH level less than two due to the added preservative. Always keep away from ignition sources. Do not allow contact with skin, mucus membranes, or eyes. Do not inhale vapors. Methylene chloride waste stream also contains other halogenated and non halogenated solvents. Acetone, hexane, and methanol are flammable, volatile liquids. Always keep away from ignition sources. GC Vials containing solvents, chemical spikes and client samples. Glass vials containing sulfuric acid, hexane, methanol and soil sediments are collected separately. Solids contaminated with hazardous or toxic constituents.

5.8.2.3 Client samples, lab waste and expired chemicals that do not fit into these waste streams are lab packed according to compatibility.

5.8.2.4 Satellite waste containers that are 5 gallons or less in size may be transported on a cart with closed sides. Satellite containers larger than 5 gallons need to be transported with a drum dolly.

5.8.3 Sources for these waste streams are:

5.8.3.1 Wet Chemistry

A) All aqueous liquids from the analysis of cyanides, nitrates, nitrites, alkalinity, ammonia, BS&W, corrosivity, density, TOC, fluoride, BTU, iodine, oil and grease, petroleum hydrocarbons, phosphates, settleable solids, suspended solids, sulfides, total inorganic carbon, turbidity, and any other wet chemistry analysis are disposed of into the wastewater stream or corrosive waste stream, depending on the pH of the waste. Use Poly containers for these waste streams. A Poly barrel is used to bulk the necessary satellite waste streams for offsite shipment.

B) All aqueous liquids from the analysis of BOD's are discarded via the sanitary sewer with following exceptions:

Any aqueous liquid known or suspected to contain organic materials in concentrations above permitted limits (according to Pierce County Sewer Limits and / or RCRA).

- Any aqueous liquid known or suspected to contain heavy metals in concentrations above permitted limits (according to Pierce County Sewer Limits and / or RCRA).
- Any aqueous liquid with only the characteristic of corrosivity unless the liquid is neutralized in an elementary neutralization unit.
- Any aqueous liquid known or suspected to contain solids not permitted by the Pierce County Sewer Limits, unless the solids are filtered.
- Any other aqueous liquid prohibited by the Pierce County Sewer Limits for disposal in a sanitary sewer.

C) All aqueous liquids prohibited from the sanitary sewer will be segregated by the liquid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

D) All solid debris from any wet chemistry analysis are discarded via the normal trash as this material is non regulated with the following exceptions:

E) Any solid with heavy metal concentrations above the following limits: arsenic 100 ppm; barium 5000 ppm; cadmium 500 ppm; chromium 3000 ppm; lead 5000 ppm; mercury 0.2 ppm; selenium 1 ppm; zinc 3000 ppm; silver 500 ppm.

F) Any solid with a PCB concentration at or above 50 ppm.

G) Any solid debris prohibited from being disposed of via normal trash will be segregated by the solid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

H) All out dated standards and reagents, off specification chemicals, and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable U and P listings shall accompany the wastes.

5.8.3.2 Metals Digestion

A) All aqueous liquids from the digestion of samples for metals analysis shall be placed in either the elementary neutralization unit or the corrosive waste stream with the following exceptions:

- Any aqueous liquid known or suspected to contain organic materials in concentrations above permitted limits (according to Pierce County Sewer Limits and / or RCRA).
- Any aqueous liquid known or suspected to contain heavy metals in concentrations above permitted limits (according to Pierce County Sewer Limits and / or RCRA).
- Any aqueous liquid known or suspected to contain solids not permitted by Pierce County Sewer Limits unless the solids are filtered.

B) All aqueous liquids that would be prohibited from the sanitary sewer after treatment in the ENU will not be treated, but the liquids will be segregated by the liquid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

C) All solid debris from the digestion of samples for metals analysis is disposed of by way of the toxic solid and soil waste stream.

D) All out dated standards and reagents, off specification chemicals, and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable EPA U and P listings shall accompany the wastes.

E) The satellite containers for High Metal liquid waste are located on the counters in the metals lab. Use Poly containers for this waste stream. Metal barrels are not used due to the acid preservatives. Poly barrels are used for bulking this satellite waste stream for offsite shipment.

5.8.3.3 TCLP Extraction

A) All aqueous liquids from the TCLP extraction process are discarded via sanitary sewer or in non-regulated waste streams with the following exceptions:

- Any aqueous liquid known or suspected to contain organic materials in concentrations above permitted limits (according to the Pierce County Sewer Limits and / or RCRA).
- Any aqueous liquid known or suspected to contain heavy metals in concentrations above permitted limits (according to Pierce County Sewer Limits and / or RCRA).
- Any aqueous liquid with only the characteristic of corrosivity unless the liquid is neutralized in an elementary neutralization unit.
- Any aqueous liquid known or suspected to contain solids not permitted by the Pierce County Sewer Limits, unless the solids are filtered.
- Any other aqueous liquid prohibited by the Clean Water Services Rules, Regulations, and Conditions of Service for disposal in a sanitary sewer.

B) All aqueous liquids prohibited from the sanitary sewer will be segregated by the liquid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

C) All solid wastes from any extraction's analysis are placed into normal trash with the following exceptions:

- Any solid with heavy metal concentrations above the following limits: Arsenic 500 ppm; barium 1000 ppm; cadmium 100 ppm; chromium 500 ppm; lead 500 ppm; mercury 0.2 ppm; selenium 100 ppm; zinc 1000 ppm; silver 500 ppm.
- Any solid with a PCB concentration at or above 50 ppm
- Any solids prohibited from the "*soil samples and debris*" bulk stream will be segregated by the solid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.
- All out dated standards and reagents, off specification chemicals, and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable U and P listings shall accompany the wastes.

5.8.3.4 Organic Extractions

A) All aqueous liquids from the extraction process are disposed of into the wastewater stream with the following exceptions:

- Any aqueous liquid known or suspected to contain heavy metals in concentrations above permitted limits (according to RCRA Regulations).
- Any aqueous liquid with only the characteristic of corrosivity unless the liquid is neutralized in an elementary neutralization unit.
- All aqueous liquids will be segregated by the liquid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

B) All solid wastes are placed into the bulk waste stream "*soil samples and debris*" with the following exceptions:

- Any solid with heavy metal concentrations above the following limits: Arsenic 500 ppm; barium 1000 ppm; cadmium 100 ppm; chromium 500 ppm; lead 500 ppm; mercury 0.2 ppm; selenium 100 ppm; zinc 1000 ppm; silver 500 ppm.
- All solids known or suspected to contain PCBs above 50 ppm are segregated from the "waste solids and soils" bulk waste stream, and placed in the PCB waste stream.
- Any solid that cannot be treated by incineration.

C) All solids prohibited from the "*soil samples and debris*" bulk stream will be segregated by the solid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

D) All out dated standards and reagents, off specification chemicals, and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable U and P listings shall accompany the wastes.

E) All solvents and solvent blends shall be segregated into bulk streams as follows:

- Waste flammable liquid: Acetone, hexane, ethyl ether, petroleum ether, acetonitrile, cyclohexane, methanol, hexane / ethyl ether blend, ethyl ether / petroleum ether blend, and petroleum ether / acetonitrile blend, Waste dichloromethane; dichloromethane from glassware and equipment rinsing, Waste flammable chlorinated liquids:

dichloromethane/hexane blend, dichloromethane/hexane/acetonitrile blend, dichloromethane/cyclohexane blend, dichloromethane / acetone blend.

- Waste PCBs: any solvent that contains PCBs at or above 50 ppm or any solvent that was a diluent for a substance that contained 50 ppm or greater of PCBs.
- The satellite waste containers for Methylene Chloride and flammable solvents are located under hood numbers 8, 9, 12, 14 inside the Extractions lab. Metal receptacles specifically made for storing flammables are the best choice of container for this waste stream. Metal barrels are used to bulk this satellite waste stream for offsite shipment.

F) All other miscellaneous solvents shall be lab packed or placed in bulk waste streams on a case by case basis.

5.8.3.5 PCB / Pesticide, Herbicide, PAHs, and VOA Analysis

A) All out dated standards and reagents, off specification chemicals, and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable U and P listings shall accompany the wastes.

B) All out dated standards and reagents, off specification chemicals and container residues that contained PCBs at or above 50 ppm or was a dilution of a material that contained PCBs above 50 ppm shall be disposed of in the PCB waste stream along with all other applicable D, F, U and P listings.

C) The extracts from PCB analysis that contain sulfuric acid and hexane are to be collected at a satellite accumulation point until there is sufficient volume to fill a 55 gallon drum which is then sent out for disposal with a TSD when it is full.

D) All auto sampler vials are to be placed in a bulk waste stream with the applicable D, F, U, and P listings.

E) The satellite containers for VOC vials are located under the bench in the VOA lab.

F) Glass vials containing sulfuric acid, hexane, methanol and soil sediments are collected separately at a satellite accumulation point until there is sufficient volume to fill a 55 gallon drum which is then sent out for disposal with a TSD when it is full.

G) The satellite waste containers for Methylene Chloride and flammable solvents are located under the hood inside the Extractions lab. Metal receptacles specifically made for storing flammables are the best choice of container for this waste stream.

H) A metal barrel is used to bulk this waste for offsite shipment.

5.8.3.6 GC / MS Analysis

A) All out dated standards and reagents, off specification chemicals, and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable U and P listings shall accompany the wastes.

B) All auto sampler vials are to be placed in a bulk waste stream with the applicable D, F, U, and P listings.

C) All extracts are to be placed into the appropriate lab pack or bulk waste stream with any applicable D, F, U, and P listings. The satellite containers for this waste stream are located under the benches holding the GC's. Poly containers or rubber coated glass

are used for this waste stream. Either a poly or a metal drum with a liner may be used for bulking this satellite waste for offsite shipment.

5.8.3.7 Metals Analysis

A) All aqueous digestion liquids shall be placed in an elementary neutralization unit and disposed of via the sanitary sewer or placed in the corrosive waste stream with the following exceptions:

- Any aqueous digestion liquid known or suspected to contain organic materials in concentrations above permitted limits (according to the Pierce County sewer limits and / or RCRA).
- Any aqueous digestion liquid known or suspected to contain heavy metals in concentrations above permitted limits (according to the Pierce County sewer limits and / or RCRA).
- Any aqueous digestion liquid known or suspected to contain solids not permitted by the Pierce County sewer limits, unless the solids are filtered.

B) All aqueous digestion liquids that would be prohibited from the sanitary sewer after treatment in the ENU will not be treated, but the liquids will be segregated by the liquid's hazardous characteristics or chemical constituents and placed into the appropriate bulk streams or lab packs.

C) The satellite container for High Metal liquid is located on the counter in the Metals Prep lab with the exception of high mercury waste which is located in the metals analysis room. Use Poly containers for this waste stream. Metal barrels are not used due to the acid preservatives. A Poly barrel is used to bulk this satellite waste for off site shipment.

D) All out dated standards and reagents, off specification chemicals and container residues shall be disposed of in a proper manner according to 40 CFR and all applicable U and P listings shall accompany the wastes.

6 SAMPLE DISPOSAL

6.1 Water Samples

6.1.1 Waste waters, drinking waters, ground waters and surface waters are all disposed of via the sanitary sewer system or via non regulated wastewater or through an evaporator with exception of the following:

- Any water known or suspected to contain organic materials in concentrations above permitted limits (according to the Pierce County Sewer Limits and / or RCRA).
- Any water known or suspected to contain heavy metals in concentrations above permitted limits (according to the Pierce County Sewer Limits and / or RCRA).
- Any water with only the characteristic of corrosivity unless the liquid is neutralized in an elementary neutralization unit.
- Any water known or suspected to contain solids not permitted by the Pierce County Sewer Limits, unless the solids are filtered.
- Any other contaminates in water that would prohibit disposal in a sanitary sewer by the Pierce County Sewer Limits

6.1.2 All waters prohibited from the sanitary sewer or treatment through evaporation will be segregated by the liquid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

6.2 Solids (Soils, Sludges and Solids)

6.2.1 All solid samples (and their containers) will be placed into the bulk waste stream "*soil samples and debris*" with the following exceptions:

- All solids known or suspected to contain PCBs at or above 50 ppm are segregated from the "*soil samples and debris*" bulk waste stream, and placed in the PCB waste stream.
- Any solid that cannot be treated by incineration.
- Any solid known to be a P listed waste.
- Any solid known or suspected to contain dioxins or dioxin precursors (such samples will be returned to the client).
- Any solid with mercury above 0.2ppm.

6.2.2 All solids prohibited from the "*soil samples and debris*" bulk stream will be segregated by the solid's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

6.3 Food Products

6.3.1 All food products will be disposed of via sanitary landfill except for the following.

- Any food product that would be RCRA or TSCA (for PCB's) regulated or for any reason banned from a sanitary landfill.

6.3.2 All food products prohibited from the sanitary landfill will be segregated by the food's hazardous characteristics or chemical constituents, and placed into the appropriate bulk streams or lab packs.

6.4 Bulking Hazardous waste

55 gallon UN approved steel or poly barrels are the storage and shipment containers for waste in the designated waste area. Each barrel needs to be labeled with the appropriate DOT diamond label, the words "hazardous waste", the proper DOT shipping name for the waste (see 49 CFR 172.101), the UN or NA number, the name and address of the generator, the U.S. EPA generator number, the date the waste was first put into the barrel (accumulation start date), the EPA waste code (if applicable), and the manifest number when available. No label can cover the UN markings on the barrel, nor can any part of the DOT diamond label be covered.

6.5 Bulking Barrel Identification and Labeling

6.5.1 Choose either a metal or plastic (poly) barrel, or soil debris box based on chemical compatibility. This information can be obtained in this SOP for that particular waste stream or from the supervisor of the waste disposal department.

6.5.2 All barrels must have identification labels. These labels are to be placed on the middle section of the barrel and within 6 inches of each other.

6.5.3 All open overpack top barrels must have an "up arrows" label attached to them.

6.5.4 All barrels and soil boxes must have a label indicating either "Hazardous Waste" or "Non-Hazardous Waste". These labels will have open spaces to fill in. It is necessary to fill in the following sections:

- Generator information: write in "TestAmerica"
- EPA ID Number: write in WAH000015016
- Accumulation Start Date: write in the date the barrel was to be put into use.
- Barrel or Box Contents: write in the name of the waste stream
- Barrel or Box ID Number: See Appendix E

6.5.5 Hazardous waste barrels generally need an additional label(s) indicating the type of hazard. We use DOT labels to identify profile information such as flammable, corrosive, poisonous, etc.

6.6 Barrel ID Numbers:

6.6.1 Each barrel needs to have a unique TA number assigned to it. This number is determined in the following manner and recorded on the Waste Disposal Record. The number is next sequential number in the Waste disposal log book.

6.6.2 This type of identification number along with a start date will be placed on each barrel and each Waste Disposal Record form at the time the barrel first goes into service.

6.6.3 A list of barrel identification numbers will be kept in a logbook in the sample disposal room. Whenever a new barrel goes into service an entry onto this form must be made which corresponds to the identifying number assigned to the barrel. See Appendix E

6.6.4 Each unique barrel number will also be entered into the TALS LIMs systems internal change of custody. See Appendix A.

6.7 Required aisle space: a 30 inch aisle space must be maintained around the waste barrels and spill containment pad to allow for the unobstructed movements of personnel, fire protection equipment, spill control equipment, and decontamination equipment to any area of facility operation in an emergency,

6.8 Maintenance Log

6.8.1 Once a week, all barrels in the disposal warehouse must be inspected. The inspection includes checking for correct labeling and start dates, leaks, lids secure, length of time the barrel has been in use, * and whether the barrel is full and needs to be sent out for disposal.

*__NOTE__: Per EPA regulations, TestAmerica Seattle is a large quantity generator and can keep hazardous waste barrels for a period of 90 days from the start date. It is important to determine the plan for final disposal to always meet these EPA regulations.

6.8.2 All barrels must be kept closed unless in use.

6.8.3 Barrels that are leaking, severely dented, or the closures do not seal are not acceptable for shipment. The waste must be transferred from the damaged barrel as soon as possible. An empty damaged drum will be considered hazardous waste if it contained waste at one time.

6.8.4 Once a barrel is full (approximately one inch from the top or six inches for barrels that contain Methylene Chloride) and has met the requirements of this section, it is ready for shipment to a TSD facility.

6.9 Bulking Liquid Hazardous waste

6.9.1 See Appendix E for barrel set up and closure instructions.

Estimate the volume in gallons that will be dumped and enter it on the Drum Inventory sheet for that drum.

6.9.2 Record the following on the Drum Inventory sheet::

- Disposal Date
- Your Name
- Description of Waste

6.10 Bulking in the bulking hood.

- Move appropriate drum for the waste to be bulked into the bulking hood.
- Use a drum wrench to twist off the drum cap.
- Insert a funnel into the drum.
- Empty the contents into the appropriate waste drum. Do this slowly to prevent splashing. You must remove enough sample so that < 1% remains.
- After all samples are dumped, remove funnel, replace drum cap and secure it with drum wrench.

Drums with a liquid content must be stored on a spill containment rack.

6.11 Soil Sample Disposal Procedure

- This procedure applies to unregulated foreign and domestic soils.
- Soil samples are generally received in glass jars and metal cylinders.
- Soil samples, including their containers are placed in a cubic yard box located in the waste disposal warehouse.
- If the soil is non hazardous the sample maybe emptied into the cubic yard box and the container recycled.
- For soil samples that have hazardous contaminates, the entire container and its contents are placed into the appropriate waste drum.

6.11.2 Lab Packs: Other Wet and Other Dry Sample Disposal

These samples usually do not fit into our bulk waste streams and will have to be lab packed for disposal. An authorized trained technician evaluates samples for the sample's waste characteristics and lab packages them for disposal.

The following lab pack waste streams are generated at the laboratory:

- Corrosive liquids (compatible acids and compatible bases) (class 8)
- Corrosive solids (compatible acids and compatible bases) (class 8)
- Poisonous liquids (P listed, organic, and inorganic) (class 6.1)
- Poisonous solids (P listed, organic, and inorganic) (class 6.1)

- Flammable liquids - These liquids contain other waste codes than the bulk waste flammable liquids. (Class 3)
- Flammable solids (class 4.1)
- Class 6.1 Packing Group III
- Class 9
- Oxidizers
- Hazardous waste containing combinations of classes 3 and 8, classes 3 and 6.1, classes 6.1 and 8, classes 6.1 and 4.1, and classes 4.1 and 8.

6.11.3 Lab Pack Materials

- Vermiculite
- UN approved steel, fiber, or plastic barrels in 5, 10, 16, 20, 30 or 55 gallon sizes
- DOT labels (classes 3, 4.1, 5.1, 6.1, and 8)
- Hazardous waste labels

6.11.4 Lab Pack Procedure

Note: We normally have Stericycle lab pack all of our used chemicals. In cases where Stericycle does not complete this process, the following instructions will be used to lab pack our used chemicals.

6.11.4.1 Even though there are many containers in each lab pack, only the outside barrel (over pack) needs to be labeled. Each over pack must be labeled with the appropriate DOT diamond label, the words "hazardous waste", the proper UN shipping name for the waste (see 49 CFR 172.101), the UN or NA number, the name and address of the generator, the U.S. EPA generator number, the date the waste was first put into the barrel (accumulation start date), the EPA waste code (if applicable), and the manifest number when available. No label can cover the UN markings on the barrel, nor can any part of the DOT diamond label be covered.

6.11.4.2 Place one to two inches of vermiculite on the bottom of the over pack. Next, place containers of hazardous waste in the vermiculite, ensuring glass containers are not touching other glass containers. Once the bottom of the barrel is filled with containers, pour vermiculite on the containers until the containers are covered with one to two inches of vermiculite. Again, place containers in the barrel, as before, and cover with vermiculite.

6.11.4.3 Continue with this procedure until the barrel is full, the maximum weight / volume of waste per barrel have been achieved, or all waste of the barrel's hazard class has been packed. If there is room at the top of the barrel, fill the void with vermiculite and seal the over pack.

6.11.4.4 Keep all lab pack liquids in an upright position in the over pack. Also, any leaking containers must be sealed or transferred to another container before being placed in a lab pack.

6.11.4.5 Barrels that are leaking, severely dented, cracked, or the closures do not seal are not acceptable for shipment. The waste must be transferred from the damaged barrel as soon as possible.

6.12 Acid / Base Neutralization Procedure

All waste that has only a characteristic of corrosivity (D002) will be neutralized in an elementary neutralization unit. Only waste that will be non-regulated / non-hazardous after treatment will be neutralized. All neutralized waste will be disposed of via sanitary sewer.

- Before emptying the water samples into the neutralization tank, make sure the valve to the drain is closed. The red light on the counter should be off.
- Empty water samples into the neutralization tank. Rinse the containers placing the rinsate in the neutralization tank.
- When the neutralization tank is full, test the ph by using a ph strip.
- Add baking soda as needed to reach a ph between 6 and 9.
- Place the "Draining, No Dumping" sign kept on the side of the tank, on the sash in front of the tank.
- Enter final pH before draining and the total amount of liquid drained into the neutralization book.

6.13 Foreign and Regulated Domestic Soil Sample and Water Residue

All regulated samples must be heat treated (autoclaved) or sent offsite for incineration. The authorized compliance technician shall arrange for their disposal with a waste contractor that has a valid foreign soil permit, such as Clean Harbors-Aragonite UT Incineration facility. *This facility normally autoclaves foreign soils and places the autoclaved samples into the "soil samples and debris" box, which is collected by Stericycle for disposal.*

6.14 Archive Sample Disposal

6.13.1 Archive samples are samples that require extended storage beyond the forty five days from completion, as requested by Project Managers or clients. Project Managers will extend the disposal date in the TALS LIMs system or notify sample receiving to place "archive" stickers on the samples.

6.14.2 The Archive Shelves should be checked once a month to see which projects have exceeded their Archive date.

6.14.3 All Archive samples that have exceeded their archive date should be pulled for disposal or for Project Manager review.

6.14.4 Contact (e-mail) the Project Manager requesting permission to dispose of the archived samples. Written approval from the Project Manager is required if the samples are to be disposed.

Note: Some samples have a set archive date as defined in the client contract agreement. These samples do not need further approval from Project Managers for disposal when their set disposal time is up.

6.15 Extraction Waste Water Treatment

6.14.1 Wastewater leftover from the liquid-liquid extraction process that has had the solvent decanted from it is neutralizing and aerated for a minimum of twelve hours before being disposed of in the sanitary sewer system.

6.15.2 Enter the date and the amount of the wastewater was placed into the neutralized tank on the disposal log hanging on the side of the neutralization tank fume hood.

6.15.3 Aeration is constantly supplied to the neutralization tank via an air compressor.

6.15.4 Allow to aerate for twelve hours minimum. Dispose of through the sanitary sewer system.

6.16 Container Disposal

6.15.1 A container is not considered empty until < 1% of the content remains. Containers must be empty before they can be discarded. Whenever possible containers that held non-hazardous samples are sent out for recycle. Containers that are not suitable for recycle have their labels removed and are discarded in the dumpster.

6.15.2 Some clients, such as BP require that all labels be removed before disposal of the container through recycling. Incineration of the container is an acceptable means of removing the label on containers that cannot be recycled. VOA vials are not eligible for recycle due to the mixed composite of the container. TA Seattle bulks the VOA vial and the sample it contains into a loose pack barrel that is sent out for incineration.

6.16.2 Containers that held acutely hazardous waste must be tripled rinsed before disposal to a landfill and the rinsate captured and bulked into the appropriate hazardous waste stream.

6.17 Uniform Hazardous Waste Manifest

6.16.1 Once the waste barrels have been labeled, filled, and sealed, they are ready to be manifested.

6.17.2 The uniform hazardous waste manifest (UHWM) should be filled out prior to loading waste on a vehicle because it is required for shipment of waste to a TSD facility (this is normally done by the waste hauler). All applicable spaces need to be filled completely.

1. Generator's US EPA number
2. Page 1 of
3. Generator's name and address
4. Generator's phone number
5. Transporter 1 name
6. Transporter 1 US EPA number
7. Transporter 2 name
8. Transporter 2 US EPA number
9. TSD facility name and address
10. TSD facility US EPA ID number
11. US DOT description of waste

Proper shipping name (and technical name if proper shipping name ends in N.O.S.)

Hazard Class

UN or ID Number

The letters 'RQ' are added to the proper shipping name if the material has an equal or greater quantity than listed in 40 CFR 172.101 Table 1

12. Number and type of waste containers

13. Total quantity of waste
14. Unit of measurement of waste
15. Special handling instructions and additional information

24 hour emergency contact name and number Emergency Response Guidebook number

16. Generator's certification, signature, and date of waste pickup
17. Transporter 1 signature and date of transport
18. Transporter 2 signature and date of transport
19. Discrepancy indication space
20. TSD signature and date of waste receipt

6.17.3 Once the UHWM is completed (with the exception of the signatures), load the waste on the vehicle and sign the UHWM (it must be remembered that the UHWM is a **legal document**).

6.17.4 If the transporter does not have placards for its vehicle, it is required that the generator supplies the placards for the transporting vehicle [49 CFR 172.506(a)]. Requirements for placarding a vehicle are in 49 CFR 172 App. A, and 49 CFR 172.504.

6.17.5 The UHWM is then separated by giving the transporter the "TSD to generator copy", the "TSD to EPA copy", the "TSD" copy, and all transporter copies.

6.17.6 The generator must keep the generator copy for three years, but it is suggested that the document should be kept for the life of the facility.

6.18 TSD Facility

6.17.1 Before waste can be sent to a TSD facility, information must be acquired about the facility's environmental, legal, and financial history as well as the company's technical abilities and their indemnification policy.

7 METHODS OF DISPOSAL

7.1 Whenever possible, the waste generated by the laboratory is to be reused, recycled, or used as an energy source. Since all wastes will not fit into these categories, the wastes must then be treated.

7.2 All waste streams that are not used beneficially (see 6.3.1) must be treated according to 40 CFR 268.42 Table 2. Below are the favored treatment standards (if there are several treatments) for bulk waste streams.

- Waste flammable liquids; fuel blending (FSUBS) or incineration (INCIN)
- Waste chlorinated flammable liquids; FSUBS or INCIN
- Waste dichloromethane; Recycling, fuel blending or INCIN
- Waste pyridine and water; INCIN
- Waste aqueous mercury salts; Incineration of mercury salts and organics (IMERC).
- Waste soils and solids ; INCIN
- Wastewater, Treatment or INCIN.

7.3 Lab packs are packed according to 40 CFR 264.316 and 265.316, and all may be incinerated according to 40 CFR 268.42(c). Check with the TSD facility to ensure it will take lab packs.

8 DOCUMENT RETENTION

8.1 All documents related to the disposal of hazardous waste must be kept for a minimum of 7 years per TestAmerica's retention policy.

9 RESPONSIBILITIES

All personnel engaging in the disposal of samples must be knowledgeable of the possible health hazards and potential chemical characteristics contained in the sample waste. This information can be obtained from the chain of custody, TAL's disposal list, pertinent Material Safety Data Sheets, or speaking with the project manager or client. All personnel must have knowledge of emergency response information, self-protection measures and accident prevention methods and procedures. All personnel handling samples which contain hazardous contaminates must be at least eighteen years of age.

10. Waste Management and Pollution Prevention

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an acceptable manner. Waste description rules and land disposal restrictions are followed. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

11. References / Cross-References

11.1 Prudent Practices for Disposal of Chemicals from Laboratories. National Academy Press. Washington, D.C. 1983.

11.2 Standard Methods for the Elimination of Water and Waste Water. American Public Health Association, 1015 Fifteenth St. NW., Washington, D.C. 20005. 19th Edition.

11.3 Code of Federal Regulations, Title 40, Protection of the Environment

11.4 State of Washington Rules and Regulations; WAC 173-307.

11.5 USDA Soil Permit and Compliance Agreement

11.6 Pierce County Sewer Limits

11.7 Code of Federal Regulations, Title 49, Transportation

11.8 Code of Federal Regulations, Title 29, Labor

12. Attachment

Appendix A ICOC / TALS Sample Disposal
Appendix B TALS Invoice Date Query
Appendix C Barrel ID Number
Appendix D Barrel set up and closure instructions

13. Revision History

Revision 15, dated 28 March 2018

- Updated approvers
- Changed MSDS to SDS
- Updated naming terminology for soil samples and debris waste box
- Updated and clarified lab packing protocol
- Clarified foreign soil disposal procedure

Revision 14, dated 15 December 2015

- Removed references to disposal reports and removed the Appendix that referred to it
- Removed references to rotation log and the Appendix that referred to it.
- Revised document to change disposal date to 30 days after invoice and to revised procedures for rotation of samples out of the initial storage areas.

Revision 13, dated 10 September 2012

- Completely reformatted

APPENDIX A

TALS Sample Disposal

(For Lab Managers, Lab Supervisors, Analysts and Sample Disposal Personnel)

TABLE OF CONTENTS

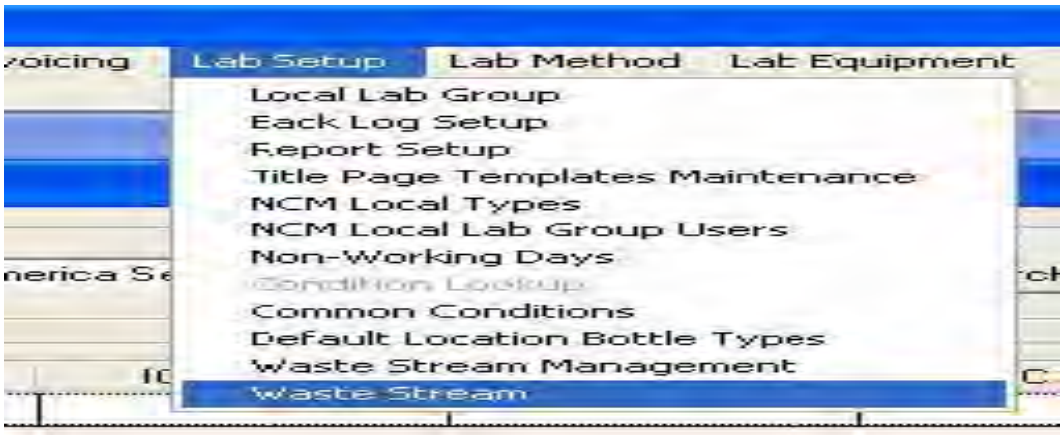
TALS Sample Disposal

TALS sample disposal is designed to track the disposal of samples in the laboratory after analysis is complete. TALS sample disposal utilizes Waste Streams and Waste Stream Containers to dispose of samples based on the samples hazard level and Waste Limit Sets associated with the Waste Stream. If a sample exceeds the criteria set in the Waste Limit Sets, the sample will qualify for disposal in that Waste Stream.

TALS sample disposal utilizes the Internal Chain of Custody module as the disposal mechanism.

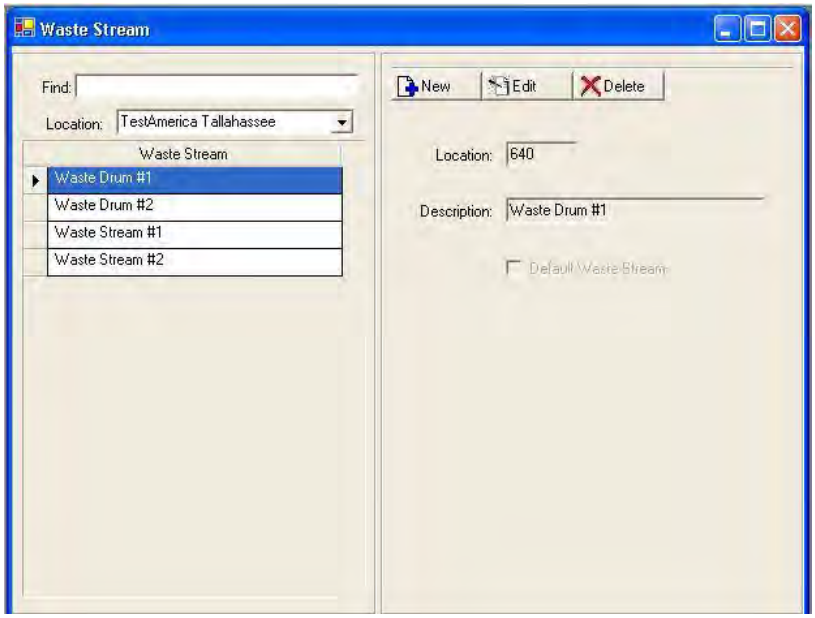
In this training brief the user will learn how to:

1. Create a Waste Stream
2. Manage a Waste Stream
3. Dispose of Samples using the Internal Chain of Custody module
4. Close out a Waste Stream Container



To Start

1. From the main menu, select Lab Setup by clicking on the application.
2.



To Create a Waste Stream

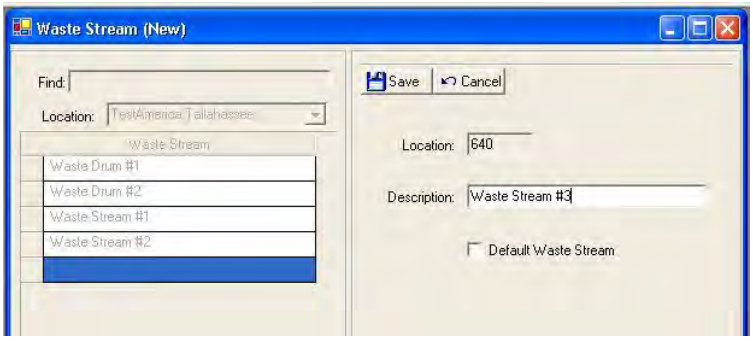
1. Click [New].

NOTE: Waste Streams can be created for other TALS locations by selecting the location from the dropdown list. Creating waste streams for other TALS locations should only be performed by experienced users and is discouraged.

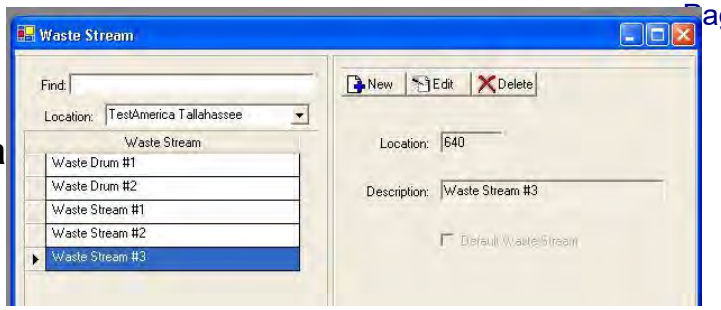
1 Enter a **Description**.

2 If the Waste Stream will be the default waste stream check the **Default Waste Stream** check box. Waste Limit Sets cannot be added to a waste stream that is marked as a default waste stream. Waste Limit Sets will be covered in the next section.

3 Click [Save].


Waste Stream (New)
Find:
Location:
TestAmerica Tallahassee
Waste Stream
Waste Drum #1
Waste Drum #2
Waste Stream #1
Waste Stream #2
Save
Cancel
Location:
640
Description:
Waste Stream #3
Default Waste Stream

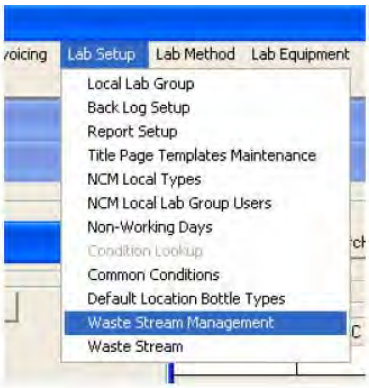
To Manage a Waste Stream



1 From the main menu, select Lab Setup by clicking on the application.

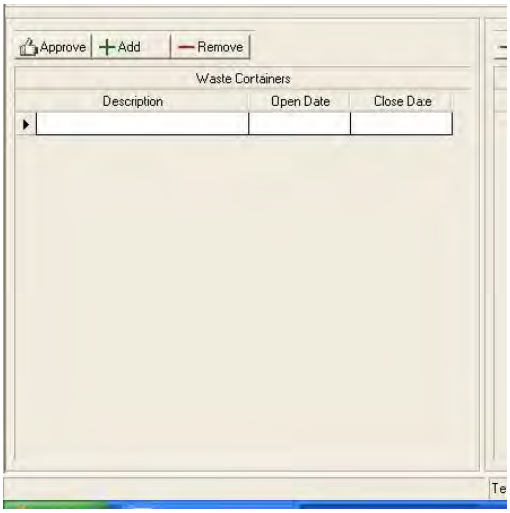
2 From the Lab Setup menu, select Waste Stream Management by clicking on the application.

Waste Containers



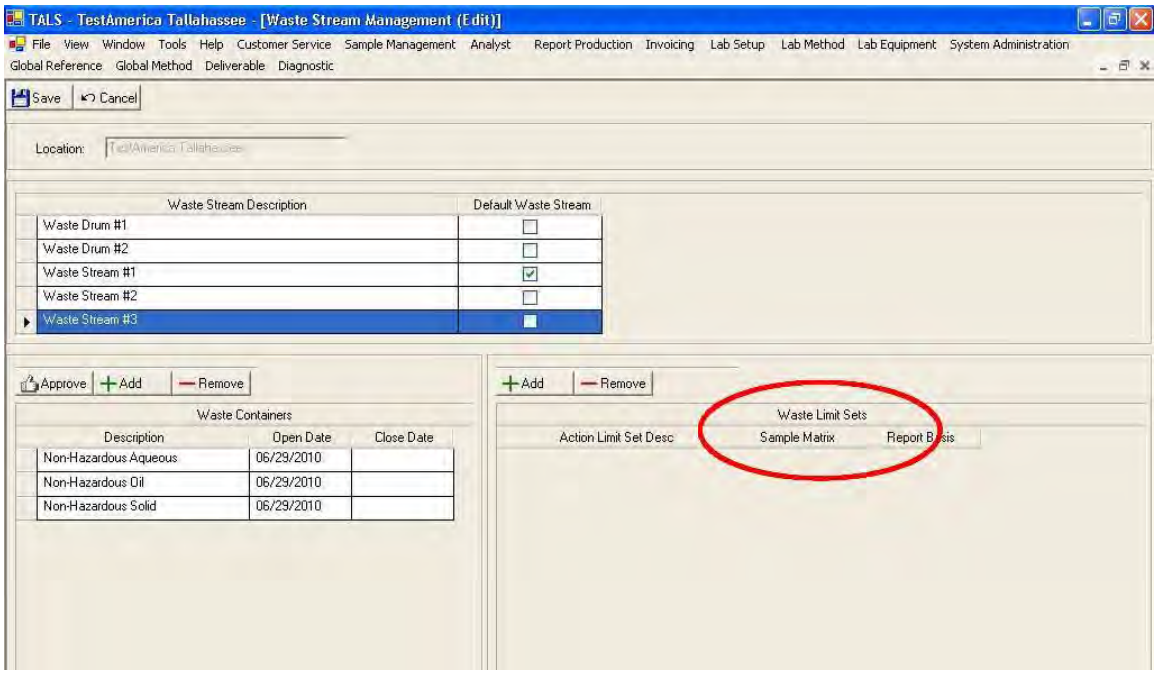
Waste Containers are assigned to a Waste Stream to distinguish different containers for different forms of waste. It is at the labs discretion how Waste Containers are handled.

1. Click [Edit].
2. In the 'Waste Containers' grid, click [Add].
3. Enter a description for the waste container in free text form.
4. Select an **Open Date** from the dropdown calendar.
5. Add additional Waste Containers as required.

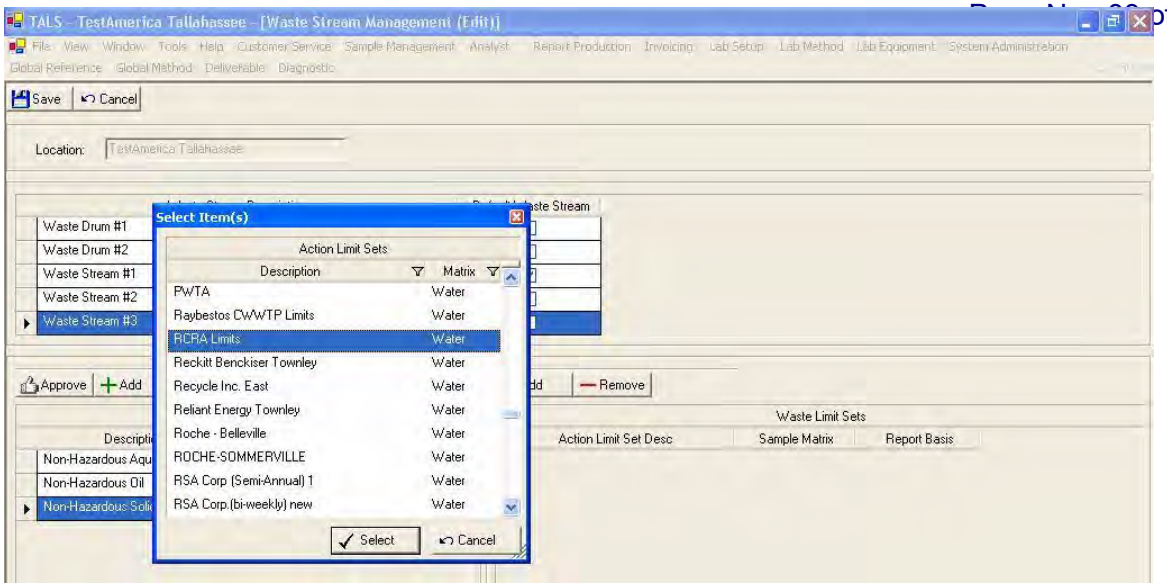


Waste Limit Sets

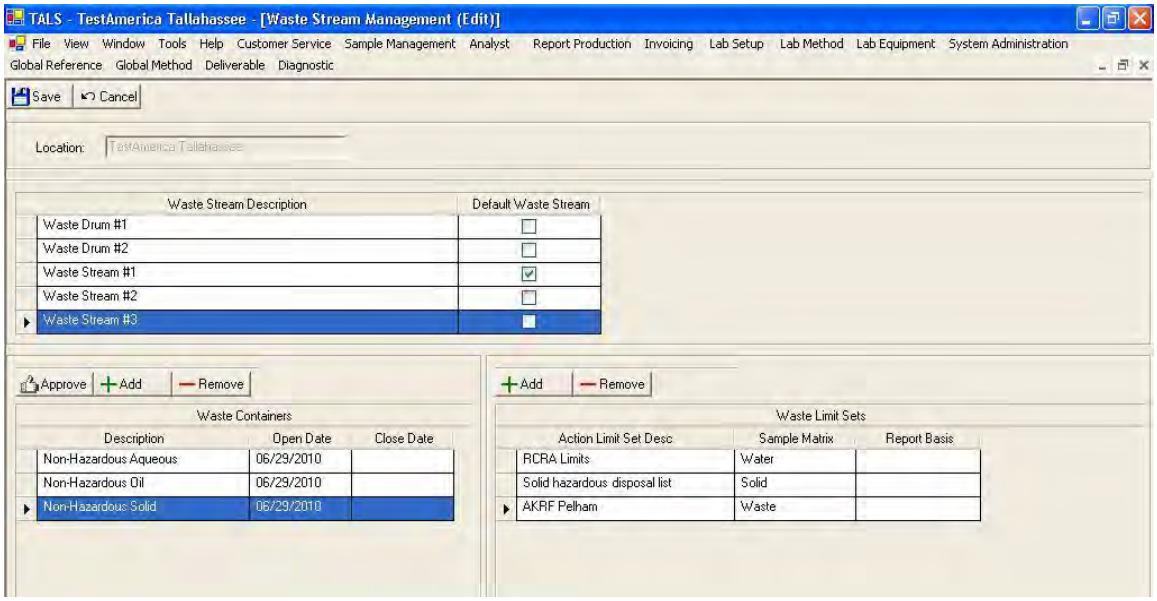
Each Waste Stream can have Waste Limit Sets applied to it based on the contents of the Waste Stream. If a Waste Limit Set is not assigned to a Waste Stream it will be a Waste Stream that qualifies all samples for disposal and anything can go into it. Waste Limits Sets are Action Limit Sets established at the laboratory. See Action Limit Sets documentation posted on the intranet for additional information on Action Limit Sets.

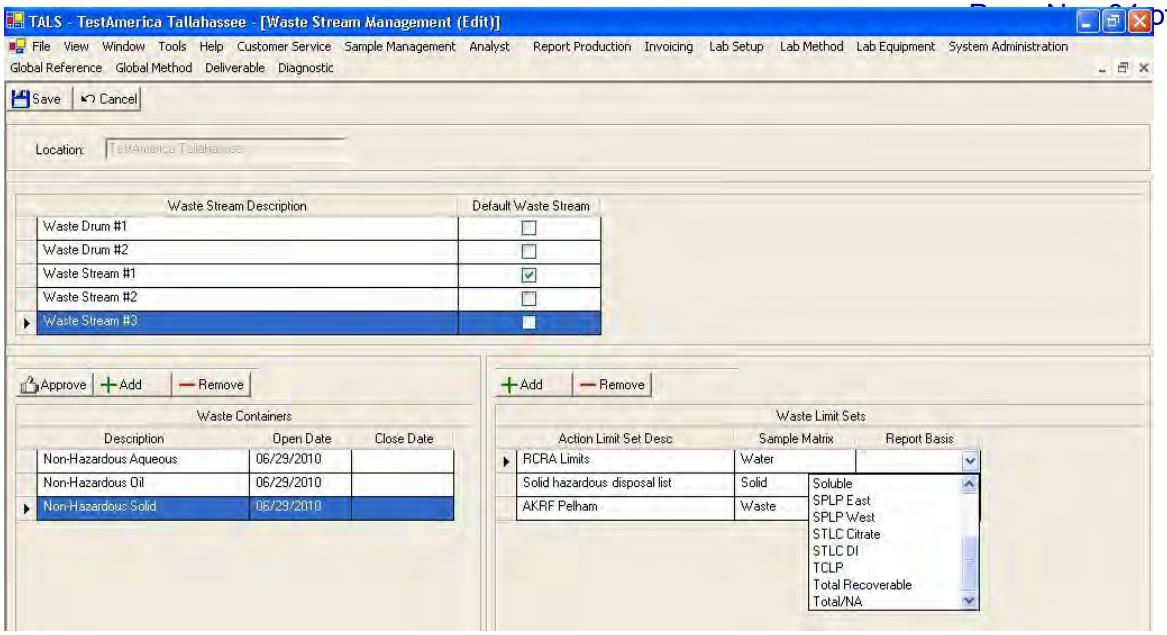


1 Highlight the Waste Stream in the top grid.
2 From the 'Waste Limit Sets' grid on the lower right, click [Add].
3 A pop up will appear listing all available Action Limit Sets in TALS. Highlight the Action Limit Set that should be applied to the Waste Stream and click [Select]. If a sample exceeds any of the limits in a Waste Limit Set, the sample will "qualify" for disposal in that Waste Stream.
4 Waste Streams can have multiple Waste Limit Sets if necessary to meet the laboratories needs.
5 A **Report Basis** must be defined for each action limit set.



NOTE: Waste Limit (Action Limit) Sets screen samples based on analyte and reporting basis only. They are not method dependent.

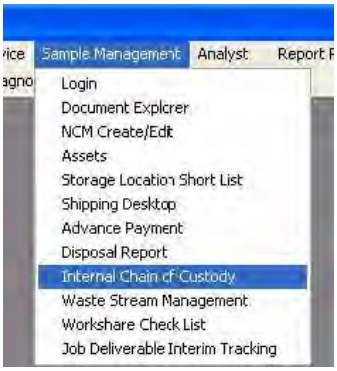




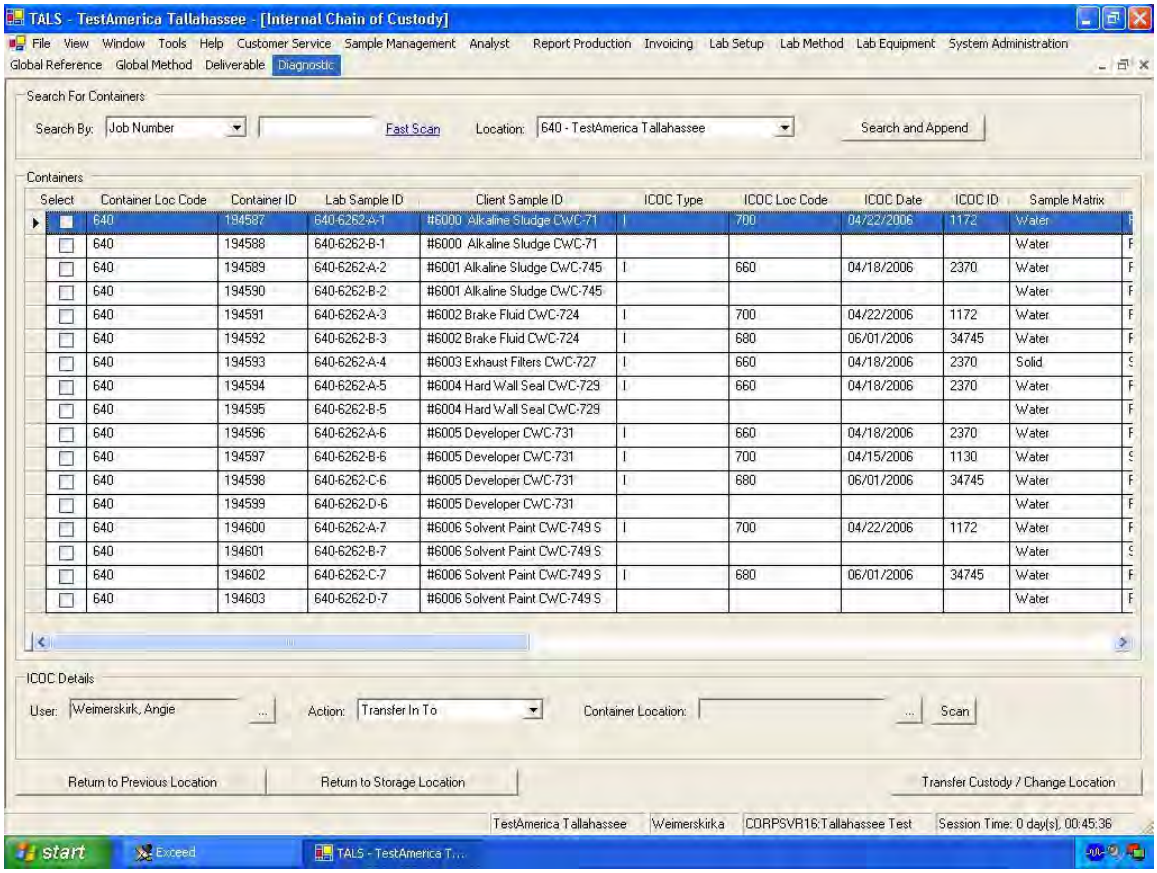
To Dispose of Samples Using the Internal Chain of Custody (ICOC) Module

Users should use the Disposal Reports to determine which samples can be disposed. See Disposal Reports documentation on the intranet for detailed information on the Disposal Reports.

1 From the main menu, select Sample Management by clicking on the application.
2 From the Sample Management menu, select Internal Chain of Custody by clicking on the application.
3 Search for the samples by Job Number, Lab Sample ID, Container ID, Login Number, ICOC ID, Batch Number or use the 'Fast Scan' option.
4 If applicable, change the location using the dropdown menu if disposing of work share samples.
5 Click [Search and Append].
6 Right-click anywhere in the grid and select 'Display Disposable Containers'. Click 'Display Report after Transfer' if desired.

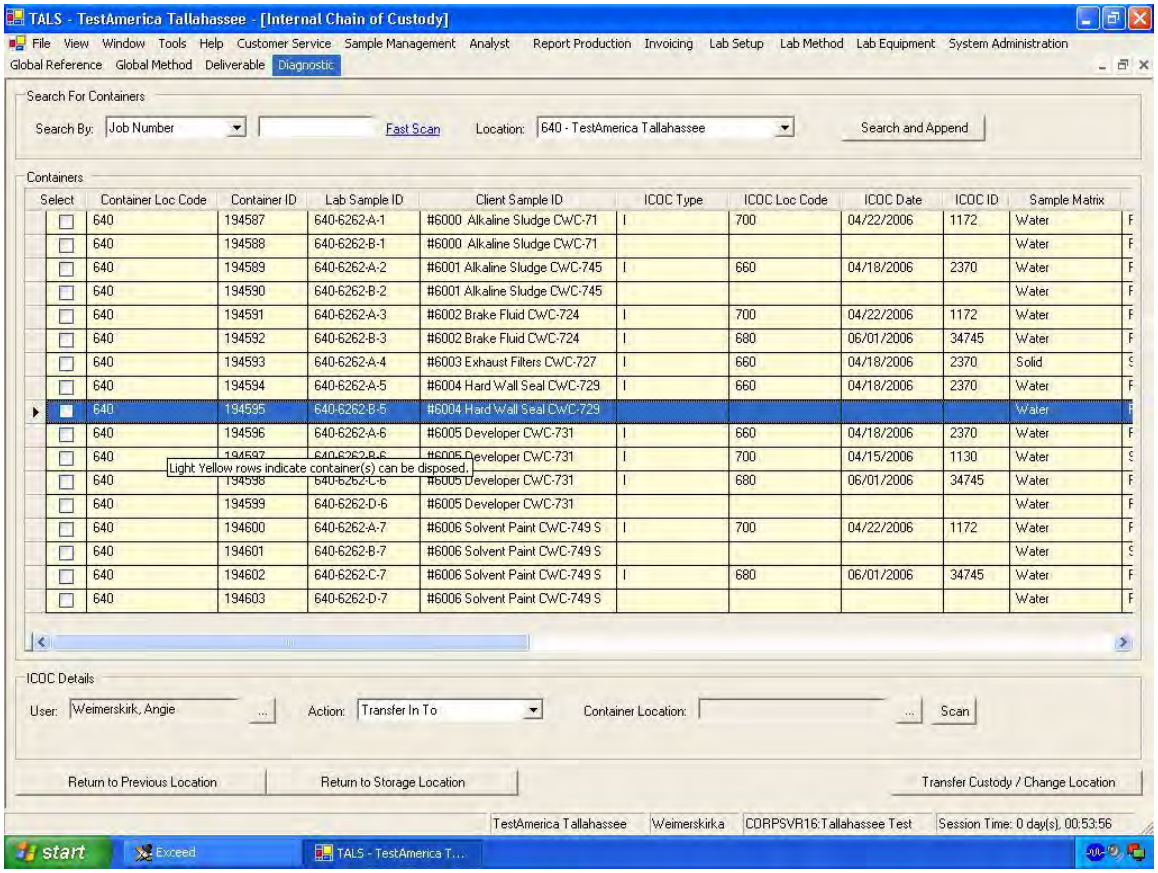


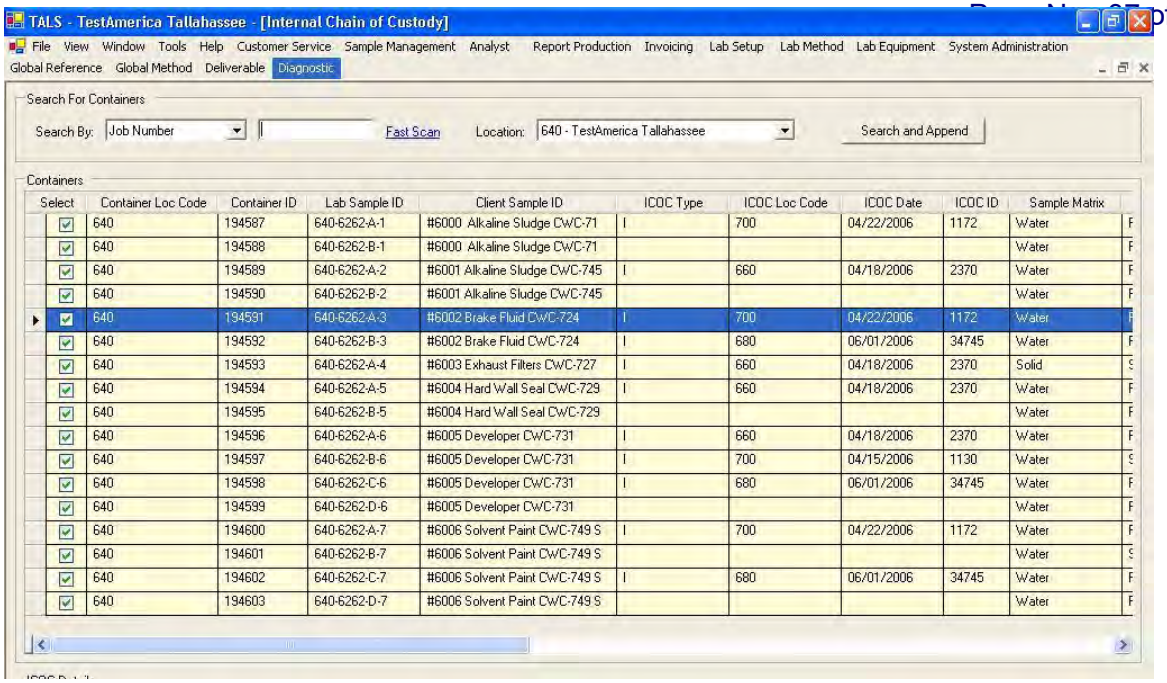

TALS - TestAmerica Tallahassee - [Internal Chain of Custody]
File View Window Tools Help Customer Service Sample Management Analyst Report Production Invoicing Lab Setup Lab Method Lab Equipment System Administration
Global Reference Global Method Deliverable Diagnostic
Search For Containers
Search By: Lab Sample ID
Fast Scan
Location: 640 - TestAmerica Tallahassee
Search and Append
Job Number
Lab Sample ID
Container ID
Login Number
ICOC ID
Batch Number
Containers
Select
Lab Sample ID
Client Sample ID
ICOC Type
ICOC Loc Code
ICOC Date
ICOC ID
Sample Matrix

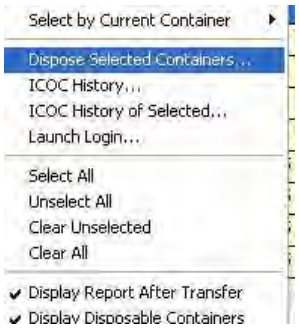

TALS - TestAmerica Tallahassee - [Internal Chain of Custody]
File View Window Tools Help Customer Service Sample Management Analyst Report Production Invoicing Lab Setup Lab Method Lab Equipment System Administration
Global Reference Global Method Deliverable Diagnostic
Search For Containers
Search By: Job Number
Fast Scan
Location: 640 - TestAmerica Tallahassee
Search and Append
Containers
Select Container Loc Code Container ID Lab Sample ID Client Sample ID ICOC Type ICOC Loc Code ICOC Date ICOC ID Sample Matrix
640 194587 640-6262-A-1 #6000 Alkaline Sludge CWC-71 I 700 04/22/2006 1172 Water
640 194588 640-6262-B-1 #6000 Alkaline Sludge CWC-71 Water
640 194589 640-6262-A-2 #6001 Alkaline Sludge CWC-745 I 660 04/18/2006 2370 Water
640 194590 640-6262-B-2 #6001 Alkaline Sludge CWC-745 Water
640 194591 640-6262-A-3 #6002 Brake Fluid CWC-724 I 700 04/22/2006 1172 Water
640 194592 640-6262-B-3 #6002 Brake Fluid CWC-724 I 680 06/01/2006 34745 Water
640 194593 640-6262-A-4 #6003 Exhaust Filters CWC-727 I 660 04/18/2006 2370 Solid
640 194594 640-6262-A-5 #6004 Hard Wall Seal CWC-729 I 660 04/18/2006 2370 Water
640 194595 640-6262-B-5 #6004 Hard Wall Seal CWC-729 Water
640 194596 640-6262-A-6 #6005 Developer CWC-731 I 660 04/18/2006 2370 Water
640 194597 640-6262-B-6 #6005 Developer CWC-731 I 700 04/15/2006 1130 Water
640 194598 640-6262-C-6 #6005 Developer CWC-731 I 680 06/01/2006 34745 Water
640 194599 640-6262-D-6 #6005 Developer CWC-731 Water
640 194600 640-6262-A-7 #6006 Solvent Paint CWC-749 S I 700 04/22/2006 1172 Water
640 194601 640-6262-B-7 #6006 Solvent Paint CWC-749 S Water
640 194602 640-6262-C-7 #6006 Solvent Paint CWC-749 S I 680 06/01/2006 34745 Water
640 194603 640-6262-D-7 #6006 Solvent Paint CWC-749 S Water
ICOC Details
User: Weimerskirk, Angie
Action: Transfer In To
Container Location:
Scan
Return to Previous Location
Return to Storage Location
Transfer Custody / Change Location
TestAmerica Tallahassee
Weimerskirka
CORPSVR16:Tallahassee Test
Session Time: 0 day(s), 00:45:36
start
Exceed
TALS - TestAmerica T...

5. Disposable containers will be shaded light yellow.

6. The user can check the boxes in the **Select** column or right-click and hit 'Select All' if all containers can be disposed.

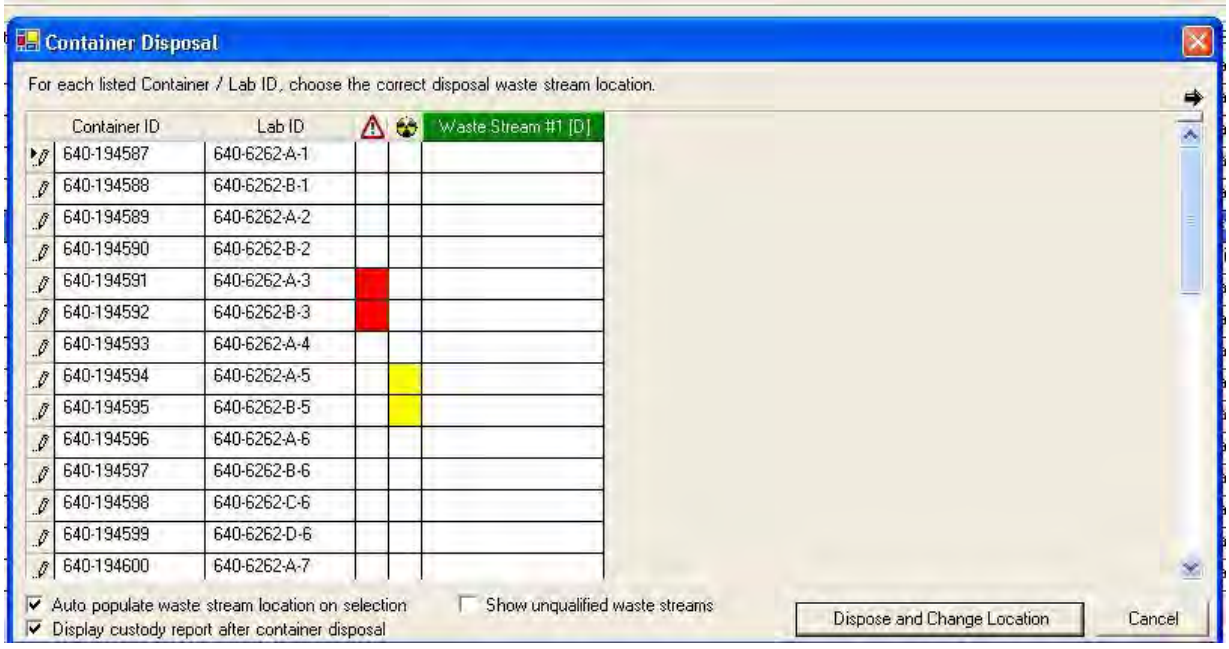






7. Right-click and select 'Dispose of Selected Containers'.

8. TALS will display the samples and any Waste Streams that the sample qualifies for based on the Waste Limit Sets applied to the Waste Stream. If a sample(s) do not qualify for any Waste Streams that have Waste Limit Sets applied the default Waste Stream will be used.



azardous or Radioactive

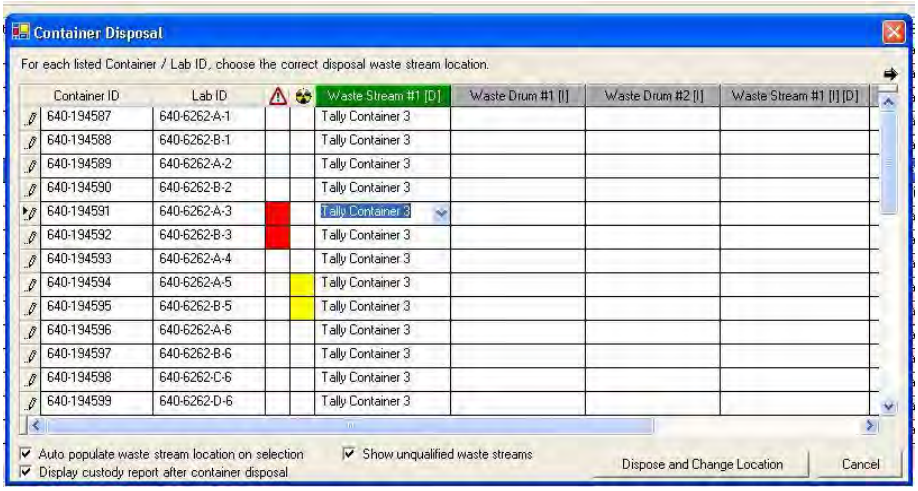
in the login it will be indicated in these columns. 10. There are check boxes on the display to:

a. Auto populate waste stream location on selection. If this is checked the list will auto populate with the first Waste Stream selected. If multiple Waste Streams need to be selected, uncheck this selection.

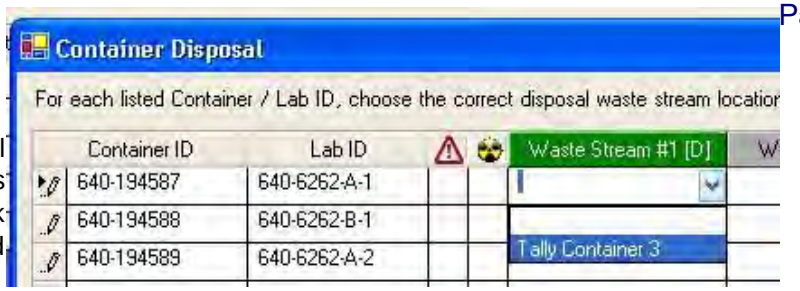
b. Display custody report after container disposal.

c. Show unqualified waste streams. Unqualified waste streams show up with a gray header.

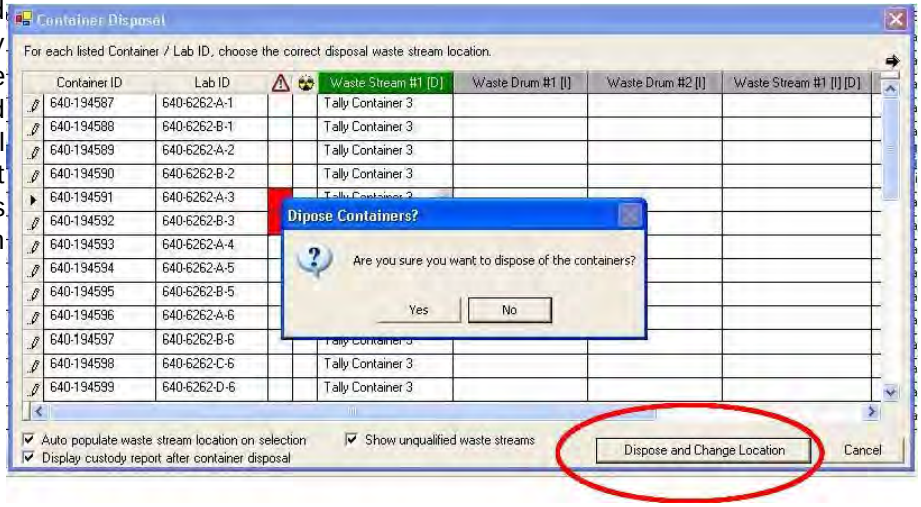
11. Select the Waste Stream from the dropdown list.

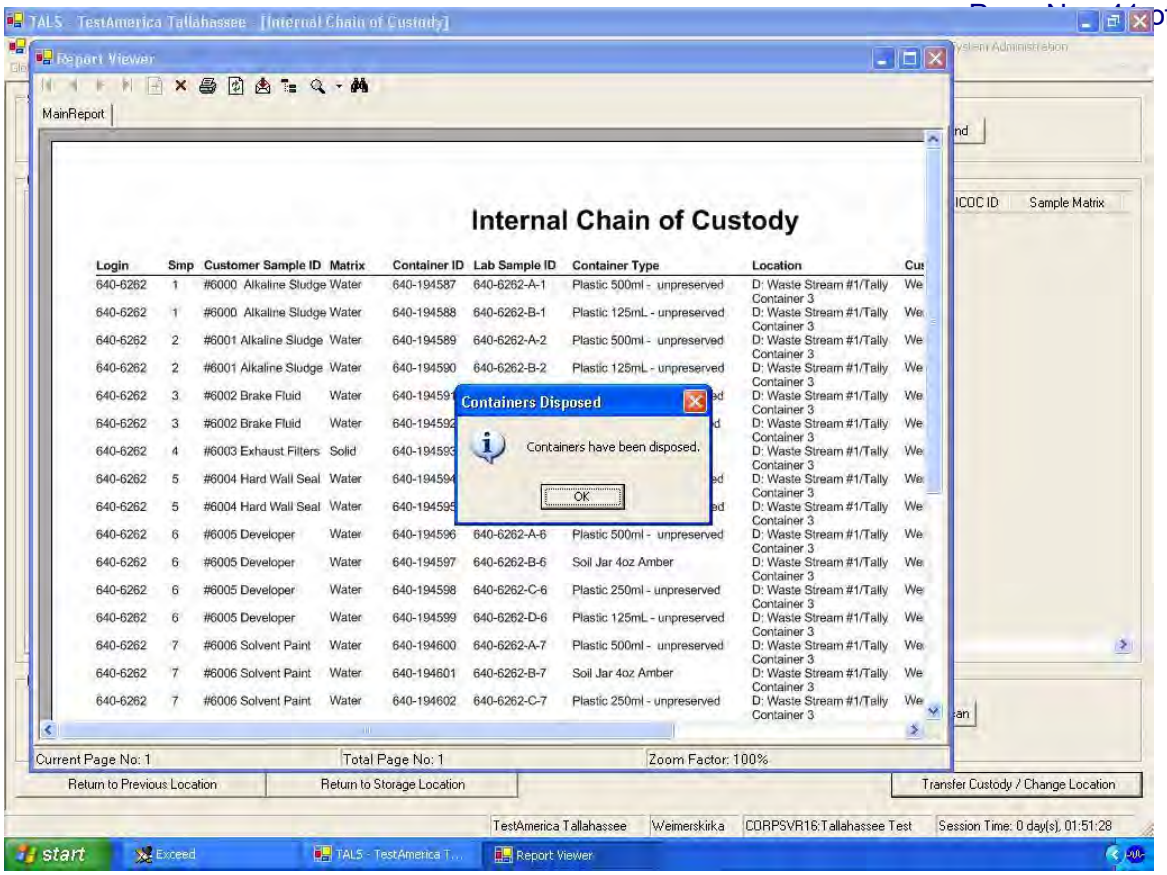


12. When all Waste Streams are selected, click [Dispose and Change Location]. Click [Yes] on the pop up.



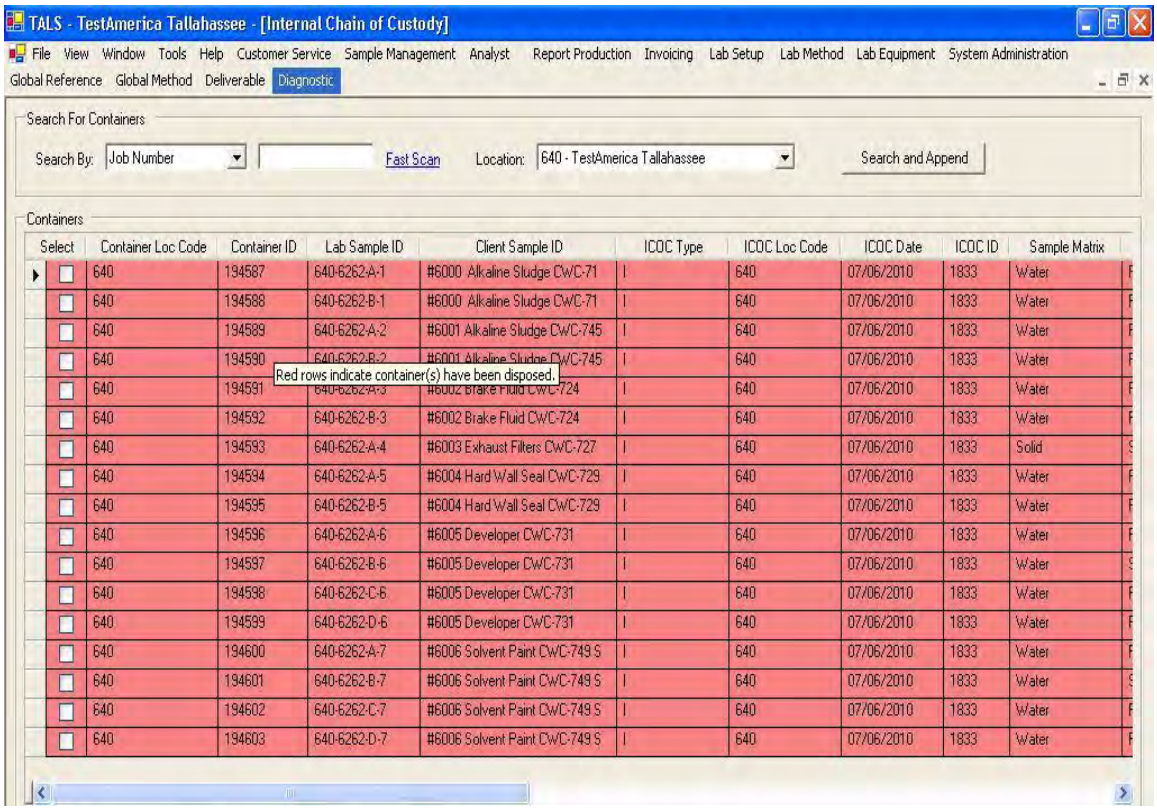
13. If checked on, a custody report will be displayed and TALS will indicate that the containers have been disposed.



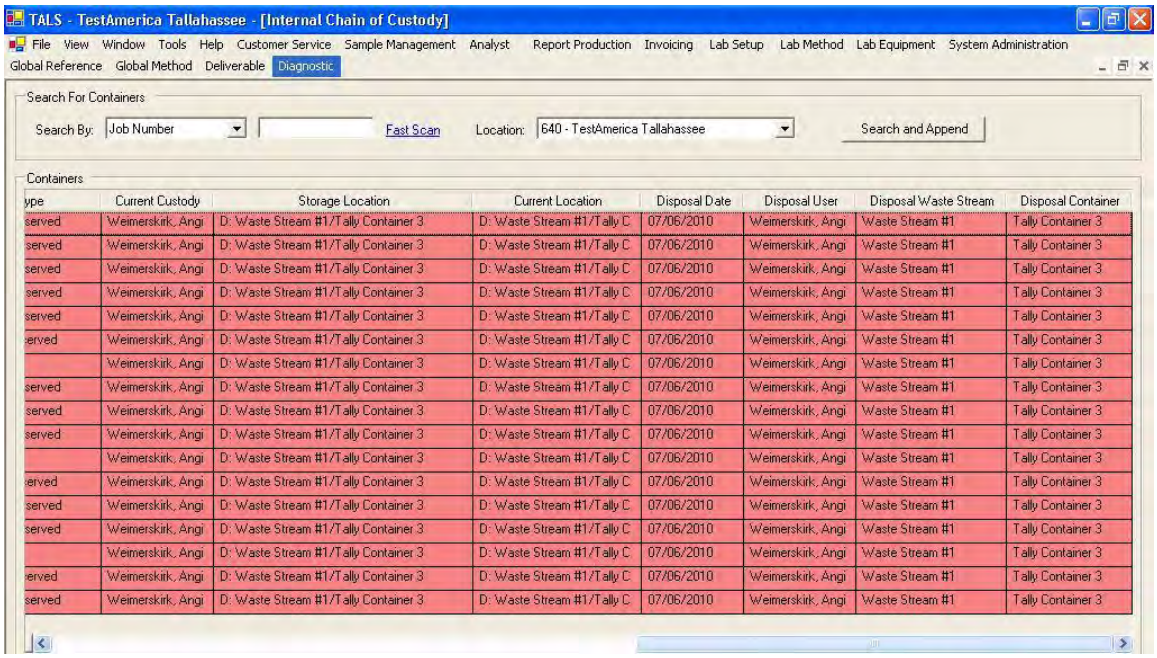


Login	Smp	Customer Sample ID	Matrix	Container ID	Lab Sample ID	Container Type	Location
640-6262	1	#6000 Alkaline Sludge	Water	640-194587	640-6262-A-1	Plastic 500ml - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	1	#6000 Alkaline Sludge	Water	640-194588	640-6262-B-1	Plastic 125mL - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	2	#6001 Alkaline Sludge	Water	640-194589	640-6262-A-2	Plastic 500ml - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	2	#6001 Alkaline Sludge	Water	640-194590	640-6262-B-2	Plastic 125mL - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	3	#6002 Brake Fluid	Water	640-194591			D: Waste Stream #1/Tally Container 3
640-6262	3	#6002 Brake Fluid	Water	640-194592			D: Waste Stream #1/Tally Container 3
640-6262	4	#6003 Exhaust Filters	Solid	640-194593			D: Waste Stream #1/Tally Container 3
640-6262	5	#6004 Hard Wall Seal	Water	640-194594			D: Waste Stream #1/Tally Container 3
640-6262	5	#6004 Hard Wall Seal	Water	640-194595			D: Waste Stream #1/Tally Container 3
640-6262	6	#6005 Developer	Water	640-194596	640-6262-A-6	Plastic 500ml - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	6	#6005 Developer	Water	640-194597	640-6262-B-6	Soil Jar 4oz Amber	D: Waste Stream #1/Tally Container 3
640-6262	6	#6005 Developer	Water	640-194598	640-6262-C-6	Plastic 250ml - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	6	#6005 Developer	Water	640-194599	640-6262-D-6	Plastic 125mL - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	7	#6006 Solvent Paint	Water	640-194600	640-6262-A-7	Plastic 500ml - unpreserved	D: Waste Stream #1/Tally Container 3
640-6262	7	#6006 Solvent Paint	Water	640-194601	640-6262-B-7	Soil Jar 4oz Amber	D: Waste Stream #1/Tally Container 3
640-6262	7	#6006 Solvent Paint	Water	640-194602	640-6262-C-7	Plastic 250ml - unpreserved	D: Waste Stream #1/Tally Container 3

14. Click [OK]. Print the disposal report if desired.
15. Searching for the samples again will show the samples have been disposed.



Select	Container Loc Code	Container ID	Lab Sample ID	Client Sample ID	ICOC Type	ICOC Loc Code	ICOC Date	ICOC ID	Sample Matrix
	640	194587	640-6262-A-1	#6000 Alkaline Sludge CWC-71	I	640	07/06/2010	1833	Water
	640	194588	640-6262-B-1	#6000 Alkaline Sludge CWC-71	I	640	07/06/2010	1833	Water
	640	194589	640-6262-A-2	#6001 Alkaline Sludge CWC-745	I	640	07/06/2010	1833	Water
	640	194590	640-6262-B-2	#6001 Alkaline Sludge CWC-745	I	640	07/06/2010	1833	Water
	640	194591	640-6262-A-3	#6002 Brake Fluid CWC-724	I	640	07/06/2010	1833	Water
	640	194592	640-6262-B-3	#6002 Brake Fluid CWC-724	I	640	07/06/2010	1833	Water
	640	194593	640-6262-A-4	#6003 Exhaust Filters CWC-727	I	640	07/06/2010	1833	Solid
	640	194594	640-6262-A-5	#6004 Hard Wall Seal CWC-729	I	640	07/06/2010	1833	Water
	640	194595	640-6262-B-5	#6004 Hard Wall Seal CWC-729	I	640	07/06/2010	1833	Water
	640	194596	640-6262-A-6	#6005 Developer CWC-731	I	640	07/06/2010	1833	Water
	640	194597	640-6262-B-6	#6005 Developer CWC-731	I	640	07/06/2010	1833	Water
	640	194598	640-6262-C-6	#6005 Developer CWC-731	I	640	07/06/2010	1833	Water
	640	194599	640-6262-D-6	#6005 Developer CWC-731	I	640	07/06/2010	1833	Water
	640	194600	640-6262-A-7	#6006 Solvent Paint CWC-749 S	I	640	07/06/2010	1833	Water
	640	194601	640-6262-B-7	#6006 Solvent Paint CWC-749 S	I	640	07/06/2010	1833	Water
	640	194602	640-6262-C-7	#6006 Solvent Paint CWC-749 S	I	640	07/06/2010	1833	Water
	640	194603	640-6262-D-7	#6006 Solvent Paint CWC-749 S	I	640	07/06/2010	1833	Water



Search By: Job Number

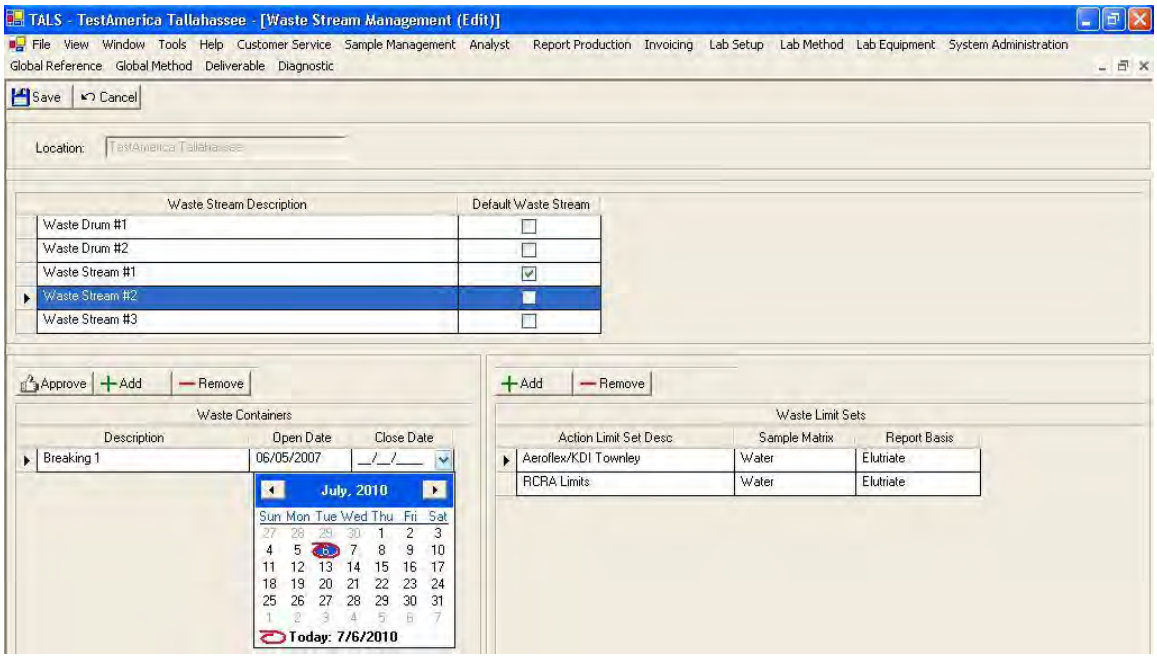
Location: 640 - TestAmerica Tallahassee

ype	Current Custody	Storage Location	Current Location	Disposal Date	Disposal User	Disposal Waste Stream	Disposal Container
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
erved	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
erved	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
erved	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3
served	Weimerskirk, Angi	D: Waste Stream #1/Tally Container 3	D: Waste Stream #1/Tally C	07/06/2010	Weimerskirk, Angi	Waste Stream #1	Tally Container 3

16. Scrolling to the right displays the disposal information.

To Close Out a Waste Stream Container

1 Go to Waste Stream Management.
2 While in Edit mode select the Waste Stream Container to close.
3 Under **Close Date** select a date from the dropdown calendar. Closed Waste Stream Containers will not be available for selection in the ICOC module.
4 Click [Add] to open a new Waste Stream Container as discussed earlier in this document.
5 If a Waste Stream Container is inactivated it will be shaded dark red. To reactivate hit [Approve].


TALS - TestAmerica Tallahassee - [Waste Stream Management (Edit)]
File View Window Tools Help Customer Service Sample Management Analyst Report Production Invoicing Lab Setup Lab Method Lab Equipment System Administration
Global Reference Global Method Deliverable Diagnostic
Save
Cancel
Location:
Waste Stream Description
Default Waste Stream
Waste Drum #1
Waste Drum #2
Waste Stream #1
Waste Stream #2
Waste Stream #3
Approve
Add
Remove
Waste Containers
Description
Open Date
Close Date
Breaking 1
06/05/2007
July, 2010
Sun Mon Tue Wed Thu Fri Sat
Today: 7/6/2010
Add
Remove
Waste Limit Sets
Action Limit Set Desc
Sample Matrix
Report Basis
Aeroflex/KDI Townley
Water
Elutriate
RCRA Limits
Water
Elutriate

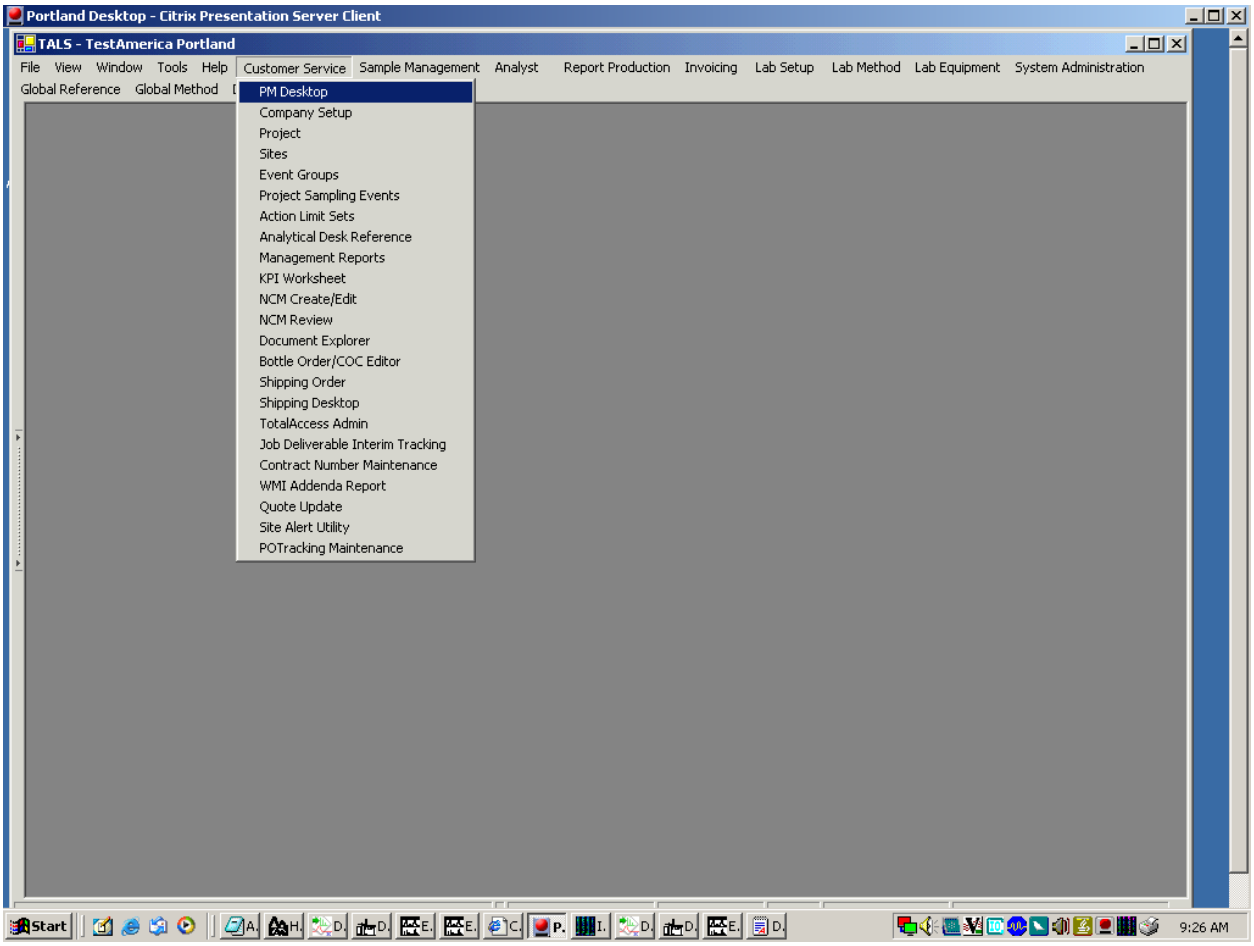
APPENDIX B

TALS PM DESKTOP

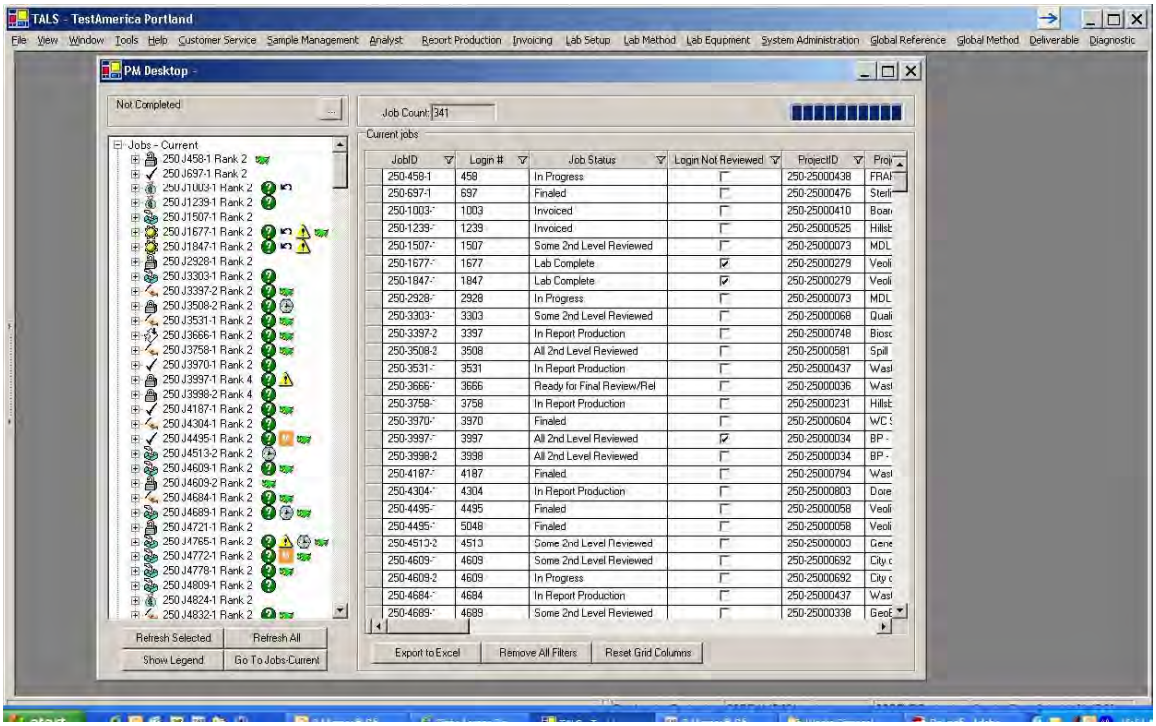
Scan out all the samples using the internal chain of custody (ICOC) program in the LIMS system. See Appendix A for directions on how to use the ICOC.

Remove any samples from the group that cannot be disposed of. See instructions in this Appendix for what to do with these samples.

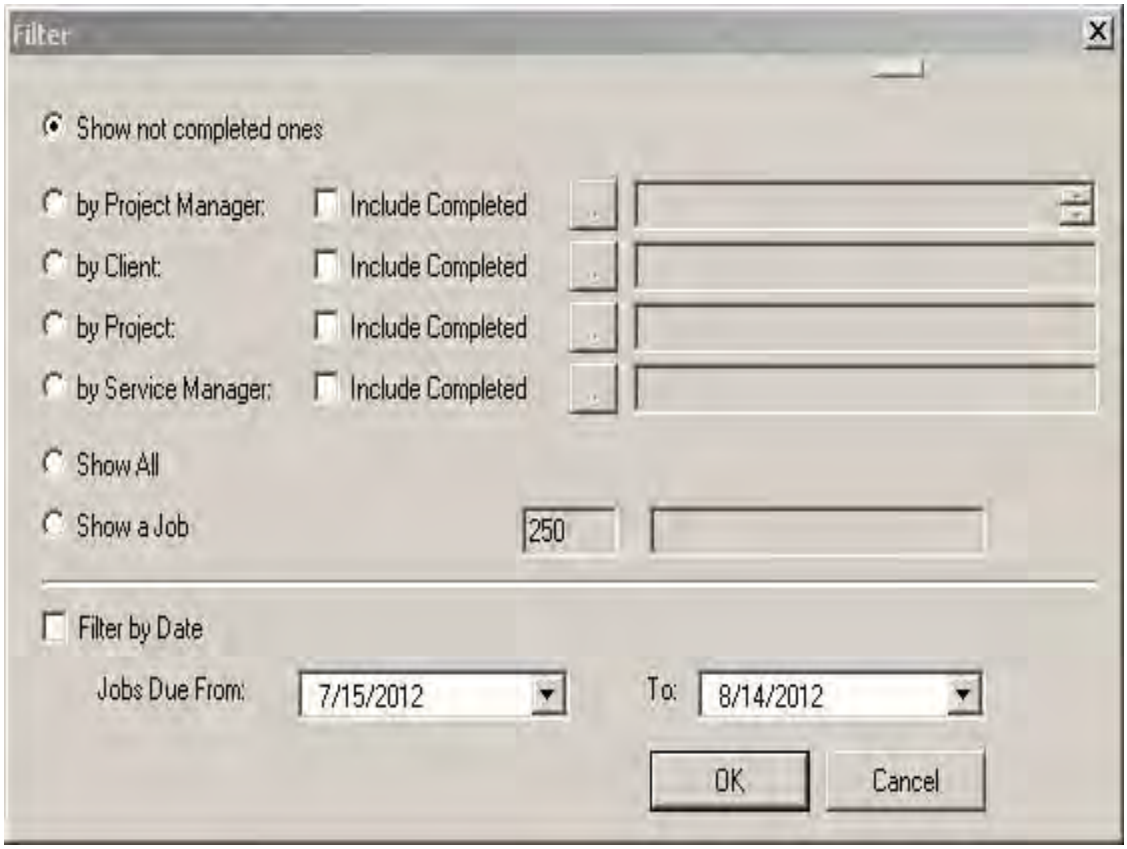
This procedure is to be used to see why a sample that appears to be old enough to be disposed of displays as not being able to be disposed of in the ICOC/Disposal report.



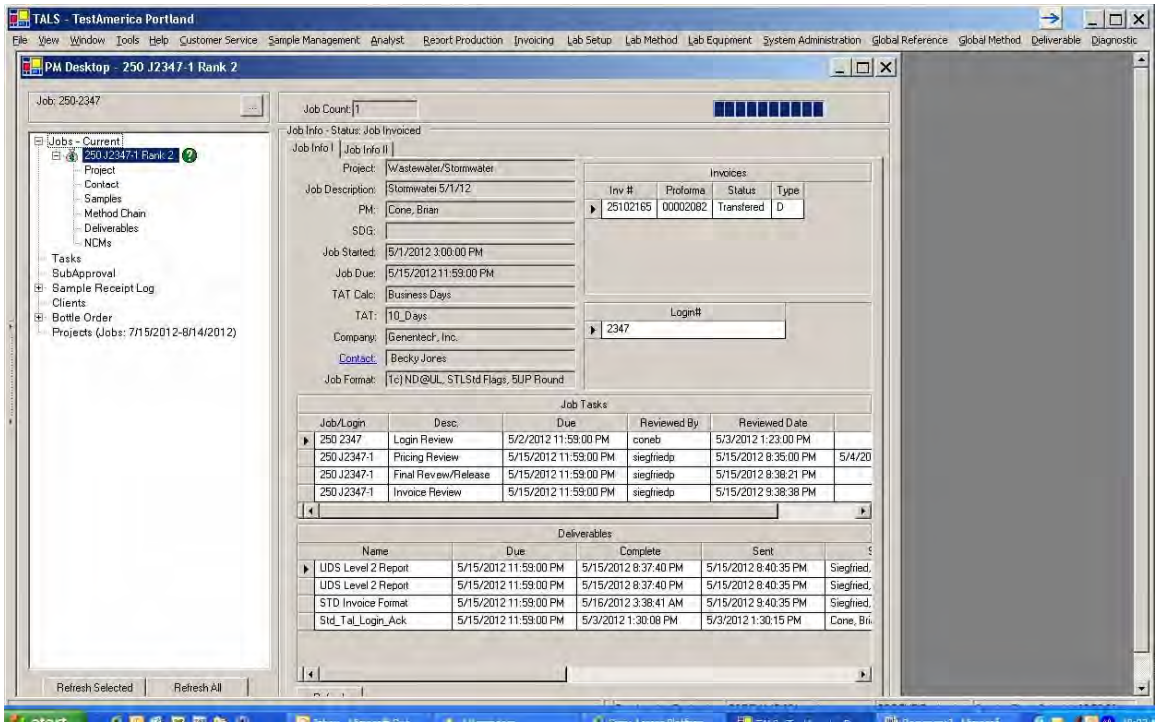
To find addition information regarding samples, click on the PM DESKTOP tab under customer service.



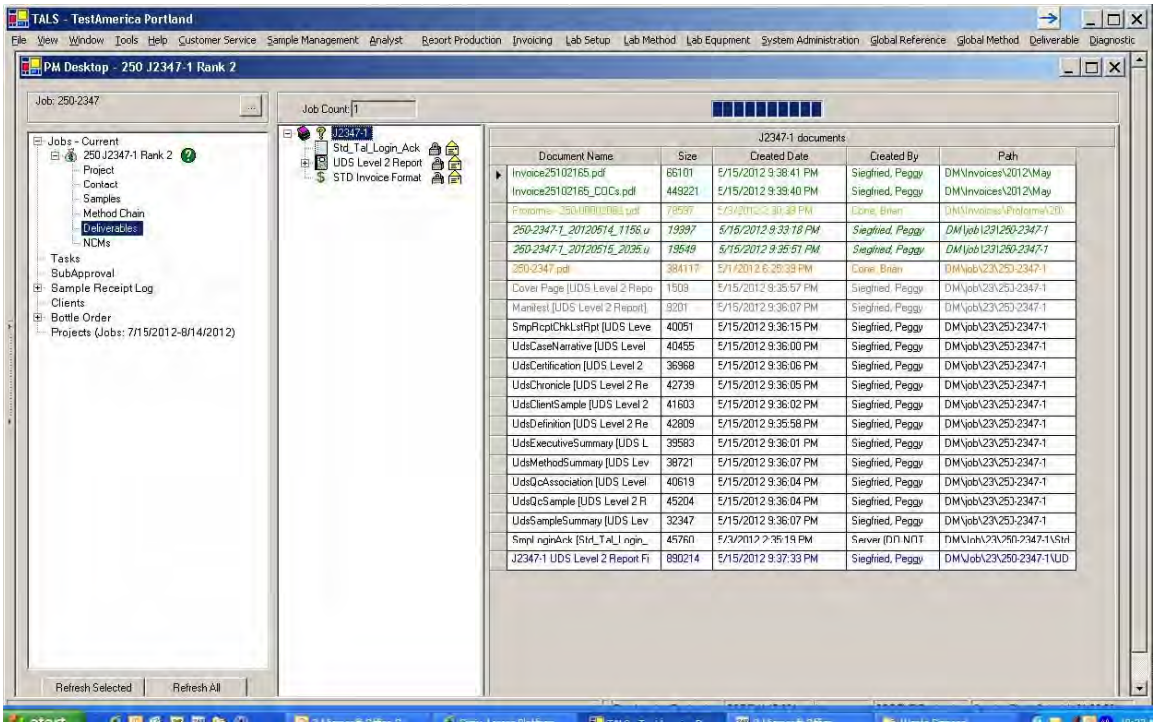
You will now have the PM Desktop screen/window open for your search. Just below the PM Desktop bar and to the left half of the screen you will see a button with three dots in it. Click this button.



The next screen to open is the “filter” screen. Here, there are many options available to you. For this search you are interested in the seventh option down on the left. Click on the “Show a Job” button then tab over twice to the long box to the right of the three digit code for your facility (which, of course, can be changed to another facility by clicking in it and just changing the number for the facility you are searching for). Now enter the numerical code for the job number you are inquiring about and hit enter.



The next screen to pop up will say PM Desktop ###-####, (this number will be the number of the Job you are inquiring about. To the left and the second item down, you will see a + sign and a money bag to its right – click on the money bag. This will bring up a screen on the right hand side. Information regarding the name of the project, the client and the project manager for the job will appear on this screen.



Next, go back to the left hand side and under the money bag is the choice “deliverables” click on this selection. The screen that comes up will display the time line of documents associated with the job number. The line highlighted in green is the date of the final invoice for the job.
Nearly all wastes can be disposed of 30 days after the invoice date highlighted in green. Longer hold dates may be requested by the client and are built into the project by the project manager. An example would be Department of Defense samples which are held for a standard 90days for all projects. The timeline built into the project cannot differentiate between sample matrixes. Therefore for clients such as BNSF, UPRR, and ERM who request that only soil samples be held for six months, will display all samples as not being able to be disposed in the ICOC/disposal request. (refer to appendix A). By reviewing this screen and the previous screen the information needed to identify the client and the disposal date will provide the information necessary to determine if a sample falls into this category and may be disposed of earlier than the date built into the project.
If samples from other clients have reached 30 days past the date highlighted in green and still return the result of not valid for disposal, you must contact the project manger before disposing of the samples.

APPENDIX C

STL Seattle

DRUM INVENTORY

DRUM #	Contents	Begin Use Date	Date Full	Ship Date	Manifest #
523	MeCl	6/1/12	6/1/12		
524	Gloves/Foam	6/4/12	6/4/12		
525	DIRT + DEBIS	6/5/12			
526	Gloves/Foam	6/5/12	6/7/12		
526	Acid waste media	6/5/12	6/5/12		

Comments:

Page 33

APPENDIX D

BARREL CLOSURE INSTRUCTIONS

M&M Industries Life Latch Plastic Pails: Container Markings Page 2 of 2

allocation of the mark:	USA	identifies country of origin
Registered Symbol of Party taking responsibility for the Mark	M2074	identifies M & M Industries.

Should you have any questions, please contact M & M Industries' Customer Service at 1-800-331-5305.

Thank you for your trust and business.

M & M Industries, Inc.

*0.6 Gallon pail:	4	2.5 Gallon pail:	15	3.5 Gallon pail:	19
1.25 Gallon pail:	6	5.0 Gallon pail:	30	6.5 Gallon pail:	30
2.0 Gallon pail:	12	5.5 Gallon pail:	30	11.3 Gallon pail:	40

Correct Closing Torque For Plugs and Locking Rings

Under 49 CFR 178.2, we are obliged to notify you as purchaser of these drums "of all requirements.. not met at the time of transfer, and the type and dimensions of any closures, including gaskets, needed to satisfy performance test requirements." Please note that as delivered, the closures are not tightened for shipment.

When design tested these closures had the following torque specifications:

PLUG TYPE	PLUG GASKETS	CORRECT TORQUE 3/4" PLUGS	2" PLUGS
Tri-Sure Steel	Rubber Black Buna, White Buna, EPDM, Butyl, White Neoprene Viton and Silicone	12 Ft. Lbs	20 Ft. Lbs
Tri-Sure Steel	Polyethylene, Irradiated Polyethylene and Telfon	20 Ft. Lbs	30 Ft. Lbs
Tri-Sure-Polyethylene Polypropylene, Nylon	Rubber Black Buna, White Buna, EPDM, Butyl, White Neoprene Viton and Silicone	12 Ft. Lbs	20 Ft. Lbs
	Polyethylene, Irradiated Polyethylene and Telfon	8 Ft. Lbs	30 Ft. Lbs
Tri Sure-Polyethylene	None (Self Gasketing)	5 Ft. Lbs	12 Ft. Lbs
		1/2" PLUG	2 1/2" PLUG
Tri Sure-Polypropylene (Poly Clad)	None (Self Gasketing)	5 Ft. Lbs	12 Ft. Lbs
Rieke Steel	Rubber Black Buna, White Buna, EPDM, Butyl, Black Visecar, White Visecar, White Dapon, White EPT, Black EPT	15 Ft. Lbs	30 Ft. Lbs
	Poly Seal (plastic) Irradiated and Non-Irradiated	20 Ft. Lbs	40 Ft. Lbs
Rieke-Polyethylene Polypropylene, Nylon	Rubber White Buna, Black EPDM, White EPDM, Dapon Irradiated and Non-Irradiated	9 Ft. Lbs	20 Ft. Lbs
Riske-Polyethylene Polypropylene, Nylon	None (Self Gasketing)	9 Ft. Lbs	20 Ft. Lbs

Rings 55 and 30 Gallon

A 12 Gauge ring will require a torque of 60 foot pounds to properly close and seal. Do not apply more than the recommended torque. this could cause the bolt threads to strip making it difficult or impossible to remove. A 16 Gauge ring will require a torque between 8 and 15 foot pounds.

Plastic Drums

All plastic drum bungs require a torque of 250 inch pounds.

UN Fiber Drums

Place cover and ring on the drum. Close the ring's lever and lock.

Under DOT regulation, any changes made to the type and dimensions of the closures or the method of closure many constitute a change in design type of this package, voiding the certification we have marked on it and requiring retesting and recertification.

WE WARRANT our containers to be free of defective materials and workmanship under normal use and service provided the container are used within the limits of their design and recognized capabilities. In the event that any container is found to be defective whether as a result of breach of warranty or negligence on our part, the sold and exclusive remedy shall be limited to repair by us of defective materials and workmanship if, in our discretion, such replacement is necessary.

THIS WARRANTY shall not apply to any container which has been subject to accident, negligence alteration abuse or misuse. We neither assume nor authorize any other person to assume for us any liability in connection with our containers. We make no warranty whatsoever in respect of accessories or parts not supplied by us. Any affirmation of fact, description, sample, model or promise made or provided by us shall not be deemed to create an express warranty that any container shall conform thereto to thereto or therewith and shall not be deemed part of the basis of the bargain. There shall be no responsibility or liability on any claim for damage or leakage once a container has been used for a shipment received in good order.

IN NO EVENT shall we be liable for damages or injury to persons or property and in no event shall we be liable for any incidental or consequential damages, except for consequential damages for injury to persons suffered in the case of consumer goods as that phrase is defined in the Uniform Commercial Code. Should any container prove so defective as to preclude the remedy of defects by repair or replacement, whether as a result of breach of warranty or negligence, the sole and exclusive remedy shall then be refund of the purchase price for such container.

EXCEPT AS SET FORTH IN THE FOREGOING WARRANTY, IT IS EXPRESSLY AGREED THAT NO WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, NOR OTHER WARRANTY, EXPRESS, IMPLIED OR STATUTORY, IS MADE BY US.

FROM :A&B Properties FAX NO. :2067220518 Feb. 04 2009 07:59PM P2

Seattle Barrel Company
4716 Airport Way South
Seattle, WA. 98108
206-622-7218

Drum Closure Notification Form

Pursuant to the requirements of the Deaprtment of Transportation in CFR 49 Part 178.2 (c)(1), this is your notification of the closing method used for the containers sold to you on the attached delivery ticket or bill of lading. This method of closure should be used to ensure that your containers have been closed in the same manner as when they were initially tested. If there is any question regarding proper closing methods, contact your local salesperson or manufacturing facility.

TIGHT HEAD STEEL DRUMS
OPEN HEAD STEEL DRUMS WITH FITTINGS

To close fittings, tighten with a torque wrench to the following manufacturer's recommended torques:

PLUG TORQUE IN FOOT-POUNDS

	TRISURE				RIEKE		
PLUG SIZE	STEEL PLUGS		POLY PLUGS		STEEL PLUGS		POLY PLUGS
	POLY GASKET	OTHER GASKET	POLY GASKET	OTHER GASKET	POLY GASKET	OTHER GASKET	
¾"	20	12	8	20	20	15	9
2"	30	20	30	30	40	30	20

FULL OPEN TOP STEEL DRUM WITH BOLTED RING CLOSURE

1. Place cover on drum.
2. Snap the closing ring over the cover and top lip of the drum. Make sure the ring's lugs point down below the ring. Also make sure that the bottom edge of the closing ring engages under the top lip of the drum.
3. Insert the bolt through the lug without threads. Next, screw on the locking nut. Finally, screw the bolt onto the threaded lug.
4. While tightening the bolt, tap the entire perimeter of the ring with a mallet, starting directly across from the bolt.
5. Tighten the bolt until 50 foot pounds of pressure is reached. The cover and ring should not spin, but the free ends of the rim should have a ¼" space maximum.
6. Drums closed in this manner have met the UN performance test requirements as specified in the container markings.

DISCLAIMERS

Tests were conducted using water for liquid containers, sand and sawdust for solid containers, or water and anti-freeze mixture for plastic drums used to contain liquids. Your product may adversely affect container materials, bung threads, or closing devices. Product compatibility with the container is a shipper's responsibility.

These instructions for container closure are based upon the closure methods used to enable these containers to pass the United Nations test requirements as outlined by the UN marking on the package.

These closure recommendations do not take into account any hazards present in your facility, or the handling, filling or shipping of your product. The use of sparkless tools and proper safety equipment is recommended. Consult your supervisor for your special safety precautions.

Any containers used for packing of hazardous materials shoud be inspected prior to filling and shipment. Containers with obvious damage or deterioration should not be filled or shipped.

Myers Container CMS

MYERS CONTAINER LLC
CONTAINER MANAGEMENT SERVICES, LLC

5.0 Supplemental Closure Requirements

Designs 0801, 1001, 1606, 3011, 5532, 5585, 8501 and G5501
These designs are only 7A Compliant with a 4 mil Polyethylene (LDPE) Bag Liner installed as described in 4.0 a (above). LDPE Liners up to 10 mil thickness will also meet this requirement providing the bag can be installed and top tied as described above. Liners for the 55 gallon size may be cut to accommodate smaller sized packagings, and must be inspected to assure that the liner is not accidentally punctured. This liner may be installed during manufacturing or by the filler / shipper of the packaging.

Design 5556 – Narrow Open Head 21"
For qualification as a UN packaging, this drum must utilize the 16-gauge bolt ring (utilizing a 5/16" Rivet Nut Bolt Ring - bolt threading into a lug which encloses a nut) provided with the drum order. The bolt ring must be closed tight in such a manner as to prevent the nut from pulling out of the lug and to a point of less than 10 foot-pounds. The bolt ring manufacturer has advised MCC LLC that a torque of 10 foot-pounds or more must not be exceeded. Design 5556 was tested and passes UN Testing at 7 psi.

Design 5559
For qualification as a UN packaging, this design requires a 30 mil liner to be installed during Manufacturing or by the filler / shipper. The liner must be installed as prescribed in 4.0 b.

Design 5577 and 5583
For qualification as a UN packaging, this design requires a 15 mil liner to be installed during Manufacturing or by the filler / shipper. The liner must be installed as prescribed in 4.0 b.

Design 5592
This design requires a Pro55M Liner. The liner must be installed as prescribed in 4.0 b, and the cover must be assembled with a head press.

Design R5503
Follow the instructions in 1.0 above and torque to 75 ft. pounds instead of 60 ft pounds.

These instructions for container closure are based on the closure methods used to enable the packagings sold to you to pass the United Nations test requirements as outlined by the UN marking on the package. The UN Marks are found on the side of the packaging.

A UN Test Summary verifying that the packaging sold to you has met the testing requirements is available upon request.

Myers Container

CMS

MYERS CONTAINER LLC
CONTAINER MANAGEMENT SERVICES, LLC

49 CFR 178.2 (c) Notification for Myers, CMS and Other UN Packagings and UN Assembly Instructions for Non-Bulk MCC LLC and CMS Packagings

Routing Instructions: ***This document must be passed along with the container within your facility, or to whom the packaging is transferred, and ultimately to the personnel responsible for shipping and closure. It must be used as a training document to complete closure of your container.***

Caution: ***Empty Drums may be pressurized and extreme caution is necessary to prevent injury while removing the drum ring. Drums may become pressurized due to travel between low to high altitudes and / or changes of ambient temperatures.***

Requirements not met at the time of transfer

Due to conditions outside of the control of MCC LLC and CMS, the person to whom the packaging is transferred and the filler and the shipper, are notified that all closures may not be closed according to assembly instructions when the packaging is transferred to your inventory. The filler and ultimately shipper must verify that all closure specifications have been met (including those supplied during manufacturing, and if applicable, by a person to whom the packaging is transferred). When present (including and not limited to) the proper assembly of the following closure components must be verified:

UN Steel Drums – 12 Gauge Bolt ring with lugs and 5/8th inch nut and bolt (1A2 only), cover gaskets (including 3/8th inch sponge, 7/16th inch EPDM, 7/16th inch high density rubber, 0.265 inch black tubular SBR), 2" plugs, ¾" plugs, plug gaskets (see page 4 for types of plugs and gasket combinations), all side fitting plugs (see 2" plug data in page 4) and specifications relating to bag liners (4 mil) and other liners (15 and 30 mil and a Pro55M liner). Liners which extend over the curl of the drum may only be used where specified by the design type and corresponding UN performance testing. 4 mil plastic bag liners may be used in UN packagings providing the liner does not interfere with the drum closure.

UN Poly Drums – All closures devices (including those where assembly requirements are embossed on the packaging), rings, all gaskets and plugs.

UN IBC Packagings (Rigid Composite and Metal) – Rings, covers, all gaskets, caps, plugs, and valve systems (including bolts and caps).

DOT Portable Tanks (DOT 57) – Also Known as Asset Tanks – Rings, covers, all gaskets, plugs and valve systems.

In addition to the above, if your design requires any additional or different specification closures or other requirements not met at the time of transfer of this container, a supplemental notification letter will be provided by a MCC LLC or CMS Account Manager or will be attached to the BOL.

It is the responsibility of the Shipper to determine the suitability of any Myers or CMS, LLC packaging for transportation of hazardous materials by Air. For shipments by Air, the shipper must refer to all applicable provisions (including the Hazardous Materials Table and 172.321) in 49 CFR, and take into account the characteristics material being shipped and the performance capabilities of the container sold to you.

Myers Container **CMS**

MYERS CONTAINER LLC
CONTAINER MANAGEMENT SERVICES, LLC

Product compatibility with the container is a shipper's responsibility. Any containers used for packaging of hazardous materials should be inspected prior to filling and shipment. Containers with obvious damage or deterioration should not be filled or shipped.

Type(s) and Dimensions of Closures (including gaskets) and other Components

It is the responsibility of the filler and / or shipper to confirm that the original closure components provided are utilized when assembling and closing this packaging (and if appropriate when added after manufacturing). These components are listed above for each packaging type. Utilization of different types of liners, bag liners, rings, bolts, nuts, covers, cover gaskets, caps, cap gaskets, plugs, plug gaskets or other components invalidate the performance rating.

MCC LLC and CMS produced Steel non-bulk packagings

UN Drum Assembly Requirements (closure instructions) for are attached to the Bill of Lading and consist of pages 3 and 4 of this notice. This notice is also found on our web site at:

http://www.myerscontainer.com/

New Poly Drums, Reconditioned Poly Drums, New IBC's, IBC's supplied by continued qualification & packagings originally manufactured by other suppliers

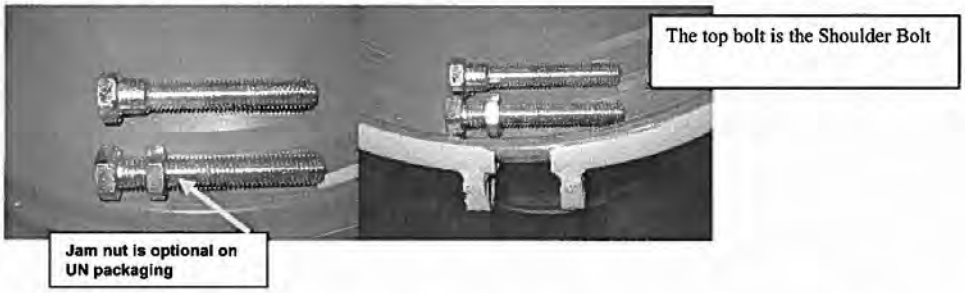
Pages 1 and 2 of this notice, and assembly requirements from the original manufacturer (closure instructions) are attached to the BOL and are to be used in lieu of information found on pages 3 and 4 of this notice. If available, you will be provided the complete notice and assembly instructions from original manufacturer.

---

12 Gauge Bolt Ring Installation –

The photograph below illustrates the different bolt types that may be used on selected designs for MC LLC and CMS LLC packagings. The bolt on top is referred to as a shoulder bolt. The larger bolt shaft near the head is inserted into the unthreaded lug and is closed using the same technique used when using a threaded bolt. UN testing confirms that the shoulder bolt is equivalent to a conventional bolt with or without a jam nut. Both types of bolts may be used on MC LLC and CMS LLC designs inner changeably.

The jam nut is optional for all UN packaging and required for 7A packaging.



Document: UN178.2 Issued: 6/14/2010 Authority: UN Lab Manager Page 2 of 5

Myers Container

CMS

MYERS CONTAINER LLC
CONTAINER MANAGEMENT SERVICES, LLC

MCC LLC and CMS Produced - UN Drum Assembly Instructions

In order for your Myers Container or Container Management Services, LLC drum to safely perform to its rated ability, these assembly instructions need to be strictly adhered to. Any other method of assembly or the use of any drum components (rings, gaskets, or fittings) that are not specified in this design type will immediately invalidate the UN and DOT performance rating of the drum. The shipper must verify the appropriate use of a liner. A liner which extends between the gasket and the curl may only be used if UN Performance tests indicate a liner was used in testing.

Fig 1	Fig 2	Fig 3	Fig 4	Fig 5

1.0 12 Gauge Bolt Ring Installation

a. Place the cover on the drum, making sure the cover gasket is seated against the lip of the drum opening (the curl) and the gasket recess on the cover. The gasket should not protrude beyond the cover or the drum curl.

b. Place the bolt ring onto the drum. Make sure that the bolt ring is oriented so that the lugs are positioned below the top surface of the drum. You will be required to pound on the cover with your palm, or a rubber mallet, or use a head press to make sure it is centered on the drum curl. Check to see that the cover and drum curl are pinched together and within the recess of the ring.

c. Thread the bolt into the lugs,and tighten to a minimum of 60 ft-lb of torque. It is necessary to hammer around the circumference of the ring while torquing in order to further seat the head onto the drum. Continue hammering on the ring circumference and torquing the bolt until the torque is stabilized at 60 ft-lb, and does not loosen when further hammering on the ring circumference is performed. The ring ends must not touch when the ring has been torqued. If a jam nut is used on bolts having threads up to the head (see photo on page 2), it must be placed on the bolt, between the drum ring lugs, and tightened against the un-threaded lug. **As previously specified, the jam nut is optional**. A Shoulder Bolt does not require a Jam Nut.

d. It may be necessary to check the torque and tighten if necessary prior to shipping.

16 Gauge Bolt Ring Closure - See page 5 - Design 5556

Myers Container

CMS

MYERS CONTAINER LLC
CONTAINER MANAGEMENT SERVICES, LLC

MCC LLC and CMS Produced - UN Drum Assembly Instructions

2.0 Fitting Installation

The table below shows the as tested torque that must be applied to each drum fitting to assure proper container performance. (All measurements are in foot-pounds.)

	Type I - Tri-sure ® Octagon Base, Round Head Plug Inserted in ® Tri-sure Flange						Type II – Rieke Serrated Base, Hexagon Head Plug		
Plugs	Steel	Steel	Self-Gasketing Polyethylene	High Density Polyethylene	Polypropylene & Nylon	Polypropylene & Nylon	Steel	Steel	Nylon
Gasket Material ------ Plug Size	Rubber (Buna-N and EPDM)	Poly-Irradiated (Polyethylene)	None (Integral Gasket)	Rubber (Buna-N and EPDM)	Poly-Irradiated (Polyethylene)	Rubber (Buna and EPDM)	Buna-N and E.P.T. Gasket	Irradiated L.D. Poly (Poly) Gasket	Poly and E.P.T. Gasket
2" Closure Torque	20	30	12	15	30	20	30	40	20
3/4" Closure Torque	12	20	5	8	8	8	15	20	9

3.0 Cap Seals

Cap seals must be installed by filler when non-metal flanges are used. For all fitting systems, the shipper is responsible to assure that fittings are properly torqued before shipment.

4.0 Bag Liners and Liners

a. If a bag liner is required by the drum design type it must be installed into the drum and the top tied closed in a horse-tail fashion before installing the drum cover and ring. During testing, the MCC LLC UN Test Lab top tied and secured the horse-tail by removing air to the extent possible, twisting and folding the horse-tail and securing with ordinary duct tape.

b. If a liner is required by the drum design type it must be installed into the drum, extended over the top drum curl, making sure there are no overlaps in the liner as it goes over the drum curl.

- Fill the container (bag) with material.
- Twist the top of the bag closed tightly to secure the material and seal the bag with a minimum of 6-3/8" cable tie. (zip tie)
- Carefully compress the bag of material into the container and fold in top flaps.
- Tape top flaps with 3" wide poly packaging tape with the tape extending a minimum of 2" over the edges. (Tape in a "H" style pattern)

^^Back To

Click Logo>> Covalence Adhesives for the Link to Approved Duct Tape Used to Close Our Boxes

^^Back To

Click Logo>> Grip Rite Fasteners for the Link to Approved Nails Used to Attach Our Boxes to the Pallets

^^Back To

Click Logo>> Grip Rite Fasteners for a Link to Brochure for Approved Nails Used to Attach Our Boxes to the Pallets

^^Back To

The BEST Haz Mat Containers in the Industry

CleanEarth SYSTEMS 1-800-282-4293

Overnight Distribution Throughout the U.S.

Home | About Us | Contact Us | Request A Quote

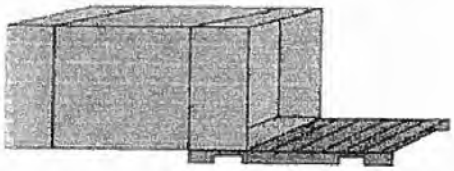
- Products
- Clean Earth Difference
- Compare
- Storage
- Savings
- Locations
- News Updates
- Regulations
- Set-Up And Closing Instructions
- Useful Links

SET-UP AND CLOSING INSTRUCTIONS

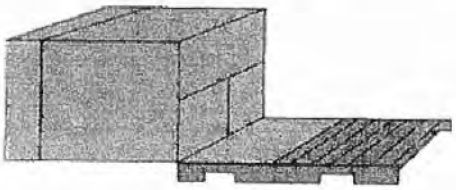
<<Back To B

Haz Box And Cubic Yard Box Set-Up Instructions

- **FIRST**
  Open Box
  Place On Side



- **SECOND**
  Square 1 Flap On One End Of Pallet
- **THIRD**
  Nail That Flap To Pallet and fold under remaining flaps so that nail is not exposed to liner (prevents puncture of liner)
- **FOURTH**
  Fold Remaining Flaps In



GALAXY INSTRUCTIONS

Contents:

1- 1 Cubic Yard Polywoven Container
1- 6 Mil. Liner
2- Closure Ties

Instructions:

Step 1: Unfold container once until two sides are at 90 degrees to each other (see picture).

Step 2: Locate lift loop on inner fold (see picture).

Step 3: Place foot on side of container currently resting on ground and with your hand, pull lift loop until remaining sides "pop" out thus forming the container (see picture).

Step 4: If unit is to be mounted on pallet, locate loops on each lower corner and secure with nails to pallet. At your request, Advanced Environmental Solutions can supply nails with large heads.

Step 5: Pull the duffle top out and around the outside of the container to make the interior cavity of the container easily accessible.

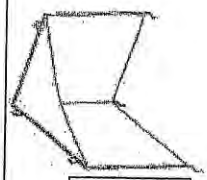
Step 6: Place liner in the container. Stretch the liner's extra portion out and around the container's outer walls to make the interior cavity of container easily accessible.

Step 7: Fill the container with materials.

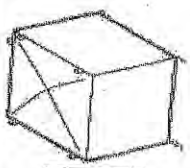
Step 8: Once full, twist the liner top and close with closure tie.

Step 9: Fold opposite flaps on to top, secure with fabric ties.

Step 10: Fold lid across the top and secure with fabric ties.



Step # 1 & 2



Step # 3

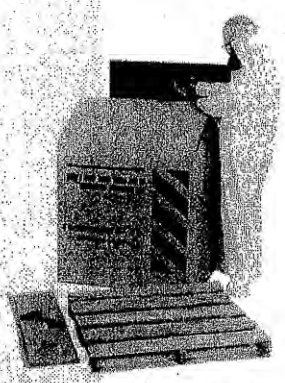
If you are experiencing any difficulties assembling your container, contact us at:

ADVANCED ENVIRONMENTAL SOLUTIONS, INC.

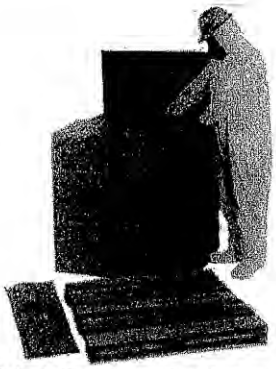
Phone: 800- 275-3549 email: help@advenvironmental.com
P.O. Box 1152
Kent, Washington 98035-1152

QUESTAR

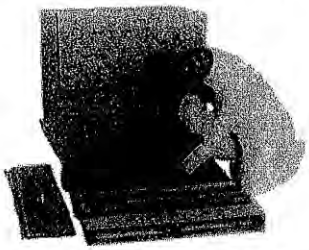
Instructions for Set-Up and Use



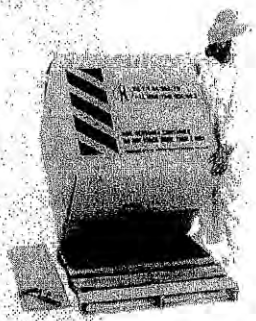
Step 1: Fold top flaps in, turn container upside down, and fold bottom flaps per instructions provided.



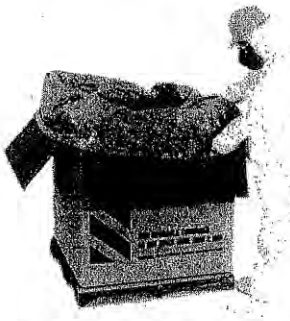
Step 2: Lock die cut bottom flaps leaving one bottom flap unsecured.



Step 3: Place container onto pallet and, using supplied Tri-Wall nails, nail each corner of the unsecured bottom flap to pallet as shown.



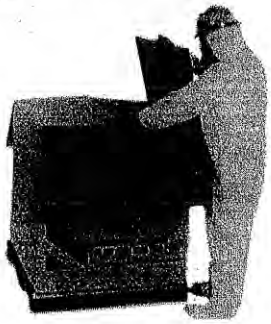
Step 4: Pull container upright onto the pallet.



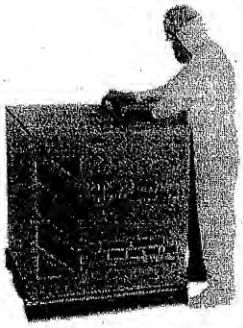
Step 5: Insert the special poly liner bag and stretch around the container walls until firmly in place.

Step 6: Fill the container (bag) with the hazardous waste material.

Step 7: Twist the bag closed tightly to secure the hazardous material and seal the bag with appropriate heavy-duty tape (2" pressure-sensitive poly tape or equivalent minimum) or twist tie.



Step 8: Carefully compress the bag of waste into the container and close the flaps.



Step 9: Seal the container permanently with an appropriate heavy-duty tape (2" pressure-sensitive poly tape or equivalent minimum).

Note: Personal protective equipment used will be contingent upon the site, safety and health plan at your company. Consult the safety supervisor at your company for instructions on handling hazardous waste.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Sample Receipt and Log-in

Approvals

Signatures on File
Dennis Bean Date
Laboratory Director

Manjit Nijjar Date
Health & Safety Manager / Coordinator

Terri Torres Date
Quality Assurance Manager

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Purpose

The purpose of this procedure is to describe the procedure for receiving samples into the laboratory for analysis and verifying the preservation of samples. This procedure also describes the process of preserving samples by the laboratory.

2.0 Summary of Method

Sample control personnel receive samples either directly from the client, company courier, or from an appropriate delivery service/carrier and assume custody of the samples when they sign and date the chain of custody. The temperature is then taken via IR Gun, or Stick thermometer if there is a temp blank in the cooler. COCs are immediately pulled from the coolers, signed and dated with time of receipt, and an initial assessment is performed checking for short holds, rushes, critical clients and DOD projects. Short holds and rushes (1, 2 & 3 day) are immediately announced over the intercom. Once the COCs have been pulled from the coolers and assessed, called "Receiving the Cooler" a Sample Receiving Triage and Labeling Checklist form (located in public:\sop\Work Instructions\PM and Sample Control) may be started if needed and then the project managers and department managers will be called to review COCs to ensure project set-up in Laboratory Information Management System (TALS) (the process internally referred to as "COOLER GREETING"). The sample receiving technician then proceeds with log-in of coolers. For projects received that are not in TALS, a copy of the COC will be made by the PM or the person checking in the cooler for project set-up. Log-ins are prioritized based on holding time, turn around times, critical clients and DOD projects. All sample delivery groups (a set of samples received for a project) are assigned a unique laboratory job number. The custodian unloads the samples, lines up the containers according to the COC, verifies the correct containers have been received for the job, insures all appropriate client and sample information is provided on the COC, creates a job number, logs sample information into TALS, completes the sample receiving checklist, labels the containers, and scans Chain-of Custody (COC) into TALS. Specific clients may require documentation on their own cooler receipt forms. Aqueous samples submitted for analysis and marked as "preserved" are verified when samples are unloaded for login by the sample custodian or other qualified person designated by the sample custodian except samples submitted for Volatile and O&G analysis. Samples are placed in the appropriate storage bin and then in the bin's designated shelf location. If required by a specific project or client, the samples are stored under controlled custody.

Whenever possible (if the client can provide a copy of the COC before samples arrive), samples can be pre-logged into TALS before samples are unloaded or received.

Rushes and projects with short holds and critical clients are dealt with within 2 hours of receipt. A page is made over the intercom that rushes, short holds and any critical client projects have arrived. Rushes and short holds must be logged-in within 2 hours of receipt, critical clients and DOD projects must be processed within 2 hours of receipt and 4-5 day projects must be processed on the same day of receipt. Projects that are on a standard turn must be processed the day of receipt or by noon the following day. If a project cannot be processed the day of receipt, the sample's cooler is placed in the appropriate storage until samples are ready for log in. Sample Control will make every effort to login all projects day of receipt.

Once a project has been logged-in and the COC has been scanned by Sample Control, the COC is stored in a bankers box and when the bankers is filled, the box will be stored The project manager then reviews the work order for correctness and updates the status of the samples in TALS. After the project manager has reviewed the information contained in TALS for completeness and accuracy and notified the lab of any short holding time samples or rush samples,. Short hold (SH) samples for wet chemistry are taken to the wet chemistry SH area, logged into the SH log book and paged over the intercom. If wet chemistry unable to run short holds in a timely manner then it is their responsibility for placing the

sample in the appropriate cold storage unit to maintain sample temperature. Rush and short hold samples for Extractions have a log-in summary report printed, the short hold test and samples effected are highlighted, the bin numbers and type of rush/short hold are written on the report and then the report is taken to the Rush/Short Hold notification board in the Extractions department. The samples may be taken directly to an analyst in the Extractions department but should never be left in the department without being handed directly to an analyst.

3.0 Definitions and Acronyms

3.1 Sample - a field derived material of various matrices that is put into a container and removed from the environment under controlled conditions and then brought to the laboratory to undergo analyses or archiving.

3.2 Relinquish - to release, surrender, retire, to let go. Relinquishing a sample is to put it in someone else's charge or responsibility.

3.3 Sample Custodian - an employee that receives samples

3.4 pH - a measurement of acidity or alkalinity (the negative log value of the molar H^+ concentration)

3.5 Preservative - any agent that prolongs the useful life of a material

4.0 **Responsibilities**

4.1 Sample Custodian or designee is responsible to:

- Assess each cooler for short holds, rushes, critical clients and DOD projects, while noting sample dates.
- Receive samples including, logging into the corporate TALS, checking temperatures, filling out sample receipt check lists, verifying sample preservation and condition, labeling sample containers, and noting sample location within the laboratory.
- Inform the lab of rushes (1, 2 & 3 day), short holds, and critical client projects that have been received.
- Inform the Project Manager (or designee) when samples are received and that login review is required.
- Inform the PM directly of any projects received out of temperature and if samples have been compromised such as water in the sample and broken samples received.
- Send out a nonconformance memorandum (NCM) that will also notify the project manager when problems are encountered (i.e.: Broken containers, Out of Temp coolers, Missing Samples, Samples received but not listed on the COC, Compromised Samples).
- Prepare and ship bottle orders to clients.
- Prepare and ship subcontracts and workshares to other labs.

4.2 Project Manager or designee is responsible to:

- Inform sample receiving of upcoming bottle orders and sampling events.
- Participate in Triage of cooler projects and possibly provide the project codes.
- Inform sample receiving of any special requirements such as special sample receipt procedures.
- Build the project to include all requested analyses and analytes.

- Inform the client of sample receipt.
- Verify the login to ensure that the proper samples are linked to the correct analyses and analyte lists.
- Notify clients of any NCMs that are deemed to affect sample results.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1 Specific Safety Concerns or Requirements

5.1.1 Make sure if glass capillary tubes or disposable pipettes are used that they are disposed of in properly marked glass disposal containers.

5.1.2 All samples should be assumed to be hazardous. Samples received broken and/or emitting noxious fumes or odors are to immediately be taken to the fume hood.

5.1.3 Cut resistant gloves should be worn when handling broken glass samples and inspecting or cleaning the coolers.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment

- H-B Instrument™ Durac™ Infrared Thermometer or equivalent
- Digital readout battery operated thermocouple thermometer
- Laboratory fume hood
- Narrow Range pH strips
- Disposable Pipettes

6.2 Supplies

6.3 Most sample containers are provided by ESS. Sample containers include:

- Clear 40-mL VOA vials: HCl, Sodium Thiosulfate, unpreserved.
- HDPE: 1 liter, 500-mL, 250-mL, and 125-mL unpreserved and preserved with H2SO4, HNO3, NaOH and NaOH/Zn Acetate.
- Glass amber: 1 liter, 500 ml, and 250 ml, unpreserved, HCl and H2SO4 preserved.
- Clear wide mouth 32 oz, 16 oz, 8 oz, 4oz, 2oz, glass bottles (for soils)
- Clear 40 ml VOA vials: MeOH, w/stir bar w&l, w/stir bar/DI water w&l, Sodium Bisulfate w stir bar w&l.

- Dropper bottles filled with reagent provided by ESS
- Chain of custody, custody seals, container labels, cooler summary form, coolers, gel ice, temperature blanks and sampling instructions and shipping supplies.
- Colored folders

7.0 Reagents and Standards

7.1 Nitric acid, (HNO3), 1:1 (or 1:3 for shipping), Trace Metals grade, ESS or equivalent

7.2 Sulfuric acid (H2SO4), 1:1, ESS or equivalent.

7.3 Hydrochloric acid (HCl), 1:1, ESS or equivalent.

7.4 Sodium Hydroxide (NaOH) 50%, ESS or equivalent.

7.5 Zinc Acetate (CH2C00), ESS or equivalent.

7.6 Methanol (MeOH), Purge and Trap grade from Fisher Scientific.

7.7 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Various samples/analyses will have different handling, preservation and holding time requirements see Attachment 5, Environmental Sampling Guide for the appropriate preservation, handling and hold times of the various analyses. Attachment 6, Table Recommended VOC Sample Preservation Techniques and Holding Times, outlines the unique preservation schemes for volatile organic analysis.

8.2 The sample pH should be verified as soon as possible.

8.3 This information is then documented on the sample check in list if possible. However due to constraints within TALS the comment tab found in the receipt tab of the login module of TALS can be used.

9.0 Procedure

Upon receipt, open the cooler and assess the COC for rushes, short holds (including pH and dissolved metals needing to be filtered in the lab), critical clients and DOD projects, while noting sampling dates, and then sign, date record the time of receipt on the Chain of Custody. If the date/time of receipt is noted on a separate piece of paper this piece of paper must accompany the original COC at all times as the original record. Initiate a Sample Receiving Triage and Labeling Checklist as needed for each project received. The checklist is located in public:\sop\Work Instructions\PM and Sample Control (see attachment 11). Complete the header information and the Triage Checks sections. Project managers will indicate the project number and any project details on the Sample Receiving Triage and Labeling Checklist during the Cooler Greeting process that is not provided on the common projects sheet. Place a temperature label on the COC or initiate a cooler checklist for every cooler received for the project. This label or checklist is where the corrected and uncorrected temperatures, device used to take temperature, how temperature was obtained (cooler temp or from Temp Blank), cooler description, with or without custody seals (w/o or w/cs), method of delivery to the lab, packing material and coolant type used, will be recorded. See Attachment 9 for an example Cooler Temp Label. If a separate piece of paper is used this must accompany the original COC at all times as the original record. The Sample Receiving Triage and Labeling Checklist can be used to make additional notes.

If indicated on the COC that the client has submitted volatile soil samples in Encores or VOA vials with stir bars, the sample containers must be frozen ASAP. The time the samples are

placed in a freezer must be noted in the sample freezer storage logbook. This will additionally must be noted in an NCM.

Samples delivered by the TestAmerica courier do not need to be relinquished by the courier and received by sample control. If the cooler is in the possession of the courier at all times, a note on the bottom of the COC stating "at lab" and the time is sufficient. The "at lab" time will be the time of receipt. If samples are received without a COC, the following steps must be taken:

9.1.1 Sample control must notify the appropriate PM upon receipt of cooler (not at the time the cooler is actually logged in) and initiate a nonconformance memorandum (NCM). See Attachment 13 on how to create and edit an NCM.

9.1.2 If the samples were sent by an inter-company subcontract lab, sample control or the project manager must notify the subcontract lab's sample control supervisor by e-mail (cc: the TA Seattle/Seattle PM) upon receipt of the cooler. Include additional information as needed (i.e. short hold times).

9.1.3 If the samples were submitted by a party other than an inter-company lab, a TA Seattle/Seattle PM must notify the point of contact at that organization.

9.1.4 If a response is not received within 24 hours, either the TA Seattle PM or sample control are responsible for the follow-up.

9.1.5 The process must be fully documented using e-mails, NCMs and phone logs. Copies of this documentation must be maintained.

9.2 Open or supervise the opening of sample coolers or containers in the sample receiving area of the laboratory. If it is suspected that a sample has broken or is leaking into the cooler, open the cooler inside the hood.

9.3 TestAmerica Seattle does not accept radioactive samples. It is the client's responsibility to ensure that samples being sent for analyses within the TestAmerica Seattle facility not have radioactivity above background.

9.4 **Taking and recording sample temperatures.**

9.5 Corrected and Uncorrected temperatures must be recorded. As a general rule of thumb, all samples will need to have a temperature recorded whether on ice or not. If samples are hand-delivered from the client and are not on ice, take the temperature of the samples with the IR gun, note in the checklist and create an NCM stating samples were not received on ice when it is applicable. Place the samples in the walk-in until log-in when applicable. For samples that have a temperature that is outside the required temperature conformance, the PM (or their designee) must be notified immediately in person.

9.5.1 For samples received in a cooler with a temperature blank, the temperature of the temperature blank should be recorded using a calibrated digital thermometer. Remove the cap of the temperature blank, insert the thermometer to a point where its tip is approximately in the center of the temperature blank, wait 10 to 15 seconds for the reading to stabilize, and then record the temperature.

9.5.2 If a temperature blank wasn't provided, the temperature blank exceeds the temperature criteria or if the project is for the department of defense (DOD); select a sample container from the center of the cooler. If available select an amber glass container. Remove any bubble wrap or packaging from the sample and take the temperature of the container using the IR gun. Position the IR gun 1/3 of the distance from the base of the container at a distance of approximately 0.5 to 1 inches from the container, press the trigger of the IR thermometer and direct the IR beam at a 70 to 90

degree angle, and then record the temperature.

9.5.3 If a temperature blank wasn't provided and the selected sample container exceeds the temperature requirement; take the temperature of at least two more containers from either edge of the cooler using the IR gun. Position the IR gun 1/3 of the distance from the base of the container at a distance of approximately 0.5 to 1 inches from the container, press the trigger of the IR thermometer and direct the IR beam at a 70 to 90 degree angle, and then record the temperature. Note if samples were taken on the same day of receipt with evidence of thermal preservation this step is not required, however this must be noted in an NCM.

9.5.4 If one of the recorded temperatures is within requirements notify the appropriate PM and initiate a nonconformance memorandum (NCM). Include details of all temperatures taken and any additional observations of the cooler condition. Proceed with the log-in.

9.5.5 If the temperature blank and the selected sample container exceeds the temperature requirement or if there was no temperature blank and all the selected sample containers exceeded temperature requirements, the following steps must be taken:

9.5.5.1 Make note of any additional observations of the cooler condition or take photographs of the affected coolers and place in an NCM and or on the Sample Receiving Triage and Labeling Checklist if a log-in is not initiated.

9.5.5.2 Sample control must notify the appropriate PM immediately in person. If the PM or their designated back-up is unavailable (ie. Saturday receipt) an e-mail must be sent to the PM, their immediate back-up, and the CSO Manager. Samples will not be logged in until approval to proceed has been received by the PM. All samples should be placed in the walk-in until the PM directs sample control how to proceed.

9.5.5.3 If the samples were sent by an inter-company subcontract lab, sample control must notify the subcontract lab's PM by e-mail (cc: the CSO Manager). Samples will not be logged in until approval to proceed has been received by the PM. All samples should be placed in the walk-in until the PM directs sample control how to proceed.

9.5.5.4 If a response is not received within 24 hours, sample control will follow-up with the PM and the CSO Manager. For rush and short-hold samples the PM needs to be notified that the TAT or hold time might be missed if a response is not received ASAP.

9.5.5.5 The process must be fully documented using e-mails, NCMs and phone logs. Copies of this documentation or photographs must be maintained in the master job folder (may be done electronically in TALS).

Note: If the temperature blank was received frozen, note on the receipt checklist and write an NCM. If samples were delivered with no cooler, take the temperature with the IR Gun, and also note in cooler section under Receipt/Info Tab under Cooler Description list type as "none".

Note: The IR gun cannot be used to measure the temperature of metal tubes. If a temperature blank or more suitable sample container was not packed in the cooler with the metal tubes, take the temperature of a tube at one of its plastic end caps. Describe the anomaly in a NCM.

9.6 BP LaMP require the monitoring of sample temperatures every 15 minutes during the unloading

and labeling of BP, Arco and OPLC sample containers. A Party Table in the sample control area can be used for this purpose. Fill the table with loose ice from the ice machine in sample control. Place the samples in the ice on the table. Use the IR thermometer to measure sample temperatures as described above and record these temperatures on the Login Container Summary Form. Maintain appropriate sample temperatures until log-in is complete and samples are placed in the appropriate cold storage units. Samples can also be unloaded onto a cart and the cart placed in the walk-in while being processed. Indicate on the Log in Container Summary Form if this is done instead of taking the temperatures every 15 minutes.

9.7 Prioritization

9.7.1 Place all short-hold projects in the yellow taped area in sample control. Assign each project a priority number with the shortest hold project getting the lowest number.

9.7.2 Place all rush TAT projects in the red taped area in sample control. Assign each project a priority number with the shortest TAT getting the lowest number.

9.7.3 Place all other projects in the green taped area in sample control. Assign each project a priority number based on shortest TAT and/or hold time with the shortest TAT and/or hold time getting the lowest number.

9.7.4 Call Cooler Greeting for the project managers to ensure project set-up when all coolers received at a given time have been prioritized. Cooler greeting will typically occur at least two times per day, once in the morning after FedEx and UPS receipts and in the afternoon upon the courier return. This review by the project managers will also insure the prioritizations do not need adjustment. If they do the priority numbers will be reassigned at this time. At this time the project managers will complete the PM/PMA section of the Sample Receiving Triage and Labeling Checklist.

9.8 Cooler inventory and Sample log-in

9.8.1 Log-in should start with any short-hold designated projects (yellow) starting with the lowest priority number, then move through to any designated Rush TAT projects (red) starting with the lowest priority number and then finally to the standard designated projects (green), starting with the lowest priority number.

9.8.2 Open or supervise the opening of sample coolers or containers in the sample receiving area of the laboratory. If it is suspected that a sample has broken or is leaking into the cooler, open the cooler inside the hood.

9.8.3 Verify that the chain-of-custody information is complete, including client name & address, project name, unique sample identification, location, time and date of sample collection, preservation type, sample type, collector's name, analyses needed, TAT required, relinquished by name with date and time and any special remarks concerning the samples.

9.8.4 The samples and accompanying chain of custody undergo an initial inspection by sample control staff **immediately** using the TA Seattle Sample Acceptance Criteria Checklist (see Section 10.23.2 for TALS checklist questions or Attachment 2 for example client checklist) and Sample Acceptance Policy (see Attachment 3). Sample inspection information, non-conformances and corrective actions must be documented in an NCM in TALS. Non-conformances documented in TALS include: limited sample volume, air bubbles in VOA vials that are larger than 1/4 inch, replacement of cracked lids, broken containers, samples received out of hold, samples received with less than 1/2 of their hold time remaining, samples preserved incorrectly and samples not received as well as any other anomalies that would merit an NCM.

9.8.4.1 ***BP LaMP and BNSF projects received without custody seals require that an NCM be generated to inform the PM of the program requirement discrepancy.***

9.8.4.2 If multiple containers have been received for a sample, and one or more are compromised, those sample bottles will be marked by sample control with “DO NOT USE”. Take a custody seal and seal the bottle. Write on the custody seal “DO NOT USE”. If all samples containers available for any test are compromised for a sample, the PM must be contacted immediately and then the client will be contacted immediately thereafter for instructions. However, if there are still adequate un-compromised containers preserved appropriately to perform all requested tests, the information is simply recorded in the NCM, the affected containers are labeled by sample control with “DO NOT USE” and the analysis will proceed using the unaffected containers.

9.8.5 A cooler receipt form is completed in TALS. The form may be custom for the client or the standard receipt form designed by the corporate TALS developer group. (See Attachment 2).

9.8.6 Use the Unpacking checks section of the Sample Receiving Triage and Labeling Checklist to assist in completing and recording the information below:

9.8.6.1 Verify that the sample IDs, sample dates and times on the containers match the sample IDs, sample dates and times listed on the COC.

9.8.6.2 Verify that all sample labels are legible.

9.8.6.3 Verify that all sample containers are intact and are not leaking

9.8.6.4 Check all VOA vials for headspace.

9.8.6.5 Check that VOA vials are labeled as preserved.

9.8.6.6 Check for multiphasic samples.

9.8.7 Verify that the tests indicated on the accompanying chain of custody match the containers received.

9.8.8 Verify that the proper sample containers and preservatives were used and adequate sample volume was collected. For any sample submitted in an inappropriate container, determine if extra volume exists in a correct container type. If required, preserve the additional volume using the appropriate reagent as described in Section 10.28. When required by contract and for all aqueous samples scheduled for metals testing, pH adjustments must be documented in TALS via an NCM. pH adjustments should not be recorded on a post-it note. If necessary record pH adjustments onto the back of the original COC or on the accompanying Sample Receiving Triage and Labeling Checklist. Document in an NCM. For BP LaMP, and DOD projects, pH adjustments must be documented in the **Login Container Summary Form**. Document on this form the amount of acid added to each sample and the lot # of the acid. For DOD projects, document the pH strip lot number on the form as well. Keep the Login Container Summary Form with the COC and place in the bankers box at the end of the business day.

Note: When a BP LaMP consultant submits soil samples for volatiles in a bulk container, Sample Lab personnel will preserve the samples within 48 hours of collection according to one of the options specified by BP LaMP in Attachment 7. In the event that samples are

received beyond the 48-hours of collection, the laboratory PM should request the consultant to authorize (in writing) the preservation and analysis of the soil samples. Document the event in an NCM.

When any client submits volatile soil samples in bulk, lab personnel must preserve the samples with in 48 hours of collection. Sample control must deliver the samples to the lab ASAP.

9.8.9 Open sample containers inside the fume hood if needed. There is a metal tray to put over the sink in the fume hood if more space is needed.

9.8.9.1 Checking the pH: Basically two methods can be employed. Take a small aliquot from the sample bottle using a capillary tube or disposable transfer pipette. Drop the aqueous sample onto the pH paper. The other is to pour a small amount of the sample into a plastic cup container and then dip the pH paper into the aqueous sample in the plastic container. If further confirmation of the pH is needed, then a more narrow range of pH paper is used. The range is dependent on the pH being checked. BP LaMP requires that preservation verification be checked using a narrow range pH paper. Note any adjustments made on the Login Container Summary form as well as in an NCM. Do not check the pH of waste samples, water VOA vials, and O&G samples.

9.8.10 Discard any sample remaining in the plastic container into a proper waste satellite container and discard the plastic cup container. A satellite waste container is available in the sample control hood.

9.8.11 If all samples are properly preserved, mark on the checklist in TALS. If the project is a DOD project, make note of this on the Cooler Receipt Form. For BP LaMP and DOD projects, record the pH readings, adjustments and reagent lot numbers on the Log-In Container Summary Form.

9.8.12 If any samples are not properly preserved, record the sample ID and generate an NCM which notes the samples not properly preserved. Preserve samples as soon as possible following the instructions in section 10.29.

9.8.13 A special requirement exists for water samples submitted for metals analysis (EPA Methods 200.7, 200.8, 245.1, 6010, 6020 and 7470). If the pH is < 2, no corrective action is required. If the pH is > 2, 1:1 nitric acid is added with mixing to lower the pH to < 2 and then it's allowed to stand for at least 24 hours before re-verifying the pH. The sample is labeled as preserved by sample receiving (green label) and the time and date of preservation is noted on the label as well as the NCM to insure that the 24 hour period has passed prior to the read back by the analyst prior to digestion.

9.8.14 **Note: For BP LaMP samples needing a pH adjustment, remember to add the same amount of preservative to the field blank. If a field blank wasn't provided, prepare a method blank from laboratory grade deionized water and add the same amount of preservative to the method blank. Log the method blank into TALS for analysis. Note in Login Container Summary and in an NCM.**

9.8.15 Verify the remaining holding time for each sample and parameter. If the shipment contains samples with short holding time tests required (including pH and metals samples requiring filtering in the lab), sample receiving will enter the information either on the Short Holding Time Analyses notification form (Attachment 4) which is posted in

a binder located in the wet chem department. Place the short hold samples for wet chemistry on the bench next to the Short Holding Time Analysis binder. For Extractions print out a log-in summary report with the short hold test and sample(s) highlighted and the storage location of the samples and type of rush/short hold noted on the report. Place the log-in summary report for extractions on the Rush/Short-Hold notification board in the Extractions and notify the department of the addition to the Rush/Short-Hold board. Sample receiving will then announce over the paging system that samples with short holding time tests have been received and are available for analysis. The project manager will then log-in review the job and communicate to the laboratory about the rush or short holding time status via email.

9.8.16 For BP LaMP projects, the person who receives the samples will retain custody of the samples until they are placed in storage, that is, that person needs to unload the cooler, log the project into TALS, label the samples and place them in the appropriate storage units. The technician will then relinquish custody of each container to the appropriate storage units using the “Internal Chain of Custody” feature in TALS. If this is not possible then the person will have to relinquish custody to another sample control technician.

9.8.17 Geographical Area of Origin

9.8.18 Sample control personnel must note the geographical origin for each sample shipment. The laboratory maintains a USDA permit to receive samples from foreign countries (including Hawaii and Canada), quarantined areas within the continuous United States and other permitted laboratories. Areas currently under quarantine in the U.S. can be identified on the USDA map posted in the department. The permit requires the lab to follow a compliance agreement. According to this agreement and as it relates to sample receipt,

1) Only an authorized sample custodian may handle and process foreign and regulated domestic soil shipments.

2) The laboratory shall not receive regulated soil samples or water residues (e.g., leachates) from other laboratories without written approval from the local USDA PPQ office AND unless such laboratories have valid Soil Permits and Compliance Agreements for the import of foreign soil.

3) Upon receipt and until such time it is heat-treated but no longer than six months, store regulated soils in the foreign soil fridge located in the lab. Foreign soils requesting volatiles analysis can be placed in the foreign soils fridge. These soils must have a “volatiles first” sticker placed on the cap. Samples are labeled and are then taken directly to the volatiles department to be extracted. Once extracted the volatiles department will place the samples in the foreign soils fridge in the main lab The key for the foreign soils fridge is hung on a post directly across from the fridge.

4) As appropriate, label each sample container as “Regulated Soil under USDA Permit (Autoclave Before Disposal)”. If there are numerous soil samples, place the samples into a bag or cardboard box and place a “Regulated Soil under USDA Permit (Autoclave Before Disposal)” on the outside of the bag or box. If the client only provided 2 oz. jars, place the square sticker on the cap of the sample and either attach the barcode sticker with a rubber band to the container or leave the barcode stickers in the box with the samples.

9.8.19 While a project is awaiting log-in or labeling, the associated samples may be repacked with ice in the original shipping container or a TA cooler. A tagged or properly labeled and iced cooler may be stored in the walk-in or allowed to sit out in the sample control area.

The samples are then logged into the corporate TALS per the TALS instructions in attachment 8. Note if logging in samples that have Sites and Events assigned to the project see the instructions in attachment 12. If for any reason the sample control technician must add a method to a job from the lab Methods instead of the Analysis Groups from the project they must notify the PM via an NCM. The following is a summary of the login information recorded:

9.8.20 A job number is generated for each project received into the laboratory; the following information is recorded:

- Date and Time received
- Sample custodian
- Shipping method
- State or Province and Country of origination
- Cooler information
- ID of the thermometer used to monitor cooler temperature
- Chain of Custody number (for DOD or Jacobs projects)
- Container information and laboratory storage location
- The laboratory assigned sample number (sequential, starting with 1)
- The laboratory assigned container number (sequential, starting with A)
- Service Center Revenue Tracking (for coolers received from a TA service center, sample receiving personnel are responsible for scanning the label from the inside of the cooler lid; see Attachment 10)
- Any additional comments

9.8.21 The Standard checklist is completed which includes the following questions:

- Radioactivity either was not measured or, if measured, is at or below background
- The cooler's custody seal, if present.
- The cooler or samples do not appear to have been compromised or tampered with.
- Samples were received on ice.
- Cooler Temperature is acceptable.
- Cooler Temperature is recorded.
- COC is present.
- COC is filled out in ink and legible.
- COC is filled out with all pertinent information.
- Field sampler’s name present
- There are no discrepancies between the sample IDs on the containers and the COC.
- Samples are received within Holding Time.
- Sample containers have legible labels.
- Containers are not broken or leaking.

- Sample collection date/times are provided.
- Appropriate sample containers are used.
- Sample bottles are completely filled.
- There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs
- VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.
- If necessary, staff have been informed of any short hold time or quick TAT needs
- Multiphasic samples are not present
- Samples do not require splitting or compositing
- Residual Chlorine checked.

9.8.22 Samples are logged into the laboratory TALS, a central database for project tracking and includes the following information:

- Client Sample ID;
- Sample matrix;
- Sample Date and Time;
- Time Zone
- MS/MSD (if requested on the COC)

9.8.23 Container types and container storage locations are assigned to each sample. Storage locations contain bins or labeled shelves to identify the exact location of each sample.

9.8.24 Tests are associated with the sample information previously completed as follows:

- Attach the specific project number which automatically stipulates the following:
  - Client address;
  - Client contact;
  - Purchase order number (if applicable);
  - The TestAmerica Seattle project manager;
  - The "requested" due date;
- All requested analyses for each sample.

9.8.25 TALS assigns a unique identifier to each sample as follows:

- Three digit laboratory code (this is 580 for Seattle);
- Five digit job number (as assigned in 10.10.1);
- Letter container identifier (alpha sequential, can be multiple letters);
- Single digit sample number (numeric sequential).
- Example: 580-12345-A-3

9.8.26 TALS then generates a label with the unique identifier and a bar code for each sample container of each sample received, which is placed upon the container. See example label in Attachment 1.

9.8.27 All containers are labeled with the appropriate TALS sample container label. This is reviewed by the technician labeling the containers. If needed for large or complex projects a verification check of the labels by a second technician to insure proper labeling should be completed.

9.8.28 For BP LaMP samples, an Internal Chain of Custody must be created. The ICOC assigns locations for each container that has been received. Samples are scanned in and out to track samples.

9.8.29 BP LaMP require the tracking of all bottle lot numbers. Scan the bottle and reagent lot numbers that are on the sample containers for all containers received for the project. If the container lot number is covered by the client label or the containers are not from out lab, document in a NCM. This information is recorded in TALs under the Lot Number column under the Container tab.

9.8.30 Place the samples into the designated bin in either walk-in refrigerator #1 (Wet Chem and extractable waters) walk-in refrigerator #2 (Waste and Soils) or the metals shelves (metals waters only) or (upon client request for complete custody samples) relinquish to the analyst. **For suspected highly contaminated samples, place the container into a Ziploc bag that is securely fastened before placing the sample into the refrigerator designated for contaminated samples.** All volatile samples are stored in the appropriate refrigerator in the volatiles laboratory (water samples are stored in refrigerators #31 and # 49; bulk soil samples, MeOH preserved containers and sodium bisulfate vials are placed in refrigerator #5; stir bar vials and voa vials with DI water are placed in freezer #554 (place voas with DI water on their side to avoid breakage); and any suspected highly contaminated samples for volatiles are placed into a Ziploc bag and stored in the refrigerators designated for contaminated samples - #17). The volatiles department is in its own building, Samples are placed in numerical order in the appropriate cold storage units. Waste samples are placed in order in the waste fridges, volatile water are placed in order in designated voa boxes, volatiles soils designated for the fridge or freezer are placed in a designated bin. All foreign soils are placed in the Foreign Soils Fridge #43. Foreign soils needing volatiles first will have a volatiles first sticker placed on the container lid and taken asap to the volatiles department for extraction. The job number and bin number are written on a sheet located on the outside of each door to help analysts locate samples. Metals waters are placed in bins and then placed on racks in the main lab. Samples requesting Grain Size are placed in the Grain Size fridge. When clients request soil/sediment samples to be freezer archived or submit tissue samples (clams, Geoducks, fish), those samples are stored in CSU # 7, 20, 19, 18, 9, 12, 24, or 25.

9.9 Workshare

9.9.1 The project manager must obtain approval to send workshare samples from the destination laboratory through the use of the established “CSO Sample Acceptance” and “CSO Pre-Arrival” NCMs.

9.9.2 A workshare COC from TALS must accompany samples from the location where they were logged to the location where the analysis will be performed.

9.9.3 The workshare COC must be reviewed by the sending lab in order to ensure samples are being sent to the correct location, and by the receiving lab in order to ensure samples have been received by the correct location.

9.9.4 A project/job method intended to be workshared to a TALS lab must be added by the PM from the destination laboratory’s method list; simply changing the destination of a local method is not a substitute.

9.9.5 If jobs are being cancelled in one TALS location in order for samples to be shipped to and logged at another laboratory, existing TALS container labels must be covered or removed in order to avoid confusion.

9.9.6 Sample receipt checklists must be filled out for all received workshare samples.

9.9.7 Only the received containers are to be scanned and transferred into the laboratory's custody via the Internal Chain of Custody (ICOC) module

9.10 Rush and short hold samples for Extractions must have a log-in summary report printed. After printing the report the short hold test and sample(s) should be highlighted. In addition the bin number(s) for the samples and type of rush/short hold must be written on the report. The report is taken to the Rush/Short-Hold notification board in the Extractions department and the department is notified of the addition to the Rush/Short-Hold board. The samples may be taken directly analyst in the Extractions department but should never be left in the department without being handed directly to an analyst.

9.11 The COC for the completed Job number login is then scanned into the job, The COC is placed in the days completed login folder, which is then placed into a bankers box located in Sample Control and when it is filled, is then transferred to archives. The login summary report, Chain-of-Custody (COC) form, and all other documents that pertain to the project are kept with the original COC and placed in the days completed login folder. Any special instructions are recorded in the method comments, sample comments, job comments or "sticky notes" in TALS.

9.12 The sample control technician will perform an initial review of the work order and will check the login release checkbox at the top of the login form in TALS. **If for any reason the sample control technician must add a method to a job for the lab Methods instead of the Analysis Groups from the project they must notify the PM via an NCM and add note in on the log-in and report review checklist.** The project manager (or designee) reviews the contents of the job, against the login info in TALS prior to distribution to ensure that all chain of custody, project, and/or client requirements are clearly communicated in TALS. The Project Manager completes review of the log-in and then checks the log-in review task in TALS to indicate that this review has been completed. The Project Manager checks the pricing review task in TALS to indicate that this review has been completed. (See Attachment 8 for more detailed instructions).

9.12.1 The laboratory only accepts samples outside of normal business hours when the client has discussed the issue with the project manager. The samples delivery/receipt is then addressed on a project to project as needed basis between the project manager and client.

9.13 If samples are received after regular business hours and login personnel are not available to complete the entire receiving and log-in process, the person accepting the samples must check the cooler in completely and then store in walk-in #1.

9.14 For **all samples** preserved in the laboratory, the preservation and lot number of the preservative must be recorded in the NCM that discussed the incorrect preservation found upon receipt. When adding preservative, do not add more than 10% of the volume of the bottle. Preserve samples as follows:

9.14.1 Nitric Acid Preservation (for samples collected for metals analysis).

The 1:1 nitric acid is stored in dropper bottles, in the Sample Receiving department. Containers are preserved using 15-20 drops of 1:1 nitric acid (for a large number of sample containers to be preserved, a 2 mL dispensing pipette may be used). **Mark on the sample bottle the date and time of preservation.**

9.14.2 Sulfuric Acid Preservation (for samples collected for TOC, Oil and Grease, TOX, or ammonia analyses).

The 1:1 sulfuric acid is stored in dropper bottles, in the Sample Receiving department. Containers are preserved using 15-20 drops of 1:1 sulfuric acid (for a large number of sample containers to be preserved, a 2 mL dispensing pipette may be used.

9.14.3 Hydrochloric Acid Preservation (for samples collected for volatile organic analysis and semivolatile analysis).

Voa vials are purchased from ESS, and contain 0.2 ml of 1:1 Hydrochloric acid. The vials are stored in the **volatiles laboratory**. Semivolatile ambers are also purchased from ESS and contain 2 ml of 1:1 HCl. 1:1 HCL dropper bottles are purchased from ESS and are stored in the Sample Receiving department. 15-19 drops roughly equals 1 ml of acid.

9.14.4 Sodium Hydroxide Preservation (for samples collected for cyanide analysis).

Plastic 500 ml polys are purchased from ESS containing 1 ml of 1:1 NaOH. Dropper bottles containing 50% NaOH are in Sample Receiving and are purchased from ESS. 15 drops roughly equals 1 ml of base.

9.14.5 Sodium Hydroxide/Zinc Acetate Preservation (for samples collected for sulfide analysis).

Plastic 500 ml poly with Sodium Hydroxide/Zinc Acetate are purchased from ESS. Each container has 6 mls Zinc Acetate and 6 pellets of NaOH. 1% Zinc Acetate dropper bottles are kept in Sample Receiving. 15-20 drops roughly equals 1 ml of acid. 15 drops of NaOH roughly equals 1 ml of base.

All non-volatile containers are stored in the back warehouse. A small supply of non-volatile containers is stored in the Sample Receiving area. All volatile containers are stored in the volatiles department.

9.15 Calibration

Digital thermometers are calibrated once per quarter as described in SOP TA-QA-0024 unless otherwise specified by the manufacturer. The IR gun is calibrated daily (SOP TA-QA-0024).

10.0 Pollution Control

It is TestAmerica's policy to evaluate each procedure and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

11.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Waste Disposal SOP TA-EHS-0036.

11.1 Waste Streams Produced by the Method

11.1.1 There is no special waste produced by this method.

12.0 References / Cross-References

12.1 Standard Methods for the Examination of Water and Wastewater, Online Edition, Method 1060.

12.2 USEPA Method 5035A "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002

12.3 Federal Register March 12, 2007, also known as the Methods Update Rule

12.4 State of Washington Department of Health Memorandum, Volatile Organic Chemical (VOC) Testing Program, September 25, 1995.

12.5 TestAmerica Seattle Quality Assurance Manual, Current Version, Holding Times, Preservation, and Container Requirements.

12.6 Attachments

Attachment 1: Example Sample Label
Attachment 2: Example Cooler Receipt Form
Attachment 3: Sample Acceptance Policy
Attachment 4: Example Short Holding Time Analyses Form
Attachment 5: Environmental Sampling Guide
Attachment 6: Recommended VOC Sample Preservation Techniques and Hold Times
Attachment 7: BP LaMP Preservation Options
Attachment 8: TALS Instructions
Attachment 9: Example Cooler Temp Label
Attachment 10: Work Instruction for Tracking Service Center Revenue
Attachment 11: Example Sample Receiving Triage and Labeling Checklist
Attachment 12: Logging in samples that have Sites and Events
Attachment 13: Non-conformance memos (NCMs)

13.0 Revision History

- Revision 28 dated 12/4/2018
  - Added requirement for NCM for missing custody seals (BP LaMP and BNSF), section 9.8.4.1.

- Revision 27.1 dated 6/5/2018
  - Small wording change for clarification in section 2.0

- Revision 27 dated 4/25/2017
  - Added the removal of packing material, section 9.5.2
  - Updated the location of the ice machine, section 9.6
  - Added check for leaking containers, section 9.8.6.3
  - Removed reference to log-in review checklist, section 9.8.19, 9.12 and Attachment 11
  - Added log-in release checkbox, section 9.12
  - Clarified the optional use of the Triage and Labeling Checklist, throughout
  - Updated Attachment 9

- Revision 26 dated 4/11/2016
  - Updated section 2.0 to more accurately reflect current practice
  - Added shipping supplies, section 6.3
  - Updated section 9.8.8 to add details about the container summary form.
  - Updated section 9.8.15 to current practice
  - Updated section 9.11 to current practice

- Revision 25 dated 11/7/2014
  - Updated section 2.0 to more accurately reflect current practice
  - Updated IR thermometer reference, section 6.1
  - Updated section 9.0 to add details about triage procedures and requirements for volatile

samples requiring freezing
- Added section 9.4 for added detail on taking and recording cooler/sample temperatures
- Added section 9.7 to detail the prioritization process
- Added section 9.8 to add details regarding cooler inventory and the log-in process
- Added section 9.9 to cover the requirements for workshare to other TALS labs in the network
- Added attachments 12 and 13, section 12.6
- Updated attachment 8 to current TALS Instructions

- Revision 24 dated 2/27/2013
  - Updated sections 2.0 and 10.1 to make signing and dating the COC on receipt a clear requirement and that if the date/time of receipt is noted on a separate paper that this must be retained with the original COC as the original record.
  - Updated section 2.0 to include the department managers as part of the “Triage” page process.
  - Updated section 10.1 to make a clear requirement and that if the temperature of a cooler is noted on a separate paper that this must be retained with the original COC as the original record.

- Revision 23 dated 2/27/2013
  - Updated Section 2.0: Summary of Method. Added DOD projects, Sample Control dwell times, scanning of COCs, and initial review of projects before handed to PMs. Added Short Hold board instructions for the Wet Chemistry Department and RUSH/Short Hold instructions for extractions department, no ph testing on volatile waters and O&G samples and COCs placed in the appropriate bin based on priority level. .
  - Added DOD projects and w or w/o custody seals to Section 10.0.
  - Added colored folders to Section 6.3
  - Added disposable pipettes to Section 6.1
  - Added project code to Section 4.2
  - Added DOD projects to Section 4.1
  - Added use a custody seal to seal a compromised sample and to write “Do Not Use” on seal to Section 10.5.1
  - Included more COC information needed in Section 10.7
  - Added preservatives, sample control can extract bulk samples for BP and to record ph strip lot # for DOD projects to section 10.8
  - Added the Sampler Receiving Supervisor for review to Section 10.10
  - Added instructions for BP and BNSF projects to Section 10.11
  - Added Sample Receiving responsible for writing the work order number on the foreign soils fridge door and how to label 2 oz jars to Section 10.13
  - Added metal tray to Section 10.14.1
  - Added VOA to Section 10.14.1.1
  - Added waste satellite container to Section 10.14.2
  - Added DoD to Section 10.14.3
  - Added date to Section 10.14.5
  - Added to login Method Blank for analysis to Section 10.14.6
  - Added ID of the thermometer used to monitor cooler temperature to Section 10.16.1
  - Changed four digit number to five digit number in Section 10.16.5
  - Added metals shelves and the description for the new procedure for storing samples in the Volatiles department to Section 10.16.9.
  - Added new colored folder system used in Sample Receiving to identify TAT, critical projects and initial review of projects performed by Sample Receiving Supervisor and COCs are

scanned and then placed in the master folder to Section 10.16.10.
  - Added the description of the initial review of projects and checklist completed by Sample Receiving Supervisor and Report Review Checklist completed by the Project Managers to Section 10.16.11 and the Sample Control Data Entry Checklist to Attachment 11.
  - Added to store samples in walk-in to Section 10.17.
  - Changed volume of HCl from 0.5mls to 0.2mls in Section 10.18.3

- Revision 22, dated 6 August 2012
  - Added section 10.5.1 to address the handling of compromised sample containers (ROMD .
  - Addressed pH as a short hold test in sections 10.1 and 10.10 (ROMD
  - Added new instructions on assessing cocs, erasable blue cards and instructions on where to leave folders to section 2.0
  - Added 1, 2, & 3 day for rush notification and notifying PMs immediately when samples are received out of temperature, compromised or broken to section 4.1.
  - Added diss. metals needing lab filter as a short hold and revised instructions in regards to TRIAGE and check-in to section 10.1.
  - Added to write an NCM when a temp blank is received frozen and to notify PMs when samples are received out of temperature to section 10.3.
  - Added Sample Cart as an option to use for unloading BP LaMP and BNSF samples to section 10.4.
  - Added notify PM immediately when samples are received compromised to section 10.5.1.
  - Added BNSF to section 10.8.
  - Added Dissolved Metals needing lab filtering and updated location of short hold binder to section 10.10.
  - Revised location of volatile foreign soils and instruction on how to handle them to section 10.12.
  - Added Residual Chlorine checked to section 10.15.2.
  - Added BNSF to several sections.
  - Added scanning of sample containers for BP LaMP and BNSF projects to section 10.15.8.
  - Updated new sample locations and where to store tissue samples to section 10.15.9
  - Removed master folder to be returned to sample receiving and added PMA duties to section 10.15.11
  - Added locations of where ESS containers are kept to section 10.17.5.
  - Added Service Center Revenue Tracking to information recorded in TALS, section 10.1.5
  - Updated TALS (TALS) instructions, Attachment 8.
  - Added Attachment 10

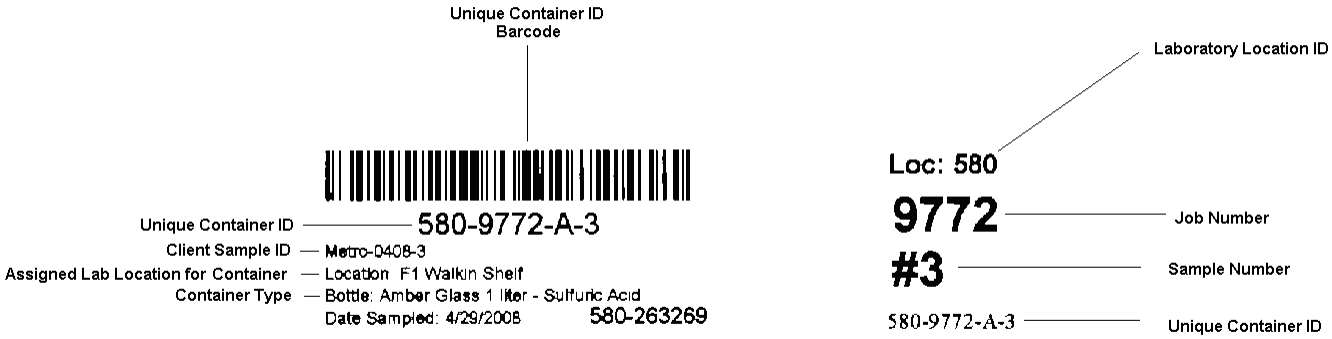
- Revision 21, dated 25 April 2011
  - Added steps for Triage, critical clients, additional Sample Control duties, pre-login of projects, placing coolers in walk-in with copy of COC and Project Manager Assistant to Section 2.0.
  - Added information in regards cooler assessment, informing lab of rushes, short holds, critical clients, subcontracts and workshares to Section 4.1
  - Added PM participation in Triage to Sections 4.2
  - Added ESS as our supplier to Section 6.3
  - Added additional sampling containers to Section 6.3
  - Added HNO3 1:1 and HNO3 1:3 (for shipping purposes) to section 7.1;
  - Added H2SO4 1:1 to Section 7.2
  - Added NaOH 50% to Section 7.4, CH2C00 to Section 7.5 and MeOH to Section 7.6
  - Added steps for Triage and Temperature Label to Section 10.1
  - Added use of thermometer, steps for samples received with no cooler, recording

corrected/uncorrected temperature, and temperature needed for all samples to Section 10.3.
  - Added steps for recording of temps for BP and procedure for maintaining temperature during unloading process for BP projects to Section 10.4
  - Added documentation of ph, ph adjustments on Login Container Summary form and steps to take for receiving bulk volatile soil samples to Section 10.8.
  - Added locations for foreign soils to Section 10.12
  - Added documentation of ph verification on Login Container Summary form to Section 10.13.1.1
  - Added Satellite Waste Container to Section 10.13.2
  - Added Login Container Summary form to Section 10.13.6
  - Added information regarding amount of acid to add for ph adjustments, container and preservative information and supplier of dropper bottles.
  - Updated the Environmental Sampling Guide.
  - Removed Special Handling Form (Attachment 5)

- Revision 20, dated 26 March 2010
  - Added procedures for handling rush TAT samples Sections. 2.0 and 9.6
  - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
  - Added removal of expired standards Section 7.11.
  - Added information on foreign soils Sections. 9.12 and 9.13
  - Added information on handling samples awaiting log-in. Section 9.15
  - Added sample acceptance criteria. Section 9.6 and Attachment 3
  - Added procedures for handling samples without a COC Section. 9.1.
  - Added procedure for handling high concentration samples. Section 9.16.7
  - Added description of how and when the IR gun will be used. Section 9.4
  - Added procedure for ensuring samples do not warm excessively during log-in. Section 9.5
  - Added procedure for BP bulk volatile containers to Section 9.9
  - Added procedure for BP sample pH adjustment and preservation options. Section 9.14.6 and Attachment 8
  - Added procedure for metals requiring preservation at receipt to Section 9.14.5
  - Added step 10.7 to verify tests indicated on the COC match the tests indicated on sample containers.
  - Updated section 10.8 to remove reference to the color flags used to notify wet chemistry staff regarding short holding time tests.
  - Updated Attachment 4: Short Holding Time Analyses Form
  - Updated Attachment 5: Special Handling Form.

- Revision 19, dated 19 March 2009
  - Added step 10.7 to verify tests indicated on the COC match the tests indicated on sample containers.
  - Updated section 10.8 to remove reference to the color flags used to notify wet chemistry staff regarding short holding time tests.
  - Updated Attachment 4: Short Holding Time Analyses Form
  - Updated Attachment 5: Special Handling Form.
- Revision 18 dated 31 March 2008
  - Integration for TestAmerica and STL operations.
  - This revision is a complete rewrite and an expansion of scope.
  - This SOP is the combination of SOPs 0001.17, 0006.12, and 0038.11.

Attachment 1. Example Sample Label



Attachment 2. Example Cooler Receipt Forms

TA Seattle

Cooler ID No. _____ TAL Work Order _____

COOLER RECEIPT FORM

Project _____

Cooler received on _____ and opened on _____ by _____

_____
(signature)

Temperature upon receipt:
Cooler: Corr_____°C, Uncorr_____°C Therm ID: _____
Temp. Blank: Corr_____°C, Uncorr _____°C Therm ID: _____

1.	Were custody seals on outside of cooler and intact? a. If yes, how many and where: _____ b. Were signature and date correct?	YES NO
2.	Were custody papers taped to lid inside cooler?	YES NO
3.	Were custody papers properly filled out(ink, signed, etc)?	YES NO
4.	Did you sign custody papers in the appropriate place?	YES NO
5.	Did you attach shipper's packing slip to this form?	YES NO
6.	What kind of packing material was used? _____	
7.	Was sufficient ice used?	YES NO
8.	Were all bottles sealed in separate plastic bags?	YES NO
9.	Did all bottles arrive in good condition (unbroken)?	YES NO
10.	Were all bottle labels complete (no., date, signed, pres, etc)?	YES NO
11.	Did all bottle labels and tags agree with custody papers?	YES NO
12.	Were correct bottles used for the test indicated?	YES NO
13.	If present, were voa vials checked for absence of airbubbles and noted if found?	YES NO
14.	Adequate volume of voa vials received per sample?	YES NO
15.	Was sufficient amount of sample sent in each bottle?	YES NO
16.	Were correct preservatives used?	YES NO
17.	Were extra labels added to pre-tared containers?	YES NO
18.	Corrective action taken, if necessary: a. Name of person contacted: _____ b. Date: _____	

12/10/10

Cooler Receipt Form

Fax this form and the CoC records to Jacobs Program/Project Chemist within 24 hours of receiving samples.

CoC Number _____(One receipt form per cooler)
Cooler Number/Name on CoC _____
Laboratory and Location _____
Lab SDG _____

1. Were Custody seals on outside of cooler?	Yes No
If yes, how many and where? _____	
Were signatures and dates correct?	Yes No
2. Were custody papers taped to lid inside of cooler?	Yes No
3. Were custody papers properly filled out (ink, signed, etc.)?	Yes No
4. Did you sign custody papers in the appropriate place?	Yes No
5. Did you attach shipper's packing slip to this form?	Yes No
6. What kind of packing material was used?_____	
7. Was sufficient ice used(if appropriate)?	Yes No
8. Were all bottles sealed in separate plastic bags?	Yes No
9. Did all bottles arrive in good condition?	Yes No
10.Were all bottles and labels complete (number, date, signed, analysis, pres., etc.)?	Yes No
11. Did all bottle labels and tags agree with custody papers?	Yes No
12. Were correct bottles used for the tests?	Yes No
13. Were VOA vials checked for absence of air bubbles, and if present, noted?	Yes No
14. Was sufficient amount of sample sent in each bottle?	Yes No
15. Chain-of-custody identification number:_____	
Temperature blank reading cor._____℃ un cor._____℃ Therm ID:_____	
Cooler temperature cor._____℃ uncor._____℃ Therm ID:_____	
16. Is temperature within 0-6℃?	Yes No
17. Were labels correctly associated with pre-tared containers? (not placed directly on jars?)	Yes No
CORRECTIVE ACTION FORM ATTACHED	Yes No

Jacobs Project Chemist contacted? Date/Time _____

Attach associated CoC record Conversation Confirmer forms.

Explain any discrepancies:_____
_____
_____

Attachment 3. TA Seattle Sample Acceptance Policy

Samples shipped to TA Seattle must meet the following conditions:

- The sample shall be completely documented on the chain of custody (sample identification, location, date and time of collection, preservation type, sample type/matrix, number of containers and transfer signatures);
- The sample shall be identified by a unique identifier using durable labels completed in indelible ink;
- The sample shall be collected in an appropriate container;
- The sample shall be collected in adequate volume for the analysis and necessary QC;
- The sample shall be preserved according to the requirements of the requested analytical method;
- The sample shall be delivered within at least one-half the holding time;
- The sample, if submitted for Volatile Organic analyses, shall be accompanied by a Trip Blank;
- The sample shall not contain asbestos or exceed allowed radioactivity levels; and
- The sample shall not show signs of contamination or breakage

A sample that contains asbestos, is radioactive, or shows signs of contamination or breakage shall be rejected outright at the time of receipt and will be returned to the client. Upon receipt of a sample that does not meet any of the other criteria stated above, the Project Manager shall request information from the client before proceeding. If the client can provide the information, the results obtained for the sample may be reported. Sample receipt discrepancies will be disclosed in the final report and results may be qualified.

Attachment 4. Example Short Holding Time Analyses Form

Test America Seattle/Tacoma

Short Holding Time Analyses

Analyte	Collection Date	Collection Time	Job Number	Sample Number(s)	Location	Login Initials	Date	Time	Analyst Initials	Date	Time

Notes:

Collection Date and **Time** are the date and time the field sample was collected.
Login Initials, Date, and **Time** are the initials of the sample receiving person completing the short holding time analyses form and the date and time the form was completed.
Analyst Initials, Date, and **Time** are the initials of the analyst acknowledging notification of the short holding time and the date and time the analyst completed the form.

Rev. 1
10/3009

Attachment 5. ENVIRONMENTAL SAMPLING GUIDE

The following guide can be used to assure the integrity of samples that are submitted for analysis. For each parameter the water method is listed first followed by the solid waste method. The water methods were updated based on the Federal Register dated March 12, 2007 also known as the Methods Update Rule, this update only covered waters so soils were not affected.

Parameter	EPA or Standard Method	Minimum Sample Volume	Container	Preservative	Holding Time
Alkalinity	SM 2320 B or 310.1	100 mls	250 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	14 days
Ammonia nitrogen	350.1	50 mls	125 ml HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2, Cool ≤6° C not frozen	28 days
	350.1M	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
	SM 4500-NH_3-B	500 mls	1 liter HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2, Cool ≤6° C not frozen	28 days
Bromide	300.0 or 9056	50 mls	125 ml HDPE	Cool ≤6° C not frozen	28 days
	300.0 or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
BOD	405.1 or SM 5210B	1 liter	1 liter HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	48 hours
COD	SM 5220-B or C or EPA 410.3 or 410.4	50 mls	125 ml HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2 Cool ≤6° C not frozen	28 days
Chloride	300.0 or 9056	50 mls	125 ml HDPE (acceptable P, FP, and G)	None required, Cool ≤6° C not frozen, suggested	28 days
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Chlorine, residual	330.5	200 mls	250 ml HDPE (acceptable P and F)	None required, Cool ≤6° C not frozen, suggested	immediate
Color	SM 2120 E	50 mls	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	48 hours
Conductivity	120.1 or SM2510B	100 mls	250 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	28 days

Parameter	EPA or Standard Method	Minimum Sample Volume	Container	Preservative	Holding Time
Cyanide (total & amenable)	335.4 and SM 4500-CN-E	500 mls	1 L HDPE (acceptable P, FP, and G)	NaOH pH 12 Cool ≤6° C not frozen (reducing agent as needed)	14 days
	9012	10 g	4 oz cwm	Cool ≤6° C not frozen	14 days
Fluoride	300.0 or 9056	50 mls	125 ml HDPE (acceptable P)	None required, Cool ≤6° C not frozen, suggested	28 days
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Flashpoint/Ignitability	1020A / 1010A	50 mls	500 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	NA
Hardness	130.2 or SM2340C	100 mls	125 ml HDPE (acceptable P, FP, and G)	HNO_3 to pH<2 Cool ≤6° C not frozen	6 months
Kjeldahl nitrogen	351.1	50 mls	125 ml HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2 Cool ≤6° C not frozen	28 days
	351.1M	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Kjeldahl nitrogen	351.3 (this method is no longer supported by MUR which is for waters) 351.2 is acceptable	500 mls	250 ml HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2 Cool ≤6° C not frozen	28 days
	351.3 (this method is no longer supported by MUR which is for waters)	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Nitrate nitrogen	300.0 or 9056	50 mls	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	48 hours
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	48 hours
Nitrite nitrogen	300.0 or 9056	50 mls	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	48 hours
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	48 hours
Nitrate + nitrite	353.2, 300.0 or 9056	50 mls	125 ml HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2 Cool ≤6° C not frozen	28 days

Parameter	EPA or Standard Method	Minimum Sample Volume	Container	Preservative	Holding Time
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Oil & Grease (HEM)	1664	1 liter	1 liter BR (only G is acceptable)	H_2SO_4 or HCl pH<2 Cool ≤6° C not frozen	28 days
	9071B	100 g	4 oz cwm	HCl pH<2 Cool ≤6° C not frozen	28 days
Orthophosphate (SRP)	365.1, 300.0 / 9056 / SM 4500-P E	50 mls	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	Filter within 15 minutes, analyzed within 48 hours
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	48 hours
Oxygen, Dissolved	4500-O	300 mls	300 ml BOD	Cool ≤6° C not frozen	Immediate
pH	150.1 / SM 4500-H^+ B / 9040	50 ml	125 ml HDPE (acceptable P, FP, and G)	None required, Cool ≤6° C not frozen, suggested	immediate
	9045	50 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Phenols	420.1	500 ml	1 liter BR (only G is acceptable	H_2SO_4 pH<2 Cool ≤6° C not frozen	28 days
	9065	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Phosphorus, total	365.1 / SM 4500-P E	50 mls	125 ml HDPE (acceptable P, FP, and G)	H_2SO_4 pH<2 Cool ≤6° C not frozen	28 days
	365.1M	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Solids, total	SM 2540 B	100 ml	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	7 days
	160.3M (this method is no longer supported by MUR which is for waters)	10 g	4 oz cwm	Cool ≤6° C not frozen	7 days
Solids, dissolved	160.1 or SM 2540 C	500 ml	1 liter HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	7 days
Solids, suspended	160.2 or SM 2540 D	500 ml	1 liter HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	7 days

Parameter	EPA or Standard Method	Minimum Sample Volume	Container	Preservative	Holding Time
Solids, volatile	160.4	500 ml	1 liter HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	7 days
Solids, settleable	160.5 or SM 2540 F	1 liter	1 liter HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	48 hours
Specific Conductance	120.1 or SM3510B	100 mls	250 ml HDPE	Cool ≤6° C not frozen	28 days
Sulfate	300.0 or 9056	50 mls	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	28 days
	300.0M or 9056	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Sulfide	376.1/376.2	500 mls	250 ml HDPE (acceptable P, FP, and G)	Zinc acetate and NaOH pH>9 Cool ≤6° C not frozen	7 days
	9030	100 g	4 oz cwm	Cool ≤6° C not frozen	7 days
Total Organic Carbon	415.1 / 9060 / SM 5310B	25 mls	125 ml HDPE (acceptable P, FP, and G)	H_2SO_4 or HCl or H_3PO_4 to pH<2 Cool ≤6° C not frozen	28 days
	9060	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
Turbidity	180.1 / SM2130B	100 mls	125 ml HDPE (acceptable P, FP, and G)	Cool ≤6° C not frozen	48 hours
METALS					
Hexavalent Chromium	7195	100 mls	250 ml HDPE (acceptable P, FP, and G)	PH to 9.3-9.7 and cool ≤6° C not frozen	24 hours after prep
Hexavalent Chromium	SM3500 Cr D or SM3500 Cr B	100 mls	250 ml HDPE	Cool ≤6° C not frozen	24 hours
Mercury	245.2/7470	100 mls	250 ml HDPE (acceptable P, FP, and G)	HNO_3 pH<2	28 days
	7471	10 g	4 oz cwm	Cool ≤6° C not frozen	28 days
All other metals (ICP/ICPMS)	200.7/200.8/ 6010/6020	100 mls	250 ml HDPE (acceptable P, FP, and G)	HNO_3 pH<2 or at least 24 hours prior to analysis	6 months
	6010/6020	10 g	4 oz cwm	Cool ≤6° C not frozen	6 months

Parameter	EPA or Standard Method	Minimum Sample Volume	Container	Preservative	Holding Time
ORGANICS (Semi VOA)					
Semivolatiles/PAH	625/8270	1000 mls	1 liter BR	Cool ≤6° C not frozen	7 days ext 40 days
	8270	30 g	8 oz cwm	Cool ≤6° C not frozen	14 days ext 40 days
Chlorinated Pesticides & PCBs	608/8081 or 8082	1000 mls	1 liter BR	Cool ≤6° C not frozen	7 days ext 40 days
	8081 or 8082	10 g	8 oz cwm	Cool ≤6° C not frozen	14 days ext 40 days
PCB Congeners	1668A / 1668C	1000 mls	1 liter BR	Cool ≤6o C not frozen	1 Year ext 1 Year
	1668A / 1668C	10 g	8 oz BR cwm	Cool ≤6o C not frozen	1 Year ext 1 Year
Organophosphorous Pesticides	8141M	1000 mls	1 liter BR	Cool ≤6° C not frozen	7 days ext 40 days
	8141M	10 g	8 oz cwm	Cool ≤6° C not frozen	14 days ext 40 days
Chlorinated Herbicides	8151M	1000 mls	1 liter BR	Cool ≤6° C not frozen	7 days ext 40 days
	8151M	10 g	8 oz cwm	Cool ≤6° C not frozen	14 days ext 40 days
EDB and DBCP	504.1 / 8011	3 (40) ml	3-40 ml vial w/Sodium Thiosulfate	Cool ≤6° C not frozen	7 days ext 40 days
UST PARAMETERS					
Hydrocarbon ID (HCID)	NWTPH-HCID	50 g	8 oz cwm	Cool ≤6° C not frozen	14 days
	NWTPH-HCID	1000 mls	1 liter BR	HCl pH<2; cool ≤6° C not frozen	14 days ext 40 days
Gasoline Range Organics	NWTPH-Gx / 8015 AK101/ VPH	(2) 40 ml	40 ml VOA	HCl pH<2 Cool ≤6° C not frozen	14 days (VPH 7 days if not preserved)
	NWTPH-Gx / 8015 / VPH/	10 g	1-40 ml vial vial w/ 10 ml MeOH	Cool ≤6° C not frozen	14 days

Parameter	EPA or Standard Method	Minimum Sample Volume	Container	Preservative	Holding Time
	AK101	5 g	4 oz cwm with Teflon-lined septum	25-mL surrogated MeOH Cool ≤6° C not frozen	28 days
Volatile Organic Compounds (Low Level-5035)	8260B / 8260C	10 g	2-40 ml voa vial s w/stir bars	Freeze within 48 hours of sampling	14 days
Volatile Organic Compounds (Mid Level-5035)	8260B / 8260C	10 g	1-40 ml voa vial w/ MeOH	Cool ≤6° C not frozen	14 days
Volatile Organic Compounds	624 / 8260B / 8260C	(2) 40 ml	40 ml VOA	HCl pH<2 Cool ≤6° C not frozen	14 days (7 days if not preserved)
Diesel Range Organics	NWTPH-Dx / AK102/103 / EPH	1000 mls	1 liter BR	HCl pH<2; cool ≤6° C not frozen	14 days ext 40 days
	NWTPH-Dx / 8015	1000 mls	1 liter BR	Cool ≤6° C not frozen	7 days ext 40 days
	NWTPH-Dx / 8015 AK102/103 / EPH	50 g	8 oz cwm	Cool ≤6° C not frozen	14 days ext 40 days

HDPE = High Density Polyethylene CWM = Clear Wide Mouth glass BR = Amber Boston Round glass

P - polyethylene

FP - fluoropolymer

G - Glass

PA is any plastic that is made of sterilizble material

LDPE - low density polyethylene

Attachment 6. Recommended VOC Sample Preservation Techniques and Holding Times

Sample Matrix	Preservative	Holding Time	Comments
Aqueous Samples with no Residual Chlorine	Cool to ≤6°C not frozen	7 days	If MTBE and other fuel oxygenate ethers are present do not acid preserve samples. If samples are suspected of biologically active compounds or aromatics, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples with no Residual Chlorine	Adjust pH to ≤2 with HCl or solid $NaHSO_4$.	14 days	Reactive compounds such as 2-chloroethylvinyl ether break down under acidic conditions. If these are analytes of interest collect a second set of samples without acid preservation.
Aqueous Samples with Residual Chlorine	Collect samples in prepreserved container containing either 25 mg ascorbic acid or 3 mg of $Na_2S_2O_3$ per 40 mL of chlorinated sample containing less than 5 mg/L residual chlorine.	7 days	If more than 5 mg/L of residual chlorine is present, additional dechlorinating agent may be required. Note that acidification of samples can interfere with some analytes.
Aqueous Samples with Residual Chlorine	Collect samples in prepreserved container containing either 25 mg ascorbic acid or 3 mg of $Na_2S_2O_3$ per 40 mL of chlorinated sample containing less than 5 mg/L residual chlorine. Cool to ≤6°C not frozen and adjust pH to ≤2 HCl or solid $NaHSO_4$.	14 days	If more than 5 mg/L of residual chlorine is present, additional dechlorinating agent may be required. Note that acidification of samples can interfere with some analytes CAUTION: Never add acid preserve directly to a dechlorinating agent prior to sample collection.
Solid Samples	Sample extruded into a empty sealed vial and frozen on-site to < -7°C.	14 days	Sample vials should not be frozen below -20°C due to potential vial seals problems and loss of constituents upon thawing.
Solid Samples	Sample extruded into a empty sealed vial and cooled to ≤6°C not frozen for no more than 48 hours and then frozen to < -7°C.	14 days	Analysis must be completed within 48 hours if samples are not frozen. (see above comments regarding freezing.)

Sample Matrix	Preservative	Holding Time	Comments
Solid Samples	Sample extruded into a empty sealed vial and cooled to ≤6°C not frozen for no more than 48 hours and then preserved with methanol.	14 days	Analysis must be completed within 48 hours if samples are not preserved with methanol
Solid Samples	Sample extruded into a empty sealed vial and cooled to ≤6°C not frozen.	48 hours	
Solid Samples	Cool to ≤6°C not frozen the coring tool used as a transport device.	48 hours	The hold time can be extended if the sample is either frozen to < 7°C or chemically preserved.
Solid Samples	Freeze to < -7°C the coring tool used as a transport device	48 hours	The hold time can be extended if the sample is either frozen to < 7°C or chemically preserved.
Solid Samples	Sample extruded into a sealed vial containing reagent water and frozen on site to < -7°C.	14 days	
Solid Samples	Sample extruded into a sealed vial containing reagent water cooled to ≤6°C not frozen for 48 hours or less and then frozen to < -7°C upon laboratory receipt.	14 days	
Solid Samples	Sample extruded into a sealed vial containing reagent water and 1 g $NaHSO_4$ and cooled to ≤6°C not frozen.	14 days	Reactive compounds readily break down under acidic conditions. If these are analytes of interest two sets of samples should be collected, one without the acid preservation.
Solid Samples	Sample extruded into a sealed vial containing methanol and cooled to ≤6°C not frozen.	14 days	

Volatiles are collected in 40-mL VOA vials that have septa seals.

If samples are to be frozen they should be placed horizontally.

Attachment 7. BP LaMP Preservation Options (for VOA Samples Shipped in Bulk Containers)

Laboratories receiving unpreserved solid samples in Method 5030/5030A sampling vessels must perform one of the following options within 48 hours from sample collection in order for a 14-day collection-to-analysis holding time to apply:

- Within 48 hours of collection, the laboratory may quickly transfer 5 grams of the solid sample into one or more 40-mL VOA vials containing a stir bar and freeze the sample.
- Within 48 hours of collection, the laboratory may quickly transfer 5 grams of the solid sample into one or more 40-mL VOA vial containing 10 mL of deionized water and a stir bar and freeze the sample.
- Within 48 hours of collection, the laboratory may quickly transfer 5 grams of the solid sample into one or more 40-mL VOA vial containing 10 mL of sodium bisulfate and a stir bar.
- Within 48 hours of collection, the laboratory may quickly extract the sample with methanol.
- The laboratory may quickly transfer 5 grams of the sample into a 40-mL VOA vial containing 10 mL of deionized water and a stir bar and analyze the sample within 48 hours from collection.

In the event samples are received beyond 48 hours of collection, the laboratory should request that the consultant project manager authorize (in writing) the preservation and analysis of the soil samples.

Attachment 8. Sample Login Procedure (on the Oasis website)

LOGIN

(For Project Managers, Project Manager Assistants, Client Service Managers, and Lab Managers)

Login

The Login application is divided into three different modules – Receipt, Login, and Job. Each of these modules performs a distinct function in the login process. The Receipt module allows the lab to receive samples, print sample container labels and note the condition of the samples and coolers upon receipt. If holding times or TAT are an issue, once the sample containers are labeled, the samples can be distributed to the lab without having to immediately complete the login process in TALS. The act of receiving the samples in TALS starts the login process.

In the Login module, sample information (client ids, sample matrix, sampling date/time) are entered for each of the received samples. A project that contains the client's requirements (created earlier by the project manager for that client) will be used to log the samples in. The project contains the method chains, project limits and reporting requirements for the samples. In the login module, methods are selected from the project in accordance with the chain of custody for each sample. By using the project as the client's template for login, the sample receiving group can log in jobs more efficiently. It is important to note that even though a job is logged in and samples may have been distributed to the lab for work, the data will not calculate until the login has been reviewed and approved by the project manager.

The last module in the Login application is the Job module. Each job is equivalent to a login when the login is first created. But a login can then be split into several jobs (each job with a separate set of reporting requirements) or several logins can be joined to create one job (a sample delivery group). Each job contains a separate set of deliverables, client contacts, pricing and turnaround times. The project manager for the project will review the login as a whole and the pricing and deliverables for each job separately.

In this User Guide, the user will learn how:

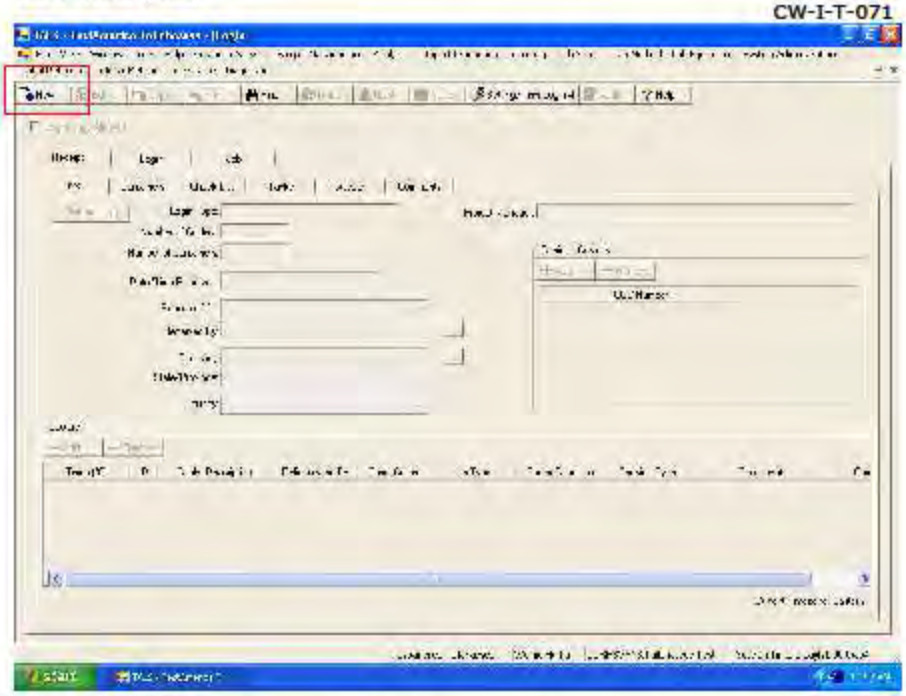
- To Receive Samples
- To Log-in Samples
- To Work with Jobs

To Start:

1. From the main menu, select *Sample Management* by clicking the application.
2. From the *Sample Management* menu, select *Login* by clicking the application.

To Receive Samples

1. Click the [**New**] button on the toolbar located at the top of the screen.

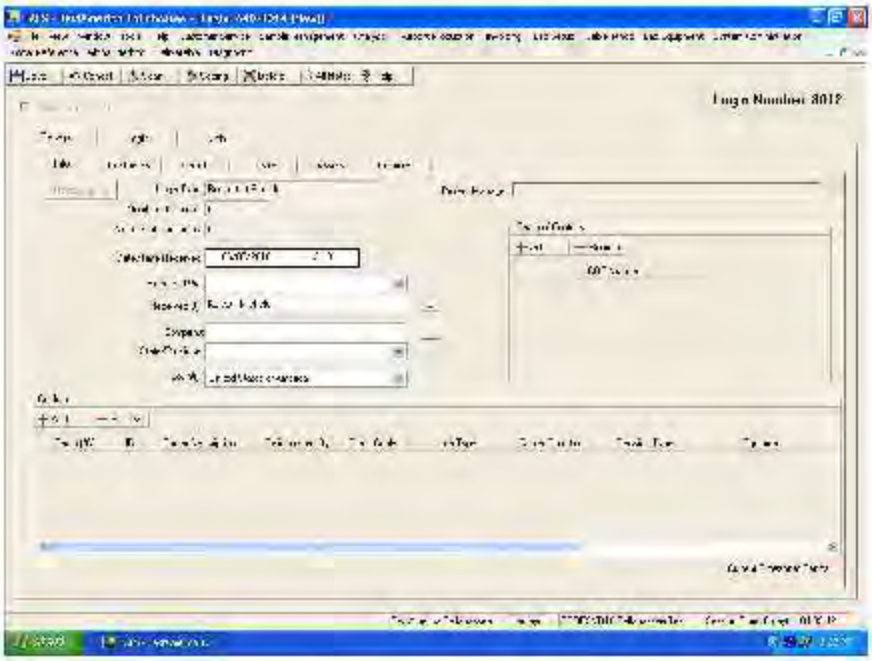


2. Click **[Yes]** to create a new Login.

Receipt - Info

When samples arrive at the laboratory, they must first be received in TALS. By keeping these modules separate, the sample receiving group can start the login, receive the samples and print the labels for the sample (s). Rush samples can then be moved directly into their respective areas to begin work before the samples are completely logged in. Until the sample login is complete, the samples will not relate to the login. All samples MUST be received before the login can be completed.

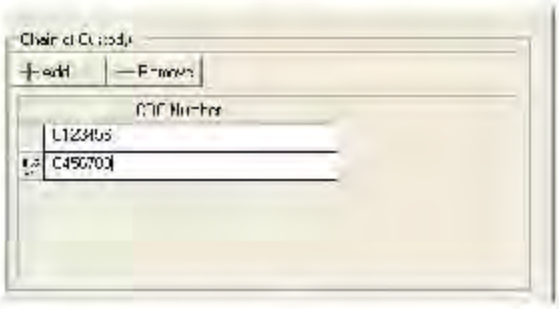
1. Grey shaded fields are non-editable. In each field below, enter the following:



- **Login Type** – defaults to Receipt of Samples when a new login is chosen.
- **Project Manager** – automatically fills in when the project number is selected on the ***LOGIN*** tab.
- **Number of Coolers** – automatically filled in with the information supplied under the 'Coolers' grid at the bottom of the screen.

- **Number of Containers** – automatically filled in with the information supplied in the ***CONTAINERS*** tab.
- **Date/Time Received** – defaults to the time the login was created. To change the date/time to the actual receipt date/time noted on the COC, use the drop down/up arrows in each field.
- **Received Via** – mode of delivery of the samples (courier, client pickup, etc) selectable from dropdown menu.
- **Received By** – the individual that received the samples and signed the chain of custody. Defaults to the current user logged onto TALS. Change by clicking [...].
- **Company** – the client that sent the samples. This is an optional field. Select a client by clicking [...].
  - *Note: If a company is chosen in this field, the **PROJECT LOOKUP** on the **LOGIN** tab will automatically be filtered to display active projects from only that company.*
- **State/Province** - enter the State where samples were collected. This is a required field. Select from the dropdown menu.
- **Country** – enter the country where samples were collected. This is a required field. Select from the dropdown menu. The default is United States of America.

2. Click [Add] to enter the Client's Chain of Custody numbers.
   - Note*: Entering one (1) chain if custody will automatically add the COC number to all containers. If more than one COC is entered, a COC must be selected manually for each container. This may be done on the **CONTAINERS** tab.*



3. Click [Add] to enter the cooler information.
   - *Note: Entering one (1) cooler will automatically add the cooler number to all containers. If more than one cooler is entered, a cooler must be selected manually for each container. This may be done on the **CONTAINERS** tab.*

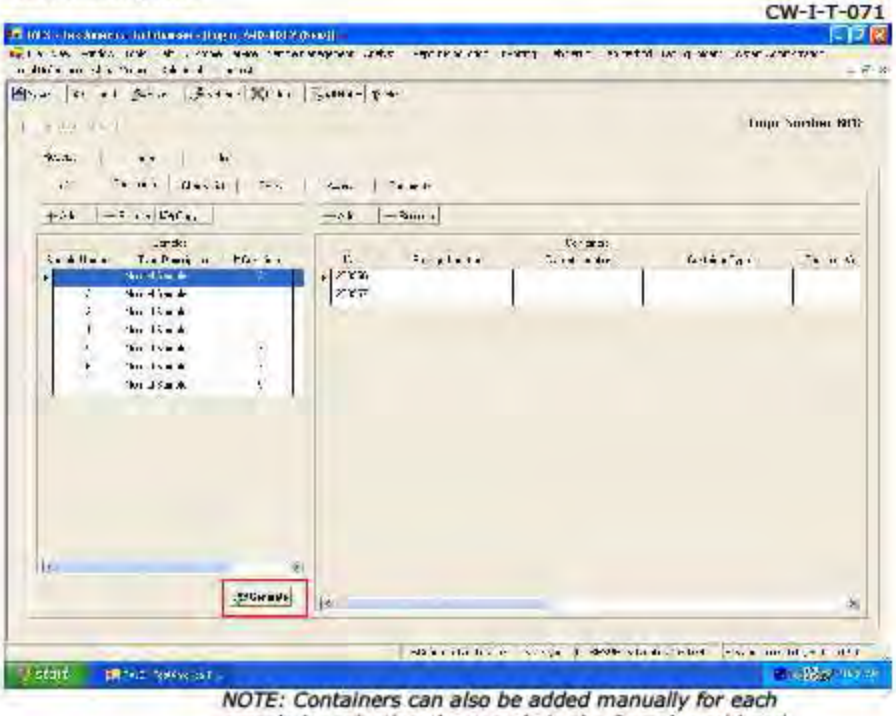
In the Cooler grid enter the following:

- **Temp (C)** - the internal temperature of the cooler upon receipt.
- **ID** – incremental number assigned to each cooler.
- **Cooler Description** –a description of the physical cooler, whether it is a thermal cooler, insulated box or other container.
- **Relinquished By** –the individual that signed the Chain of Custody and relinquished the custody of the samples to the laboratory sample custodian.
- **Client Cooler** – Check this box if this is a client provided cooler.
- **Ice Type** – type of ice that the samples were packed in (drop down list).
- **Cooler Condition** – the physical condition of the cooler upon arrival.
- **Packing Type** – the type of cushioning material used to keep the samples from breaking in transport (drop down list).
- **Equipment** – any equipment (sampling equipment, balances, palm pilots, etc) packaged with the samples.
- **Comments** – free form text field to enter any additional information about the cooler.

Receipt - Containers

1. Click [Add] to enter the total number of samples received in the ***SAMPLES*** tab of the Login.
2. Enter the total number of containers received per sample in the space provided in the **#Containers** field.

3. Click **[Generate]** to create the container records in the CONTAINERS section of the Login automatically.



NOTE: Containers can also be added manually for each sample by selecting the sample in the Samples grid and then clicking the [Add] button on the Containers grid. The box below will be displayed to enter the number of containers desired for the sample.

4. After generating the CONTAINER records, fill in the following information in each of the fields in the container grid (fields are represented from left to right – scroll to the right to see the remaining fields)
   - **ID** – unique container ID assigned by TALS – no entry is allowed in this field.
   - **Storage Location** – storage locations are set up in the system defaults and populate the drop down list used in this field.
   - **Current Location** – where the container is currently being stored.
   - **Container Type** – use the drop-down arrow to select from a

valid values list of containers.

- **Container Volume** – auto-fill based on the selection in Container Type.
- **Container Units** – will auto-fill based on the selection in Container Type
- **Condition** – condition of container when received. Enter only exceptions to a condition of good.
- **COC** – use the drop down arrow to select available Chain of Custodies. If no COCs were entered on the ***RECEIPT INFO*** tab, this field will be blank. If there is only one COC, the field will be auto-filled.
- **Cooler** – select from the drop down list if more than one cooler was received or the field will be auto-filled if there is only one cooler. If no coolers were entered on the ***RECEIPT INFO*** tab, this field will be blank.
- **Assets** – pre-defined valid values in the *Assets* module.
- **Lab ID** – combination of the login location, the login number, the alpha occurrence of the container for that sample (A = 1, B = 2, etc.) and the sample number itself. The ID and Lab ID both indicate the same container and can be used interchangeably. No entry is allowed in this field.
- **TAG** – CLP sample containers may arrive with a tag. Enter the tag ID here.

Some shortcuts are available:

A) *Simultaneously pressing the **<CTRL>** key and the **<DOWN>** arrow will automatically fill in the field below the field the cursor is on (as long as the new field is blank).*

B) *Some fields allow filling in every row of a column automatically. Simply highlight the HEADER of a column (Storage Location for example). This turns the entire column BLUE. Right-Click in the blue and select a value from the valid values list. All fields in this column will now have the selected value.*

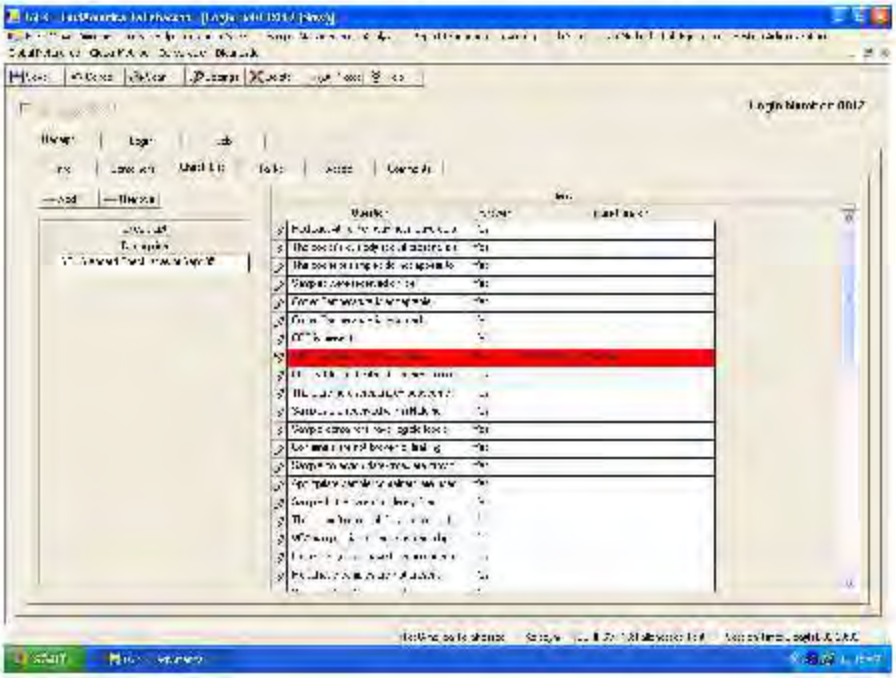
C) *Use the **[COPY]** button in the SAMPLES section. Simply fill out all of the information in the CONTAINERS section of the form for one sample. Click **[COPY]**. Choose the completed sample on the left-side of the form ("copy from") by clicking the left-most grey square (causing that row to be completely highlighted) and then choose the samples to "copy to" in the right-side of the form by clicking the left-most grey square of the row (causing that row to be completely highlighted). For more samples, simply hold the mouse button down when selecting the first sample and "drag" the highlight down the list of samples until all of the samples to copy are highlighted. Click [OK] and the container information will be copied to the new records.* **Note***: The copy function only works if the samples have the same number of containers.*

TALS Training Brief

12/28/10
CW-I-T-071

Receipt – Checklist

The Sample Receipt Checklist defines the condition of the samples when received.



1. Answer all questions.
2. A Failure Reason must be entered for all negative responses
3. If the question does not apply to this set of samples (i.e. a question pertaining to solid samples for a shipment of only waters) N/A may be used as a response. All 'No' answers will be highlighted in red.
4. To quickly enter the most common answer for all questions, highlight the HEADER of the ANSWER COLUMN. This will turn the entire column BLUE. Right-Click in the blue highlight and select an answer (Yes, No, N/A). All fields in this column will now have the selected response. Edit the answers that do not match the overall answer chosen.

Receipt – Tasks

Tasks will be covered later in this document.

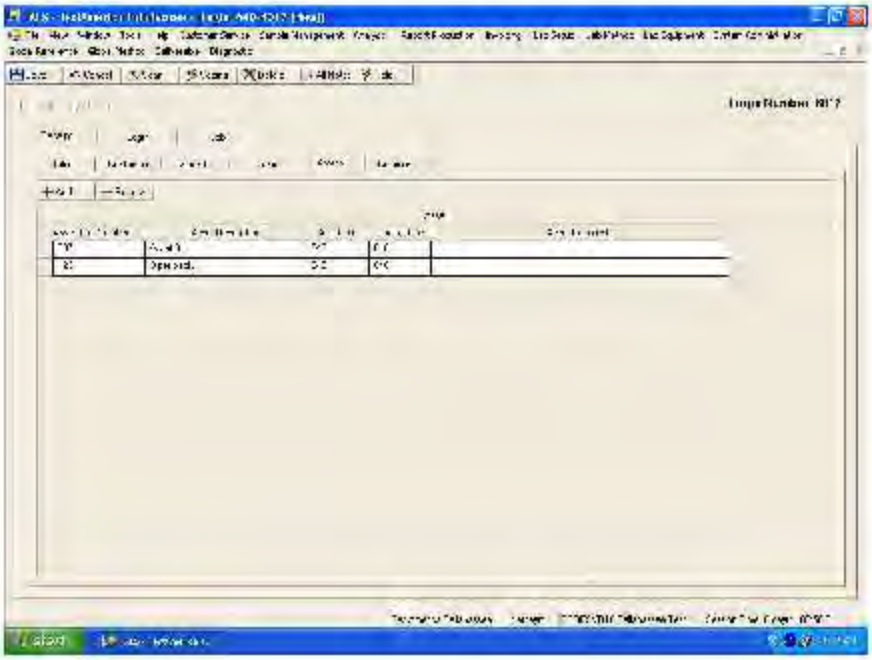
TALS Training Brief 12/28/10
CW-I-T-071

Receipt - Assets

The *Assets* tab is used to log company assets returned with a sample shipment. Assets are defined as anything a laboratory or service center will send out with a bottle order and expects to be returned to the lab. Tracking assets is optional but it is strongly suggested to track assets for any items that are either frequently never returned or exceedingly costly. The individual facility should use their judgment as to what items should be tracked as assets.

The basic premise for an asset is when an asset is included in a shipping order its asset number is stored with the shipping order. When shipped the item is marked as being out. Being associated with a shipping order, it can be determined when it was sent out and to whom. At the login receipt step, upon return to a facility, the asset can be signed back in whereby it will be marked as returned. The use of bar code scanners can be employed to speed the signing in and out of an asset.

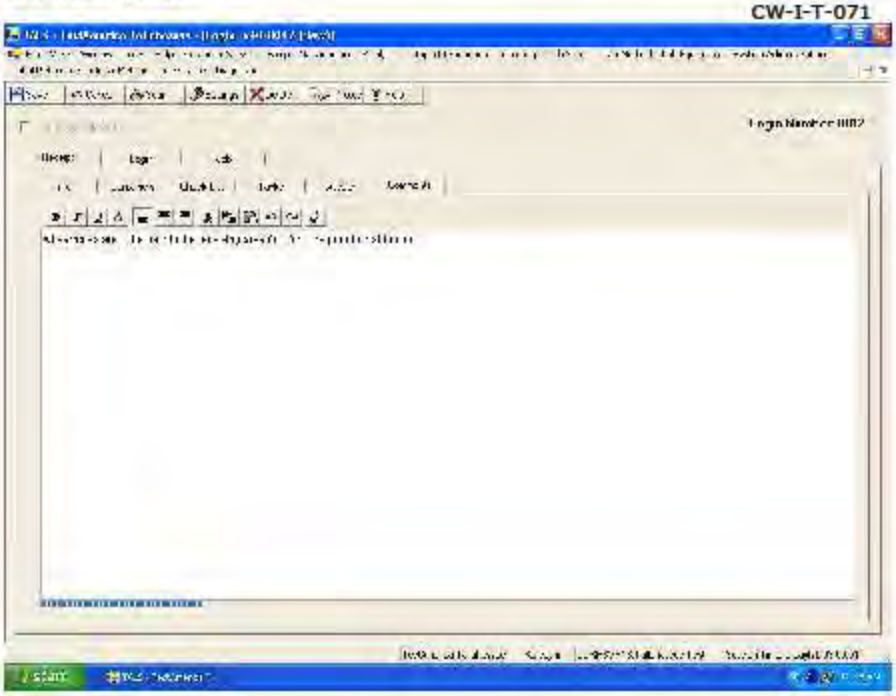
Maintenance of a location's assets is found under *Sample Management*. See the '*Assets*' TALS training brief on the intranet for information on maintaining assets.



Receipt – Comments

Free-form area to enter any comments, notes, etc. These can appear on backlogs if desired.

TALS Training Brief 12/28/10
CW-I-T-071



Save

Click **[Save]** from the menu across the top of the screen to save the receipt information entered to this point.

To Log-in Samples

1. Locate the Login by either clicking on the **[Find]** button (which will bring up a list of all the logins for this location or that have work for this location) or the [Login #] button where the login number can be entered directly. Either choice will load the login.
2. In the ***LOGIN*** tab, click the **[Add]** button next to the PROJECT NUMBER field. If a COMPANY was chosen on the ***RECEIPT*** tab, the list of projects will be filtered based on that company. Otherwise a complete list [of Active Projects] will be displayed. Filter the list based on the pop-up screen and select the appropriate project. Click **[OK].**

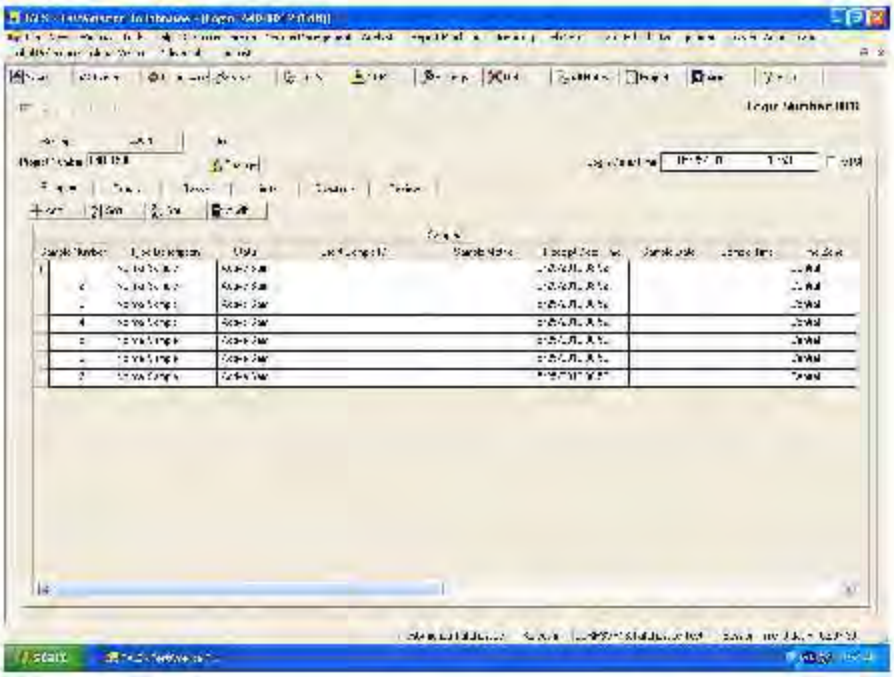
Note: If the wrong PROJECT is selected, click on the [Change] button and follow the instructions.

*Note: See **Sites and Events** documentation on the intranet for instructions for logging in samples that have Sites and Events assigned to the project.*

Page 12 of 31

TALS Training Brief 12/28/10
CW-I-T-071

Login – Samples



1. Enter the information for each Client Sample. Some fields will be auto-filled based on information from the previous tabs and the Project.

Update the following information as needed.

- **Sample Number**– assigned by the system as each sample is added. If samples are removed from the login, the sample number does not renumber.
- **Type Description** – default type will be normal sample (client sample). See how to add **Client Requested QC Samples** later in this user guide.
- **Status** – default is Active Sample. Select Hold or Inactive Sample from the dropdown list if applicable.
- **Client Sample ID** - Enter the Client Sample ID accurately, as it was entered on the COC. If the Project has "Sites and/or Events" associated with it, and the PM elected to verify against the site samples list, this field will NOT be free-form, but will be a drop-down list to select the appropriate Client Sample ID. See the *Sites and Events* documentation on the intranet for additional information on this feature.
- **Sample Matrix** - select the Sample Matrix from the drop-down list. This is the sample matrix that the lab will see on their backlogs and in Analyst Desktop. This is the matrix that is used in the reference data as well. If the client needs the matrix to be displayed on the report and EDD as something more specific such as 'ground water', use the alternate sample matrix field.

Page 13 of 31

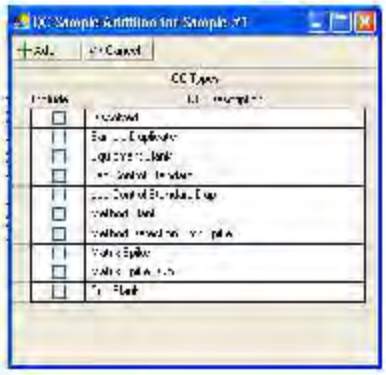
- **Receipt Date Time** – Date and Time samples were received at the laboratory. This field is copied from the date and time entered on the receipt tab.
- **Sample Date** – enter the sample date for each sample as it is entered on the COC. Click the down arrow to bring up a calendar to select the date.
- **Sample Time** – enter the sample time as entered on the COC. To enter the time in military time (24 hour clock), change the format by clicking on the [Settings] button at the top of the screen, then select 24 hour.
- **Time Zone** – change the time zone to the time zone where the samples were taken.
- **Hazard Level** – For laboratories with a Hazard Level Permit, enter the hazard level of the samples as received. The default will be Unconfirmed.
- **Alternate Sample Matrix** – as mentioned above, the client may require that a more descriptive sample matrix appear on their deliverables. Alternate Sample Matrices are added in the Site Sample module and are available as a drop down in the Login module.
- **Sample Types** – select the sample type from the drop-down list. (i.e. 'Field Blank, Trip Blank')
- **Action Limit Set** – If ONE Action Limit Set has been associated with this PROJECT, the list name will automatically fill in. If more than one exists, the appropriate list must be chosen from the drop-down choices. See Action Limit Sets documentation posted on the intranet for more information on this item.
- **Sample Comments** – enter any additional information about the sample that may need to be conveyed to the analytical group.
- **Login User** – copied from the **Receipt** tab.

Client Requested QC Samples

If sample QC is received from the client and it is necessary to create this QC at login (MS/MSD/Duplicates, etc.) follow this procedure:

1. Select the sample by clicking on the sample Number or Type Description field.
2. Right Click to pull up the **Sample Options** menu and select **Add QC Samples.**

3. Choose the appropriate QC types from the popup box which will appear and click [**Add**].



4. Populate the appropriate information for the QC samples
5. Click back to the **RECIPT TAB – CONTAINERS SUBTAB** to add the appropriate information for the containers received specifically for the QC.

Login – Groups

The analyses associated with each sample are contained in LOGIN GROUPS. Sample Receiving will build these groups from the information contained in the PROJECT.

The groups can be created a variety of ways, ranging from easy to complex. The most straight-forward way is to organize your groups in relationship to the samples and the analyses requested on each sample.

For example:

If sample #1 and #3 are both waters and are requesting GCMS VOA8260 Standard List and Lead ... create a LOGIN GROUP with these two parameters and associate this group to these samples.

If sample #2 is only getting Lead ... create another LOGIN GROUP that only contains Lead and associate this group to this sample.

In this example, you would have two login groups (with the correct analyses on each sample).

To CREATE A NEW LOGIN GROUP:

1. Click on the **[NEW]** Button.
2. From the pop-up menu choose from the following:

A. **[Analysis Groups]**

This shows all of the methods/groups the project manager has created in the PROJECT.

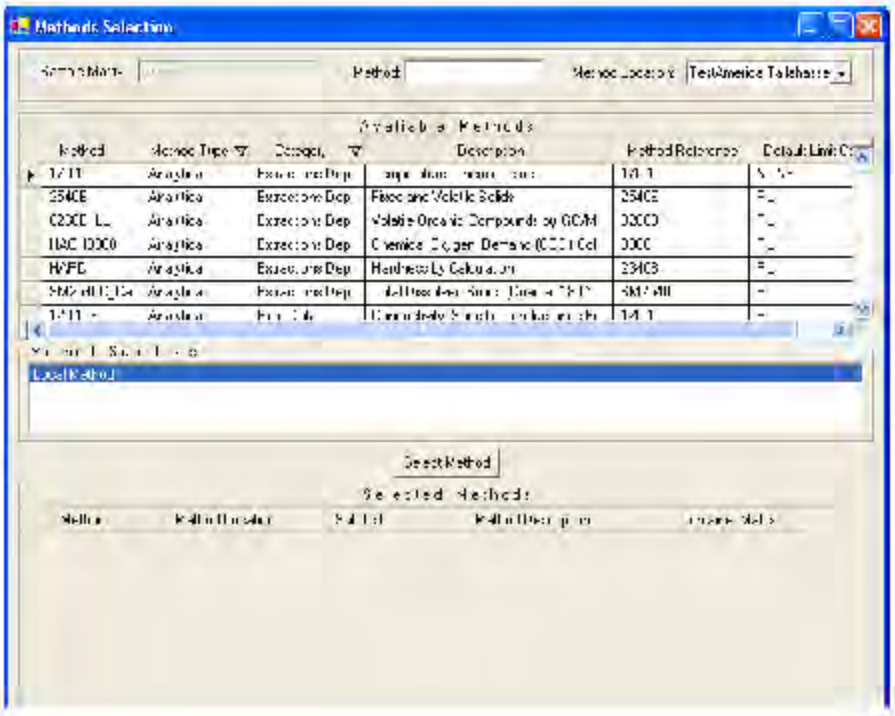
1. Paying attention to the Matrix and the Group Description, highlight the group that contains the methods of interest.
2. Once chosen, the associated METHODS will appear in the lower grid of this pop up.
3. Highlight the method(s) of choice and click on [Select Method]. A LOGIN GROUP with the chosen Methods and their corresponding preps is created.

TALS Training Brief 12/28/10
CW-I-T-071

A. [METHODS]

This shows all of the methods associated with the current lab location.

1. All available methods are shown.
2. Once a group is chosen, the associated METHODS will appear in the lower grid of this pop up.



A. Each LOGIN GROUP created may be viewed by highlighting the LOGIN GROUP in the "Login Groups" grid by clicking the left-most grey square and highlighting the entire row.
B. Once highlighted, the corresponding methods/preps will be displayed in the grid to the right.
C. The grid will be titled with the LOGIN GROUP NAME (which automatically comes from the PROJECT, but can be renamed by simply by typing over the title in the LOGIN GROUPS grid).
D. Columns in the LOGIN GROUP [DETAIL] GRID:
   - **Method Code** – the internal ID of the method
   - **Method Location** - the originating location of the method
   - **Method Description** – detailed description of the method
   - **Alt Sub-Desc** – this field is customized in the PROJECT to indicate to the lab if the list has been modified from its original state.
   - **Destination** – if method is being sent to another facility to be analyzed, this field will have the information of that location, otherwise, it will be your current location.
   - **TAT** – the Turnaround Time of the Method
   - **Condition** – the current condition of the method (Active, Cancelled, On Hold)
   - **Status** – the current status of the method (Ready, Batched, 1st Level Reviewed, etc.)
   - **Phase** – currently not used.
   - **Sub-Desc** – the default description of the sub-list chosen at creation.
   - **Holding Time Calculation** – method used to calculate holding time
   - **VTSR** – if checked, the holding time will be calculated off of the RECEIPT DATE and follow the holding time setup for this occurrence.
   - **Up Rpt Limit Type/Low Rpt Limit Type** – these fields work together to advise how the method will be reported. The "Upper Limit" is the value at which the sample will be reported (usually the RL), while the "Lower Limit" is the [lowest] value at which the system will report a value (usually the MDL).
   - **Container Matrix** – the matrix of the sample expected for this method
   - **Reporting Basis** – the basis of reporting this method such as a "Dissolved" Metal versus a "Total" Metal".
   - **Calibration Group** – currently not used
   - **Calibration Curve** – currently not used
   - **Dry Wt Adjust** – check if dry weight correction is needed for the method
   - **# Tics** – the max number of TICs to be reported. If TICs are required, the analyte Tentatively Identified Compound must be set to reportable. Do not enter TICs for logged in QC samples.
   - **Method Comments** – free flow comments
   - **Reporting Rules** – currently used for dual column analyses to indicate which rule to follow when reporting both columns.
   - **SAP** - Secondary Accounts Payable. The SAP field is a 10-character field available at the method and other charges level to support client required billing codes in Invoicing. The SAP field may be controlled by a Login Group, or through the pricing and Other Charges tabs of the Job in the Login Application. SAP information applied in the project will automatically populate in the login/job and may be modified accordingly.
   - **Do Not Report** – currently used to inhibit a method from printing on the final report, although the method is needed in the login for additional

analyses (such as reporting the calculated result of HARDNESS, but not wanting to report the individual analyses required to achieve the hardness calculation)

- **Client Sub Description** – Client Sub List Description settings will copy down from project/quote module. Settings can be modified at the login level. See the *Client Sub List Description* documentation on the intranet for more information.

Note: *A "grey background" in the METHODS/PREP window indicates this login group is not associated to the sample highlighted in the "SAMPLE GROUPS" grid. A "white background" indicates this group is associated to the sample highlighted. Additionally, the LOGIN GROUP in the "Sample Groups" grid will turn blue to indicate which LOGIN GROUP has the focus.*

Once the LOGIN GROUP(S) has been created, associate the LOGIN GROUP to the correct sample(s).

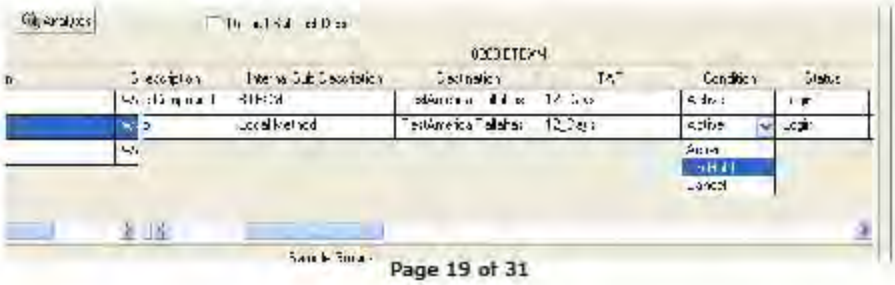
A. Simply put a check-mark in the square corresponding to the correct sample and the correct login group.

B. A shortcut is available here as well. Simply highlight the HEADER of the LOGIN GROUP in the "Sample Group" grid (shown by the "1" and "2") by right-clicking and hit Select.

C. To **CANCEL** a method (or to place a method **ON HOLD**), highlight the appropriate SAMPLE in the sample grid, and the appropriate LOGIN Group.

D. Change the CONDITION of the method from ACTIVE to either CANCEL or ON HOLD as needed.

- Changing the CONDITION of a prep method will automatically update the condition of the analytical method.



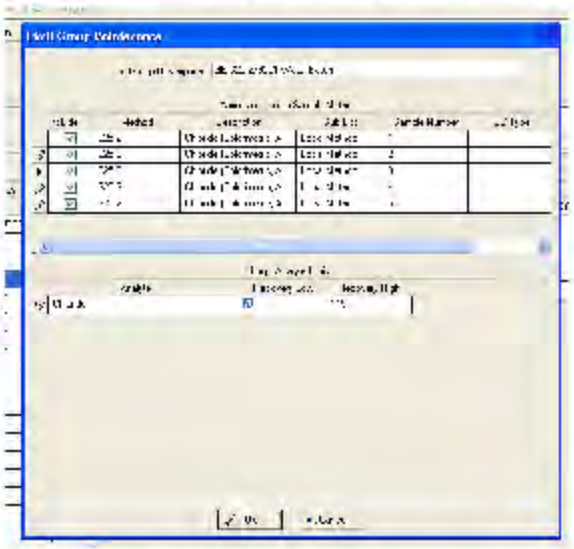
Login – Limits

This tab will be reviewed by the Project Manager to assure all of the appropriate limits have been loaded into the LOGIN.

In general the limits pulled into the Login Limits are from the reference data built in the Method Limit Groups. Reference limits can be overridden if Project specific limits are defined in the Project. See *Projects* documentation posted on the intranet for more information.

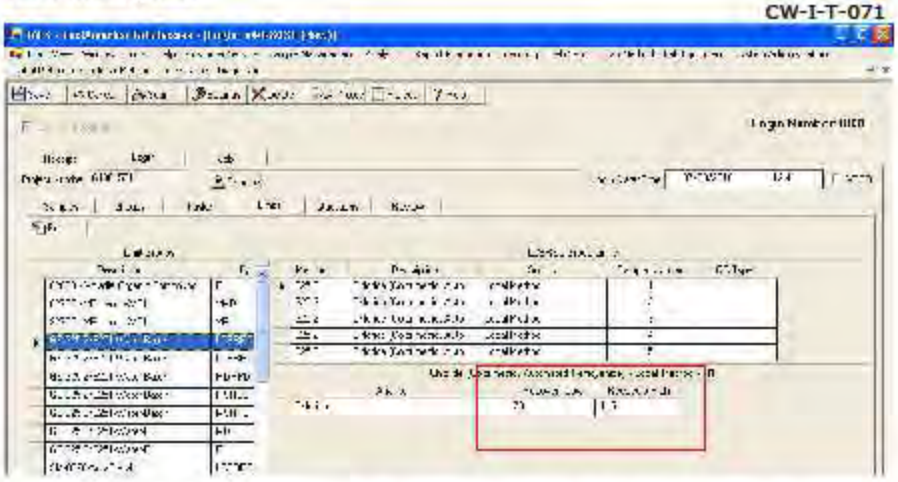
Limits can also be overridden at the Login level.

A. On the LOGIN LIMITS tab, highlight the limit to be changed and click [Edit].
B. Click [Yes] when asked to create a new limit group.
C. In the 'Limit Maintenance' pop up, select the samples the limit will be applied to (the default will have all samples checked to be included) and change the limit set in the lower grid. Click [OK].

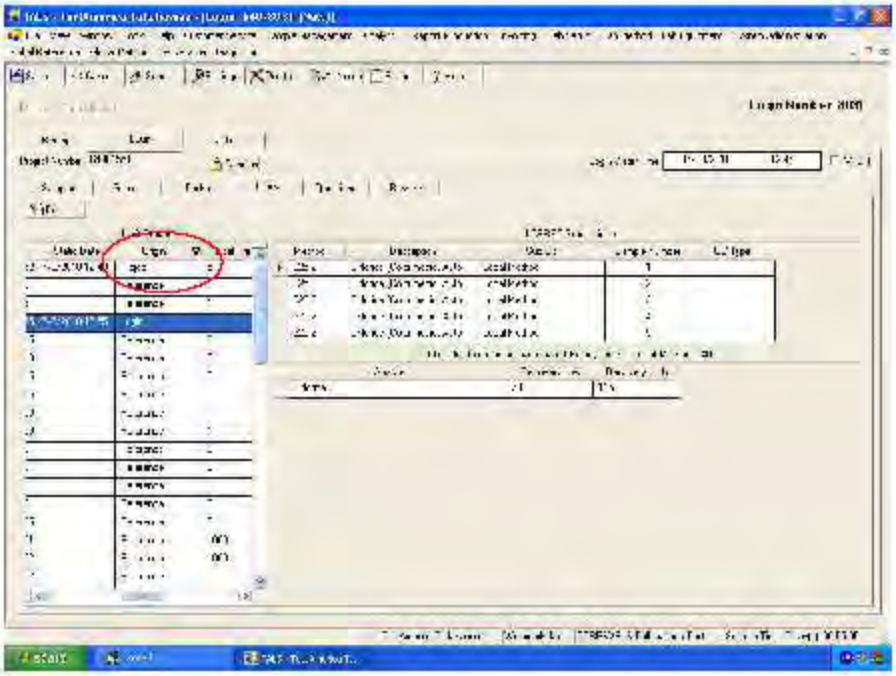


D. The new limit will be entered into the login.

TALS Training Brief 12/28/10
CW-I-T-071



To tell which limits are applied to a limit type, scroll to the right in the Limit Groups grid to the **Origin** column.



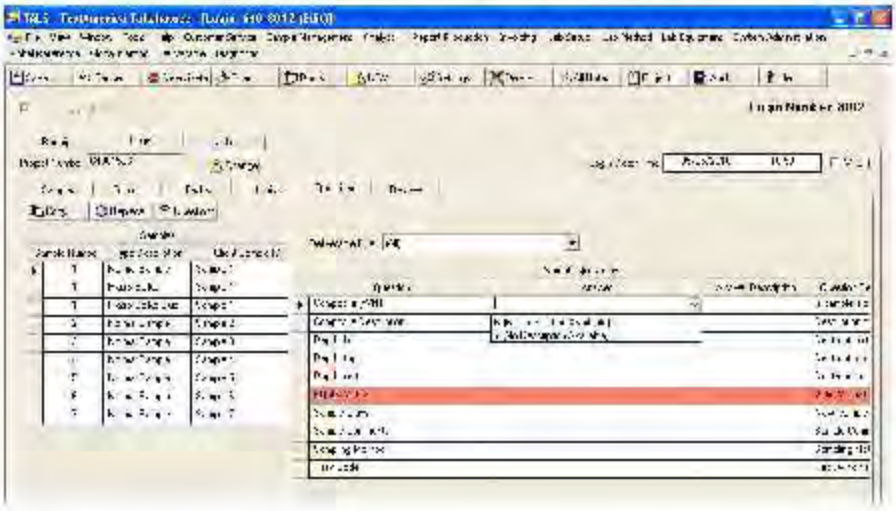
Login – Questions

This tab may have information that needs to be completed. If a client needs an EDD which requires information that is not otherwise available in TALS, **Questions** are used to accomplish this. Simply answer the question to store the answer. Login Questions are answered on a sample level basis. Questions are programmed into the EDD's and will pull into the login when an EDD that requires them is added.

Example:

Question: **What is the sample depth listed on the Chain of Custody?**
Answer: **4.5**

Note: *The answer to a particular question may be answered by the appropriate group, depending on the question. Just because this tab is in the LOGIN Module doesn't imply that the answer will come from Sample Receiving.*

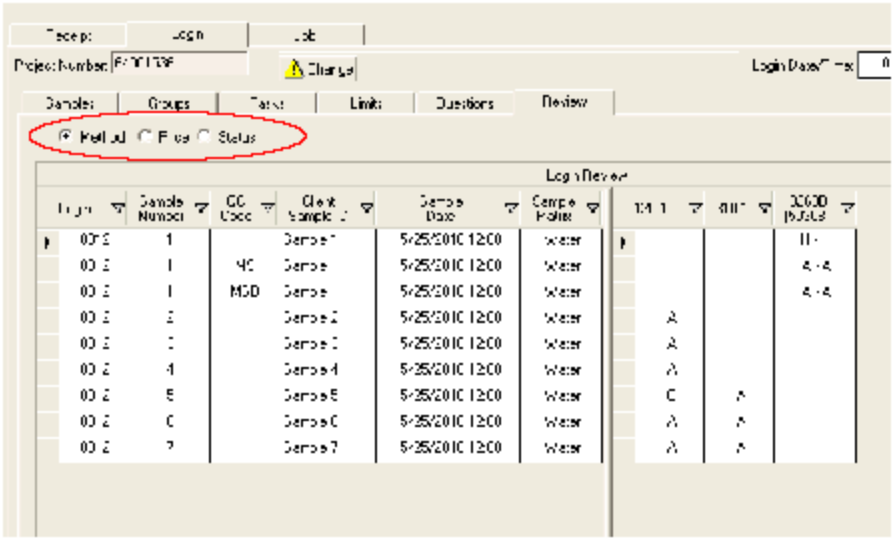


Login – Review

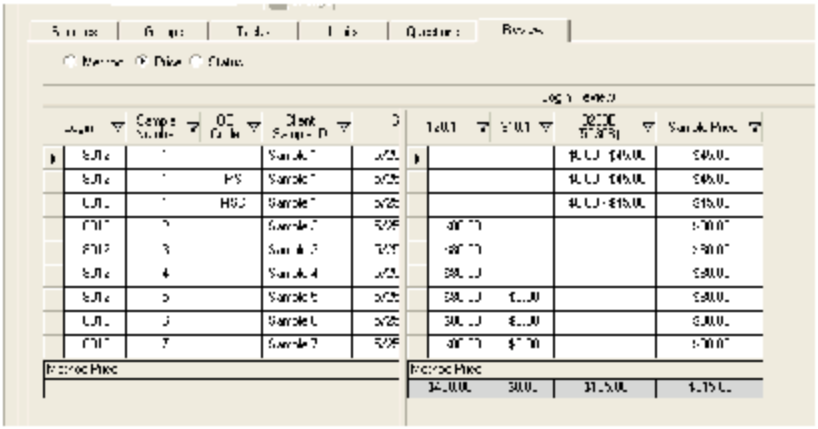
This tab shows methods, pricing and current statuses at a method chain level. Select the radio dials to toggle the views.

TALS Training Brief **12/28/10**
CW-I-T-071

METHOD

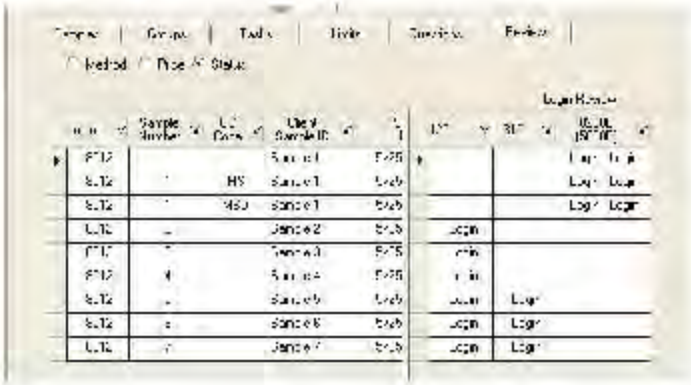


PRICE



TALS Training Brief 12/28/10
CW-I-T-071

STATUS



To Work with Jobs

Job – Job

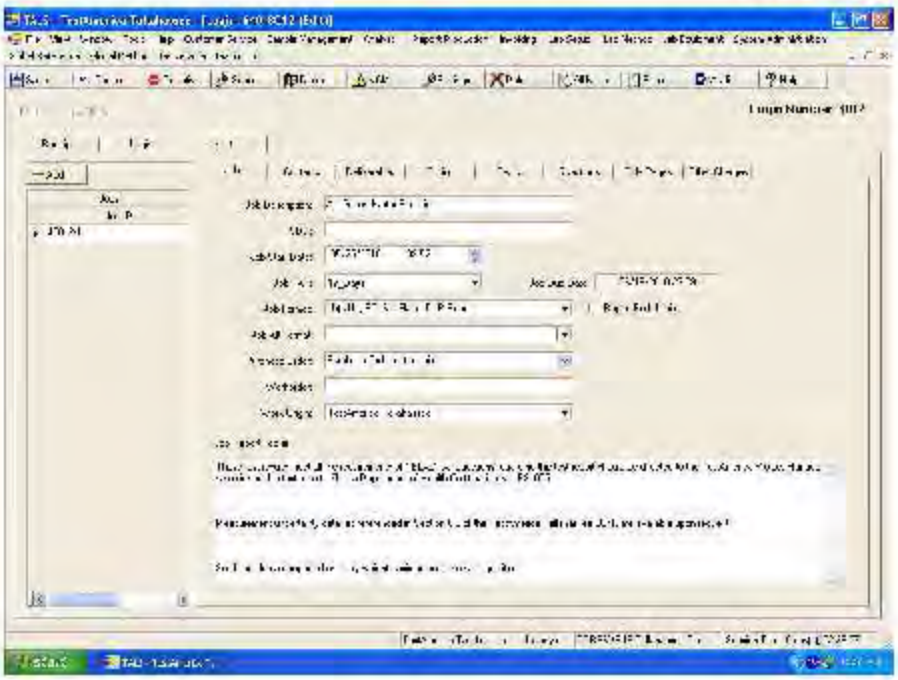
A JOB is a set of samples from a single Login or multiple Logins. Samples are reported as JOBS.

The JOB Tab is primarily a Project Manager function. This information is copied down from the associated Project.

1. **Job Description** – carried over from the Project
2. **SDG** – an alternate naming convention for joining multiple LOGINS into a JOB for reporting
3. **Job Start Date** – carried over from Receipt Date/Time
4. **Job TAT** – turnaround time defaulted from Project. Turnaround Time can be modified per Login and Job.
5. **Job Due Date** – calculated from turnaround time specified
6. **Job Format** – the default formatter from the Project. Formatter can be changed per Login and Job.
7. **Report Both Limits** – not used at this time
8. **Purchase Order** – carried over from the Project. PO can be changed per Login and Job.
9. **Workorder** - carried over from the Project. A work order can be changed per Login and Job.
10. **Work Origin** – location where samples arrived and were logged
11. **Job Report Footer** – carried over from the Project. Text can be edited at the login level.

TALS Training Brief

12/28/10
CW-I-T-071

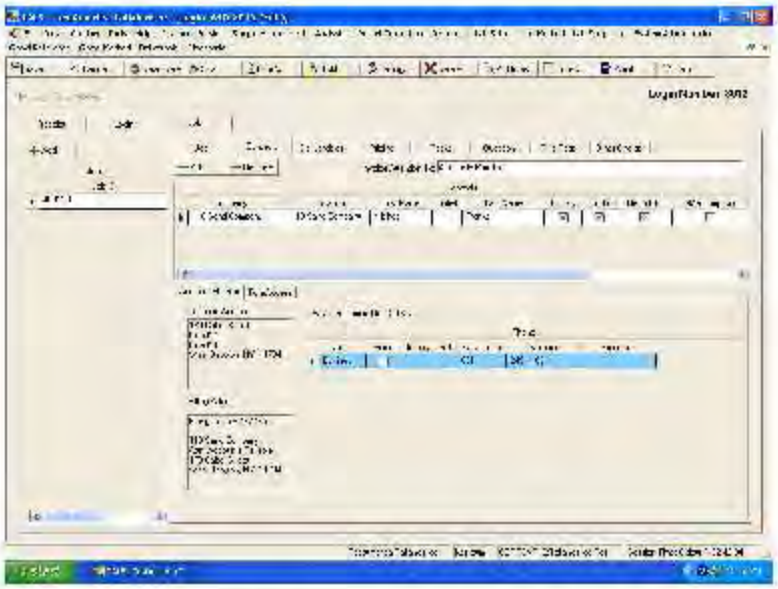


Job – Contacts

The Contact information is copied down from the Project exactly as built. The information can be modified per job. Additional contacts may be added as needed.

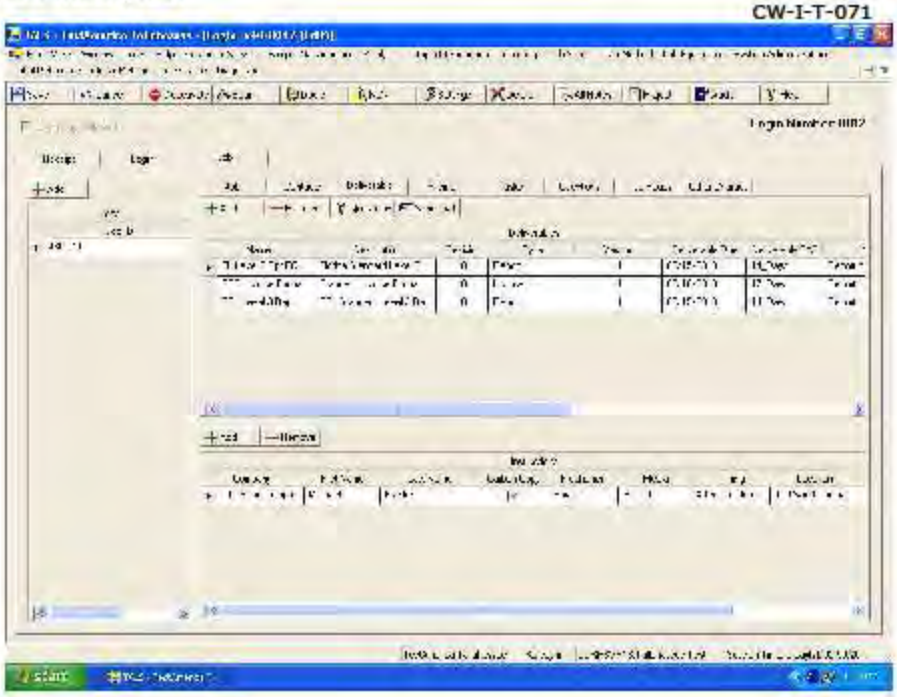
Page 25 of 31

TALS Training Brief 12/28/10
CW-I-T-071



The Deliverable information is copied down from the Project exactly as built. The information can be modified per job. Additional Deliverables may be added as needed.

TALS Training Brief 12/28/10
CW-I-T-071

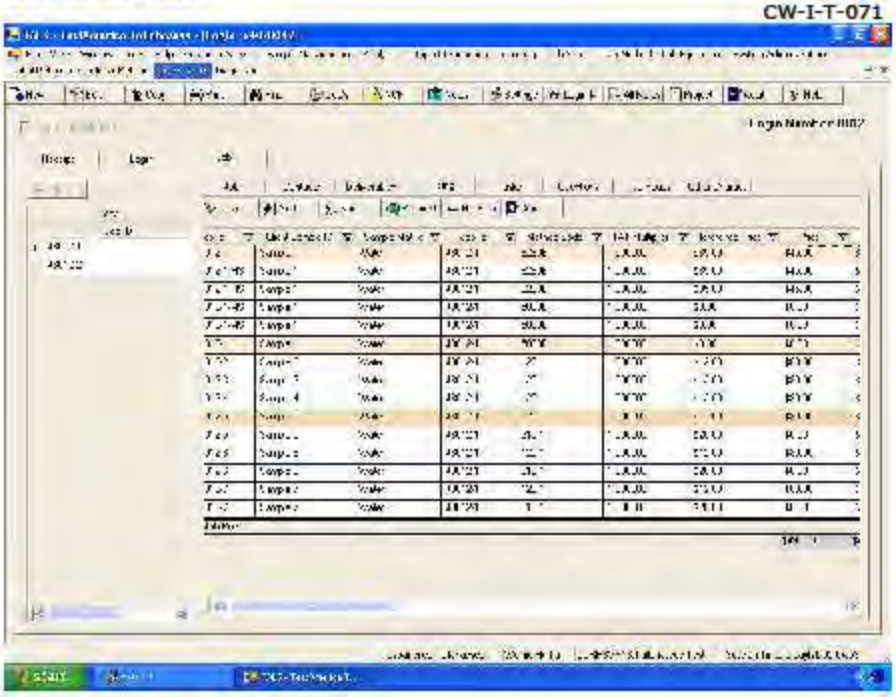


Job – Pricing

The Pricing is copied down from the Project. Pricing can be modified and additional JOBS can be added, if needed.

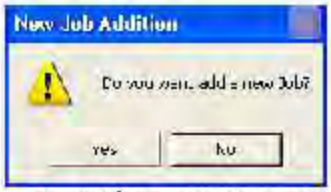
Page 27 of 31

TALS Training Brief 12/28/10
CW-I-T-071

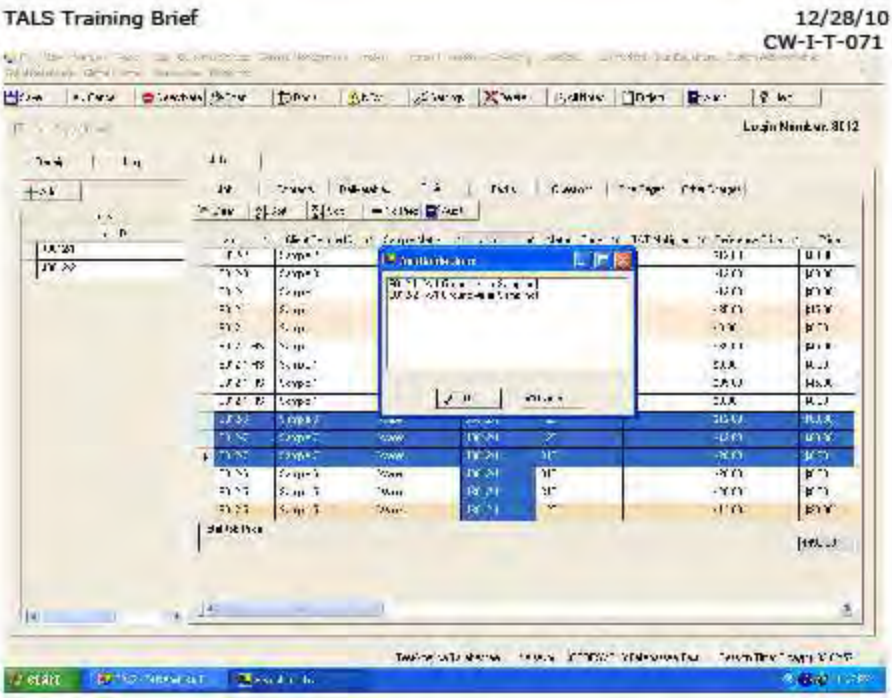


To add another JOB to a Login:

1. Click [Add]



2. A job can contain any combination of methods/samples/logins. You will assign these on the Pricing tab
3. Sort the Pricing grid by any combination of the headers. Sort and Filter the list to obtain the desired set of samples that you want on another job.
4. Left click the Job ID header to activate the column. Right-click the JOB ID header and choose the job # to assign desired job to samples/methods in the sorted grid.



5. All the JOB information [tabs] can be customized per JOB.

Job – Task

1. As Tasks are completed (simply by placing a checkmark in the space provided next to the task), the date/time and user are recorded.
2. Tasks are hierarchal, and the previous Task must be completed before completing the next task.

*Note: The Login Review Task must be checked on the **Login – Task** tab prior to setting the review tasks in the **Job** tab.*

Job – Questions

This tab may have information that needs to be completed. If a client needs an EDD which requires information that is not otherwise available in TALS, **Questions** are used to accomplish this. Simply answer the question to store the answer. Job Questions are answered on a Job level basis. Questions are programmed into the EDD's and will pull into the login when an EDD that requires them is added.

Example:

Question: **What is the lab location?**
Answer: **TAL**

Note: *The answer to a particular question may be answered by the appropriate group, depending on the question. Just because this tab is in the LOGIN Module doesn't imply that the answer will come from Sample Receiving.*

Job – Title Pages

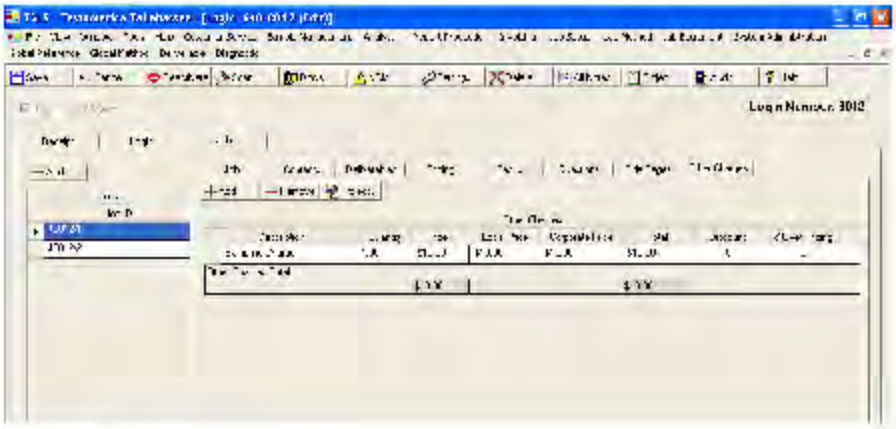
Not used.

Job – Other Charges

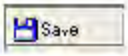
Non-Analytical charges are copied from the Project. [**Add**] / [**Remove**] **Other Charges** as needed.

*Note: Certain Other Charges are automatically added based on the **Login** or **Project.** For example, EMF (Environmental Management Fees) are automatically added to all logins and are a percentage of the total price for the job.*

TALS Training Brief 12/28/10
CW-I-T-071



SAVE the Login.



Page 31 of 31

To Ship an Order off the Shipping Desktop

ICOCs

Shipping Desktop

For detailed instructions on how to use the Shipping Desktop, ICOCs (internal and external) and the Shipping Desktop, please reference the Oasis website.

Attachment 9. EXAMPLE COOLER TEMP LABEL

TB____Cooler____Cor_ _ _Unc____
Cooler Dsc_ _ _ _ _ _ _ _ _@Lab_ _ _ _ _ _ _
Wet/Packs Packing_ _ _ _ _ _ _ _ _ _ _ _ _

Attachment 10: Work Instruction for Tracking Service Center Revenue

The purpose of this appendix is to provide instructions to be followed in the gathering of data to track revenue sourced from service centers.

Label Generation

Labels will be printed centrally and supplied to each location.
Labels will be generated with location name and a bar code.
Labels will be generated for all service center locations, including those labs that house service center functions.

When a location runs low on labels, they need to contact Janet.McAleese@testamericainc.com to request additional labels. Two week's notice should be provided to obtain additional labels.

Service Center Responsibility

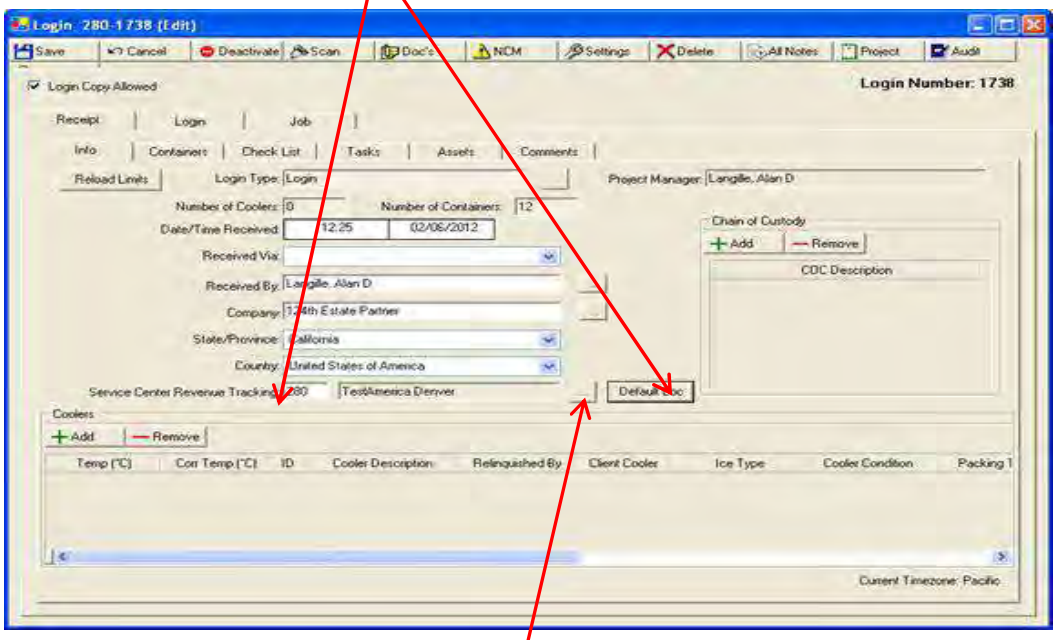
Each service center will be responsible for affixing a label on the inside of the cooler lid in the upper left corner. Service Center personnel will label every cooler that they ship out, drop off, pick up, or that is delivered to them from a client, every time.

Service Center personnel need to ensure that any old labels are removed, prior to affixing their labels.
(Note: Labels are to be removed by lab personnel, but in order to ensure integrity of the system, it would be best for SC to check)!

Lab / Sample Receiving Responsibility

For every log in; sample receiving personnel are responsible for scanning the label from the inside of the cooler lid, which will enter the "Service Center Revenue Tracking" into TALS.

If there is no label present on the inside of the cooler lid, sample receiving will select their home location by using the "Default Loc" button.



If the scanning process is not successful (scanning failure, or other reason) the service center location must be selected using the list feature in TALS.
As of July 1, 2012 this data will be considered mandatory in TALS. The Login cannot be saved successfully until the Service Center Tracking field information has been completed.

***Once the label from the cooler is scanned, the label must be removed from the cooler and thrown away*.**

Attachment 11: Example Sample Receiving Triage and Labeling Checklist

TestAmerica Seattle
Sample Receiving Triage and Labeling Checklist

Priority Level/ #: _____ Login #:_____ Date/Time Received:_____

Company Name & Sampling Site: _____

PM/PMA to Complete This Section at Cooler Greet: **Initials**_____

TALS Project #: _____ DoD: *Yes* ☐ *No* ☐
Special Instructions:

Time Zone: • Guam • Hawaii • Alaska • PDT/PST • MDT/MST • CDT/CST • EDT/EST • OTHER State:

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR)

Triage Checks: **Initials** _____

N/A Yes No

☐ ☐ ☐ 1. Are there Short Holds or Rush?

☐ ☐ ☐ 2. Are there Stir bar VOAs which need to be placed in the interim storage freezer?

☐ ☐ ☐ 3. Are there bulk soil jars for VOAs which will require MeOH preservation?

Notes: _____

Unpacking Checks: ***Initials*** _____

N/A Yes No

☐ ☐ ☐ 1. Are there no discrepancies between the sample IDs on the containers and the COC?

☐ ☐ ☐ 2. Are there no discrepancies between the sample date/time on the container and the CoC?

☐ ☐ ☐ 3. Do sample containers have legible labels?

☐ ☐ ☐ 4. Are all sample containers intact (not broken or leaking)?

TA-WI-SC-010-R00

TestAmerica Seattle
Sample Receiving Triage and Labeling Checklist

N/A	*Yes*	*No*	
☐	☐	☐	5. Was Sample Preservation verified and found to be correct? (excluding VOA, Oil & Grease, and TOC volumes)

Lot # _____ Preservation # _____ Date _____
Lot # _____ Preservation # _____ Date _____
Lot # _____ Preservation # _____ Date _____

☐ ☐ ☐ 6. Do all VOA sample vials have no headspace or bubbles >6 mm (1/4") in diameter?

☐ ☐ ☐ 7. Were water VOA vials labeled as preserved? ☐ Yes – ***use prep method 5030*** ☐ No – ***use prep method 5030_UP***

☐ ☐ ☐ 8. Are all samples single phase? (i.e., no multiphasic samples are present.)

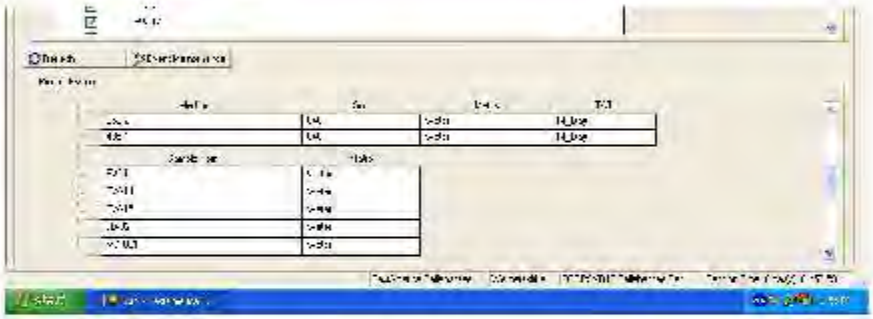
Labeling and Storage Checks: ***Initials*** _____

☐ ☐ ☐ 1. If subcontract work was requested, was volume placed on sub shelf?
☐ ☐ ☐ 2. Did the sample ID on TA label match the client's sample ID on container? ***Verified by:*** _____
☐ ☐ ☐ 3. Were Terracore/Encores delivered to VOA lab?
☐ ☐ ☐ 4. Were stickers for special archiving instructions affixed to each box?

TA-WI-SC-010-R00

Attachment 12: Logging in samples that have Sites and Events(on the Oasis website)

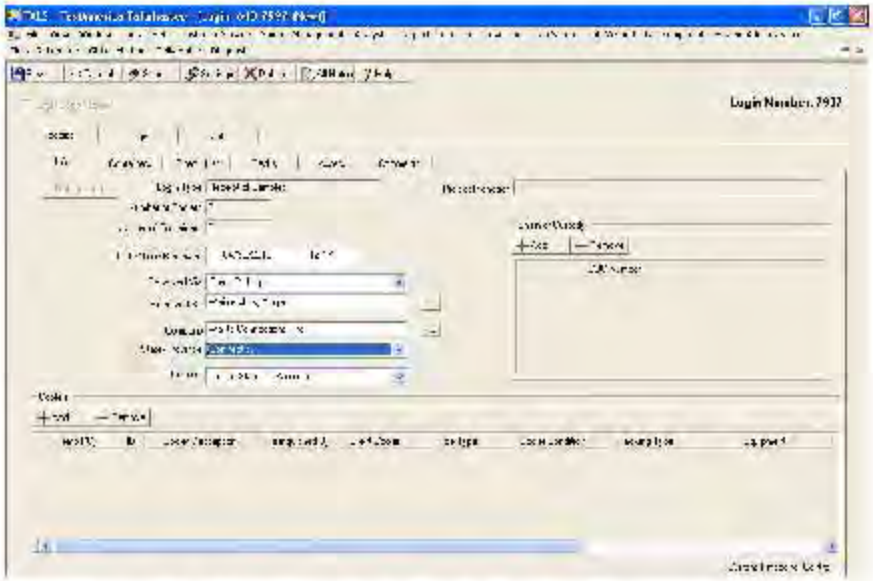
17. Add additional Project Events as needed by returning to the [Event Maintenance] module.
18. Expanding the tree of each Project Event will display the Project Event, Project Sampling Event, Methods and Site samples.



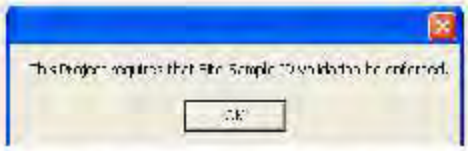
19. Click [Save] to save the project.

To Create a Login with Sites and Events

1. From the main menu, select *Sample Management* by clicking the application.
2. From the *Sample Management* menu, select *Login* by clicking the application.
3. Click [New].



4. Fill in applicable information on the 'Receipt' tab. See *Login* documentation posted on the intranet for additional information on the *Login* module.
5. From the 'Login' tab, [Add] the project number. When the project is added the user will be prompted to accept the enforcement of site samples if only one project event exists.

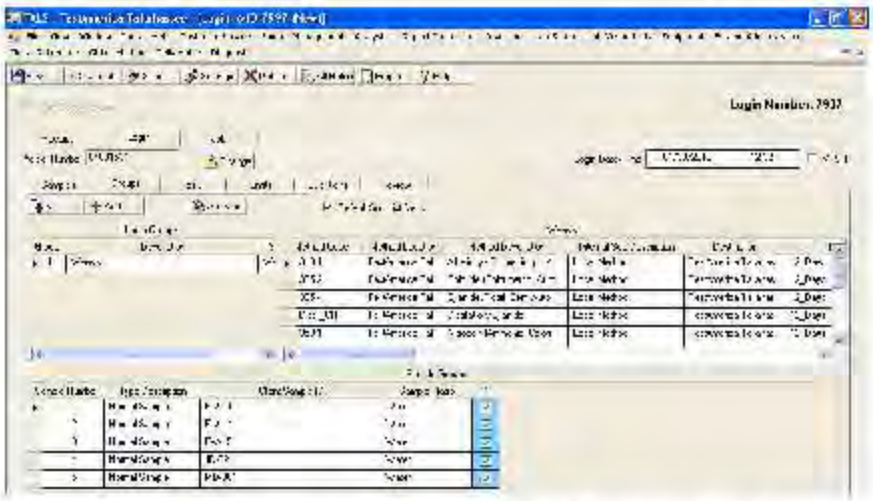


NOTE: If the project has more than one project event, the user will be prompted to select the project event from a dropdown menu then accept site validation enforcement.

6. Click [OK].
7. In the next pop up, select the site samples to include and fill in the dates and times sampled.

8. Click [OK].
9. The samples will be filled in automatically.

10. And the methods will be automatically assigned to the samples.



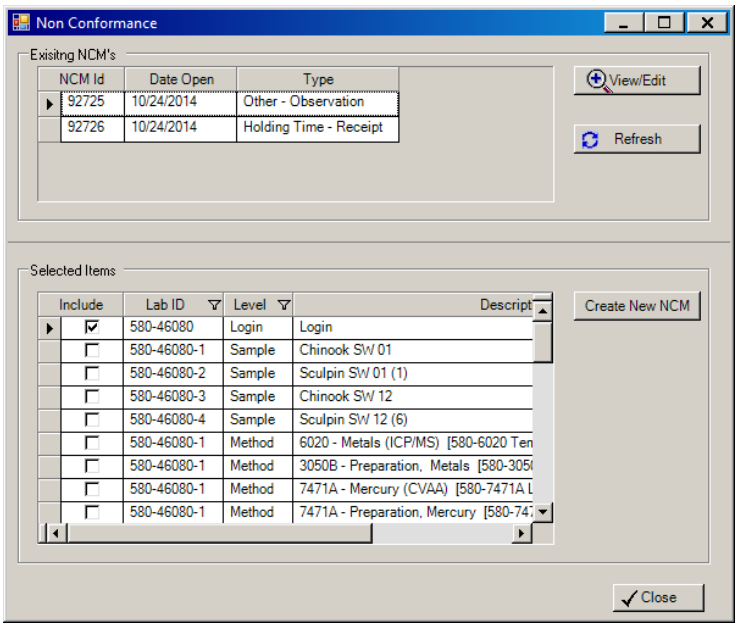
11. The user will need to return to the 'Receipt' tab and fill in the number of containers received for each sample. NOTE: The user can fill in the container information prior to selecting a Project Number if desired.
12. Other login information will be filled in according to the requirements set in the project.
13. Check on the Login Review and [Save] the login.

Attachment 13: Non-conformance memos (NCMs)

NCM Creation

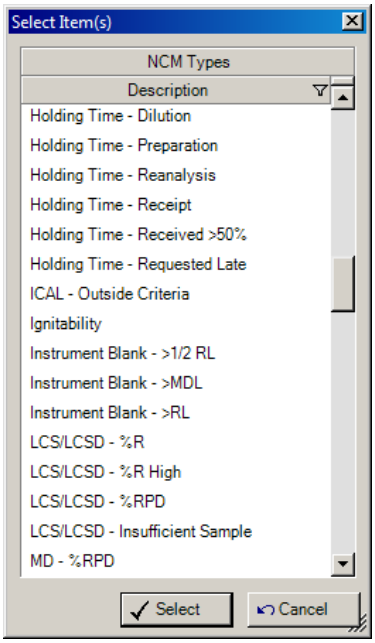
In the affected login click on NCM on top menu.

Select items and/or methods affected.

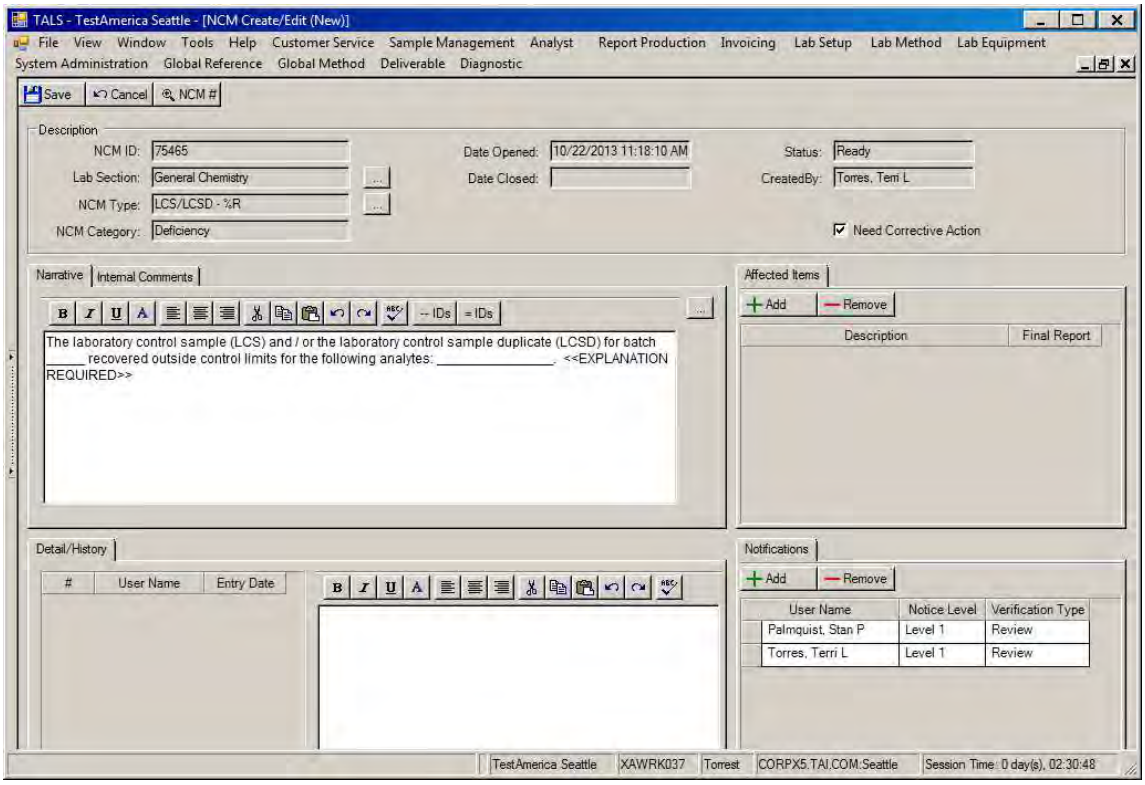


Click Create New NCM.

Select appropriate NCM Type.



The screen will now look like this:



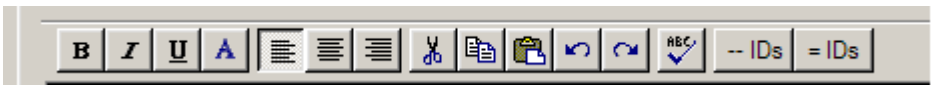
There are several tools available when writing an NCM.

- Canned Text
- Spell Check
- Cut and Paste to Word for Grammar Check

The NCM canned text should be used whenever possible. It is important that the information coming from the lab be a consistent as possible. However, not all information is contained in these modules.

Some of the NCMs will come up with the text in brackets somewhere narrative spot like this: [EXPLANATION REQUIRED]. Others will have fill in spots indicated by an underline like this: associated with batch _____ .
Anything that is in brackets or has an underline fill in spot will need to be removed the appropriate information inserted in its place. What is entered here will show up on the final report, so be thorough, define acronyms, cover all points, use proper grammar, and make it presentable.

Anywhere the sample IDs need to be listed the <&commaMerge&> should be used, either left where it appears in the canned text or added to the narrative by placing the curser where it is to be added and clicking the –IDs button. This will pull in the sample IDs for all samples you have under the Affected Items tab.



There are a couple of pieces of information that should be checked and added.

- Check that all affected samples have been pulled in
- Make sure the most appropriate NCM is used

The NCM module does have a Spellchecker. USE IT!

All samples should have the Final Report button checked if the NCM Type is a deficiency.

Below Affected Items is the Notifications screen. This box will automatically be filled in with the appropriate reviewers. Other people can be added at this point to receive notifications, or, if the designated reviewer is out, a backup can be chosen at this time.

Upon saving, emails are automatically sent to anyone in the notification box.

How to edit an NCM

- Find and open the affected login.
- Click on NCM on top menu.
- Highlight the NCM Id that needs edited and click View/Edit.
- Make necessary changes.
- If any corrective actions were required enter in the internal comments tab of the narrative. Note at the end of the NCM in the Narrative Tab: See Internal Comments.
- Put a summary of changes in Detail/History. The original narrative text will automatically be saved in this section.
- Save NCM.

Title: Chain-of-Custody, Internal Sample Transfer, Storage, and Security

Approvals

Signatures on File

Terri Torres — Date
Quality Assurance Manager

Dennis Bean — Date
Laboratory Director

Manjit Nijjar — Date
Health & Safety Coordinator

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 PURPOSE

This SOP describes the procedure for internal transfer of samples, and custody procedures used for samples within the laboratory.

2.0 SAFETY

2.1 There are no specific safety hazards associated with this SOP.

2.2 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

3.0 DEFINITIONS

3.1 Sample Custody: a sample is considered in custody if it is in a person's actual possession, in view after being in a person's actual possession, in a secured area, or restricted to authorized personnel.

3.2 Chain-of-Custody Form: A critical legal document, which records collection, possession, and transfer of samples from one person or party to another.

4.1 Internal Chain-of-Custody Report (ICOC): The report used to document the storage, handling and archival of client samples while in possession of the TestAmerica Seattle laboratory.

4.0 PROCEDURE

4.1 Samples are received in the Sample Receiving Department and custody is relinquished from the client or a designated carrier to the sample custodian. Samples not immediately processed are placed in the walk in refrigerator, and are in the custody of the sample receiving technicians. Samples submitted for 8260 and 624 volatiles analysis are placed directly into a refrigerator designated for volatiles (locations that have storage blank monitoring). Samples will either be routed as routine samples (sections 4.3 through 4.7) or as controlled (legal) custody samples, and routed as described in section 4.8.

4.2 Aqueous samples marked as “preserved” are verified to be in the appropriate pH range by sample custodians once samples have been unloaded for log-in and/or labeling following procedures outlined in SOP TA-QA-0001. The sample pH may be checked again by the appropriate personnel if the method deems such checks necessary.

4.3 Once a sample has been processed in the sample receiving area, it is placed into the appropriate refrigerator; the sample location is recorded in the TestAmerica LIMS (TALS). All possible sample locations are available within the TALS login. All refrigerators are in areas designated for TestAmerica Seattle personnel only.

4.4 Samples may be designated as “legal custody” or “controlled custody” samples per client request. In these cases, the samples will be tracked with an internal chain of custody (ICOC).

4.5 When an analyst is ready to extract or process the sample, the sample is removed from the refrigerator. If “legal custody” or “controlled custody” is required the sample is tracked in TALS using the internal chain of custody (ICOC) module. After the technician or analyst is completed with the sample it is returned to the proper location within the refrigerator. If “legal custody” or “controlled custody” is required the sample is tracked in TALS using the internal chain of custody (ICOC) module. Every effort is made to minimize

the amount of time that a sample (in the original sample jar) is stored outside of the refrigerator, both to minimize warming of the sample and to keep the volume readily accessible to other analysts. Samples completely consumed must be noted as such for disposal purposes.

4.6 Sample extracts, digests, filtrates, and dilutions are stored in the part of the laboratory where testing will be performed, unless these volumes are also required to be in "controlled custody" (as described in Section 4.8). All parts of the laboratory where preparation or analysis of samples is performed are restricted to TestAmerica Seattle personnel. The extracts are considered to be in the analysts' custody until transferred to the waste disposal area.

4.7 Samples remain in the same location in the refrigerator for 30 calendar days from receipt. Samples are then removed from the refrigerator and placed on shelves in the waste disposal area, unless samples are to be returned to the client. The waste disposal area is restricted to TestAmerica Seattle personnel. Samples are stored in this area for not less than 45 days, after which the samples are disposed of following the appropriate documented procedures. The method of disposal (consumed, returned to client, disposed of by laboratory) must be documented in TALS for the job number.

4.8 Controlled custody samples.

4.8.1 Samples requiring internal controlled custody are placed into the refrigerator by the sample receiving technician.

4.8.2 Foreign soils have a special tag placed on them to ensure that all personnel are aware that they are foreign soils and thus require specific disposal methodology. Foreign soil containers are placed in a locked refrigerator or freezer; the key is kept directly across from the refrigerator on a magnet or on top of the freezer.

4.8.3 The samples are tracked using the LIMS Internal Chain-of-Custody (ICOC) Module (see attachment I for the ICOC process flow chart).

4.8.4 For metals, general chemistry and semivolatile analyses the samples are kept in designated areas (aqueous samples for metals analysis are stored at ambient temperature in the main lab; all others are stored in either of the two Sample Walk-Ins). The TestAmerica LIMS (TALS) documents the location. Analysts and preparation technicians check the backlog sheets in the LIMS to determine which samples to remove and extract or digest.

4.8.5 For volatile analyses the samples are kept in designated areas in the volatiles laboratory. The TestAmerica LIMS (TALS) documents the location and the location is rotated by date. Analysts and preparation technicians check the backlog sheets in the LIMS to determine which samples to remove and extract or analyze.

4.8.6 The analyst returns the unused volume to the correct storage location. If it is a foreign soil, the samples (or empty container, if the sample was consumed) will be returned to the foreign soil fridge located in the main lab or the freezer located in the volatiles lab and the refrigerator or freezer should be locked.

4.8.7 If the sample is consumed during analysis this is noted by the analyst in TALS (and only the extract or digest is retained if it is not a foreign soil).

4.8.8 The sample extract or digest is kept in the appropriate area as seen in Table 1 below and is retained for a minimum of 40 days after extraction for semivolatiles,

14 days after digestion for metals and 30 days after extraction for volatiles. The fractions are then removed and properly disposed.

4.8.9 If the sample is to be returned to the client, the sample custodian will note this, and ship the container(s) as instructed under controlled chain of custody. If the sample was consumed during analysis, this will be noted by the analyst and/or sample custodian and the Project Manager will be informed so as the client can be informed. Otherwise the sample custodian will relinquish the unused sample volumes to the disposal technician when all analyses are complete, with "relinquished" and "received" signatures and the date recorded in the ICOC. Unless it is a foreign soil and then it will be kept in locked storage until such a time as the soil is autoclaved for disposal.

4.8.10 These internal controlled custody procedures may be extended to individual extracts and digests when requested/required by the client. If a client requests samples that are not foreign soils to be stored in a locked box, the client will be charged for any additional costs to acquire the locked box.

4.8.11 If the client requests that samples be held past completion of the report, the samples are stored in either the general laboratory walk-in refrigerator or freezer or on a specific shelf in the waste disposal area.

TABLE 1. EXTRACT, DIGEST, FILTRATE, OR DILUTION CONTROLLED CUSTODY LOCATIONS

Refrigerator or Freezer #	Analysis	Volume	Location	Methods*
401	Semivolatile Organics	Extracts	Main Lab	8270, 8151A, Organotins, 8082, 8081, DRO
33	Semivolatile Organics	Extracts	Main Lab	8270, 8151A, Organotins, 8082, 8081, DRO
31	Volatile Organics	Water Samples	Volatiles Room	8260, 624, Gasoline, VPH
5	Volatile Organics	Soil Samples	Volatiles Room	8260,Gasoline, VPH
13	Volatile Organics	Extracts	Volatiles Room	8260, Gasoline, VPH
554	Volatile Organics	Soil Samples	Volatiles Room	8260

*not necessarily all methods performed

5.0 RESPONSIBILITIES

5.1 Sample receiving personnel are responsible for putting samples into the proper storage location as designated, to verify preservation of water samples upon receipt and to properly adjust the pH if necessary, and to document preservation adjustments by NCM in the Laboratory Information Management System (LIMS).

5.2 Extraction Technicians and Analysts are responsible for returning the unused portions of

samples to the designated storage area and logging samples in and out of locations in TALS using the ICOC module.

6.0 REFERENCES / CROSS-REFERENCES

None

7.0 ATTACHMENTS

Attachment 1: TALS – Internal Chain of Custody (ICOC) process flow chart

8.0 REVISION HISTORY

Revision 16.1, dated 11 May 2018
- Updated approvers
- Minor grammatical edits

Revision 16, dated 9 May 2016
- Updated method in section 4.1 from 524 to 624
- Updated section 4.4 to current practice
- Added locked freezer for foreign soil throughout
- Updated methods in Table 1

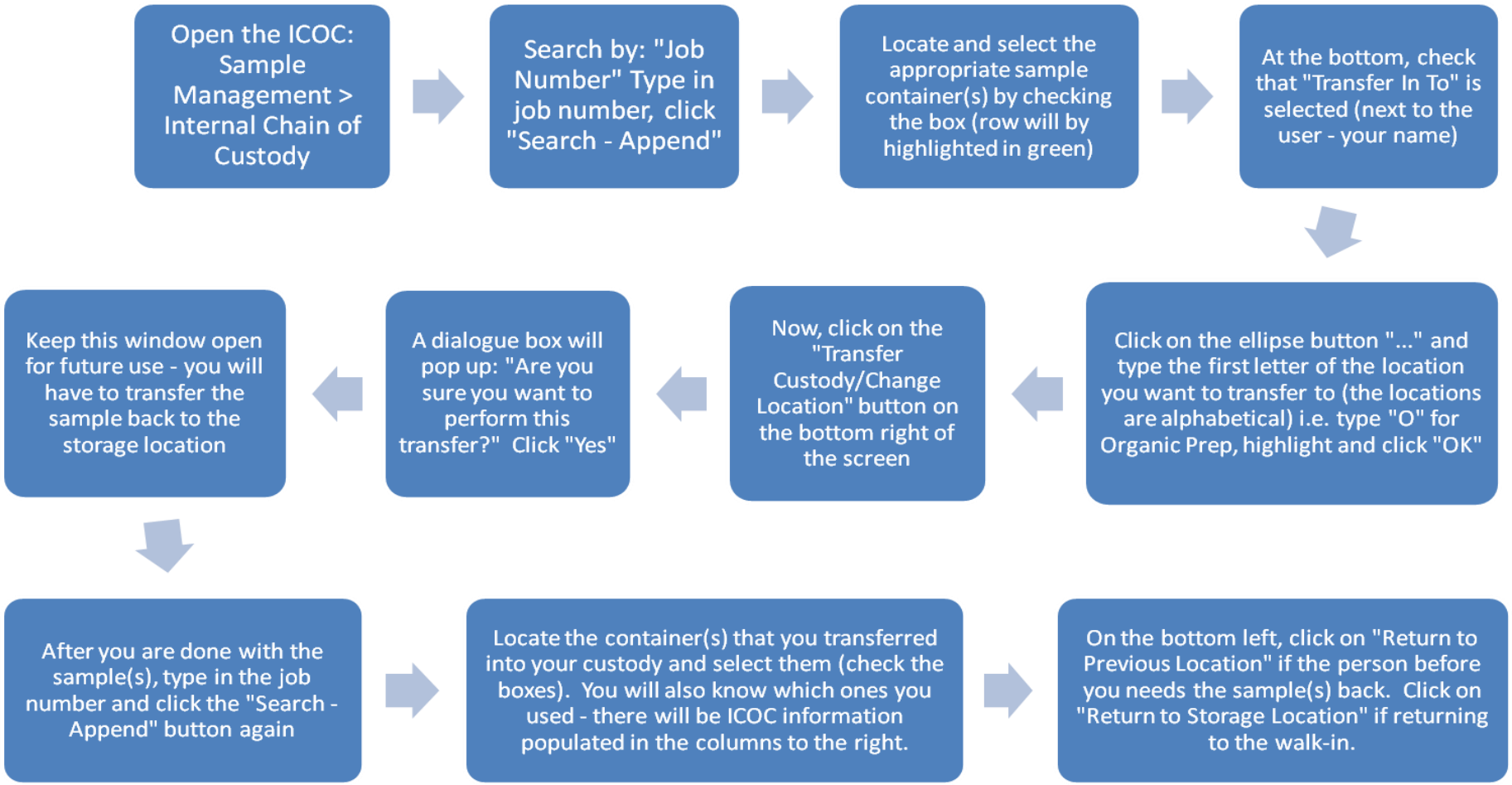
Revision 15, dated 7 May 2014
- Added what locations Volatile samples can be stored, section 4.1
- Updated ICOC to being done in TALS, sections 4.5 and 4.8.6
- Updated length of time samples are in cold storage, section 4.7
- Updated length of time extracts are retained, section 4.8.8
- Updated Table 1 to include semivolatile extracts
- Changed attachment from ICOC form, no longer in use to process flow chart for ICOC in TALS

Revision 14, dated 7 May 2012
- Added analysis that must be placed directly into Volatiles, section 4.1
- Added the time pH is taken of samples in sample control, section 4.2
- Added the location of the foreign soil fridge key, section 4.8.3
- Added 2nd walk-in, section 4.8.4.1
- Updated location of where samples need to be placed/added foreign soil fridge, section 4.8.5

Revision 13, dated 26 March 2010
- Updated the storage requirements of aqueous metals samples, section 4.8.4.
- Updated Table 1 (Controlled Custody Locations)
- Integration of Seattle and Tacoma operations

Revision 12, dated 19 April 2008
- Integration of TestAmerica and STL operations.

Attachment 1
TALS – Internal Chain of Custody (ICOC) process flow chart



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Particle Size Analysis of Soils [Methods ASTM D422-63, D7928, D6913]

Approvals

Signatures on File	
Stan Palmquist Date	Manjit Nijjar Date
Inorganic Department Manager	Health & Safety Manager / Coordinator
Terri Torres Date	Dennis Bean Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

This SOP describes the laboratory procedures for the preparation of and subsequent determination of particle size distribution in soil samples that contain sand, silt, clay, and gravel.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

Note: ASTM has set the status of methods D422-63, as "Withdrawn with no replacement".

2.0 Summary of Method

A soil sample submitted for particle size analysis is prepared according to dry preparation of soil for particle size analysis (section 10.2) or wet preparation of soil samples for particle size analysis (section 10.3). Particles greater than 63 µm (gravels to fine sands) are determined by sieve analysis while particles less than 63 µm (silts and clays) are determined by sedimentation using a hydrometer followed by sieve analysis.

After wet or dry sample preparation, the sample is passed through a No. 10 sieve. The particles retained on the No. 10 sieve (greater than 2.00 mm) are further separated by sieve analysis. The portion of the sample that passed through the No. 10 sieve is transferred to a glass sedimentation cylinder to which distilled water has been added. Seven hydrometer reading are taken over 24 hours. After the final hydrometer reading, the suspension is rinsed over a No. 230 (63 µm) sieve, dried, and further separated by sieve analysis.

Method Modification: For work in the Pacific Northwest, it is important to utilize a No. 230 sieve. We replace the No. 200 sieve in the method above with a *No. 230 sieve as the final sieve*.

3.0 Definitions

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Seattle Quality Assurance Manual (QAM).

Gravel: Greater or equal to 4750 µm

Course Sand: Greater or equal to 2000 µm and less than 4750 µm

Medium Sand: Greater or equal to 425 µm and less than 2000 µm

Fine Sand: Greater or equal to 75 µm and less than 425 µm

Silt: Greater or equal to 2 µm and less than 75 µm

Clay: Less than 2 µm

4.0 Interferences

Not applicable.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental

Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this method. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

- Top-Loading Balance sensitive to 0.01 g
- *250 mL specimen cups with lids*
- Mechanical Stirring Apparatus and Dispersion Cup
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer; ASTM 151H in specification E 100
- Thermometer: accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sieves of the following sizes:

3.0 in (75.00 mm)	No. 20 (850 µm)
2.0 in (50.00 mm)	No. 40 (425 µm)
1.5 in (37.50 mm)	No. 60 (250 µm)
1.0 in (25.00 mm)	*No. 100 (150 µm)*
3/4 in (19.00 mm)	*No. 140 (106 µm)*
3/8 in (9.50 mm)	*No. 200 (75 µm)*
No. 4 (4.75 mm)	*No. 230 (63 µm)*
No. 10 (2.00 mm)	

- *Oven with temperature range of 110°C ± 5°C*
- Timing Device with second hand and capable of counting up to 25 hours
- Aluminum Weigh Pan(s)
- Liter Plastic Beakers
- Stainless steel spatulas, spoons, metal, and bristle brushes
- Ro-tap machine

6.1 Software

- LIMS system: TALS version 1.0 or higher
- Excel 2003 or later

7.0 Reagents

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 Deionized (DI) Water

7.3 Sodium Hexametaphosphate Solution: Combine *50 g* of sodium (hexa)metaphosphate to 900 mL of DI water in a 1 L volumetric flask. Fill to 1 L mark. Mix until the solution is homogeneous. Assign an expiration date of 30 days from date of preparation.

7.4 *Isopropyl Alcohol: 70% or greater used as a foam inhibitor.*

7.5 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 The minimum quantity of sample needed for collection depends on the subsequent analyses to be performed. The typical amount needed is 115 to 230 grams (minimum) of soil. Larger amounts (from 500 to 5000 grams) may be required for particle size analysis of soils with appreciable gravel component. All samples should be collected in glass or polyethylene jars, and immediately following collection, sealed and cooled to 4°C in order to preserve the moisture content of the sample.

8.2 Unless otherwise specified by client or regulatory program, after analysis samples are retained for 30 days and then disposed of in accordance with applicable regulations.

Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass or HDPE	500 grams	Cool 0-6°C	6 months	PSEP 1986

9.0 Quality Control

Not applicable.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in SOP TA-QA-0014.

10.1.2 *Minimum Mass Requirements: The analyst estimates the moisture content and maximum particle size of each sample. The minimum sample size for fines is based on the largest particle size and dry mass.*

10.1.2.1 *The minimum sample size for 99% passing #10 sieve is 50 g dry mass, #4 sieve is 75 g, and 3/8" sieve is 165 g dry mass.*

10.1.2.2 *The minimum amount of fines in the sedimentation cylinder should be 15 g. Some materials (e.g. clean sands) will not yield detectable amounts of fines. These materials may not be appropriate for the hydrometer portion of this test (D7928).*

10.2 Wet Preparation

10.2.1 *Percent Moisture*

Place *10-20 g representative sample into a pre-tared weighing tin. Record the wet weight of the sample. Place in a 110 ± 5°C oven to dry. Record the mass of the tin and dry sample.*

Hydrometer Test (D7928)

10.2.1.1 Add *100 mL* of sodium hexametaphosphate solution to each beaker. Stir/*shake until all of the soil aggregations are broken-up (minimum of 1 minute)*.

10.2.1.2 Pour the contents of the cup into a 1000 mL sedimentation cylinder. Rinse the cup with DI water to ensure that the entire sample is transferred to the sedimentation cylinder.

10.2.1.3 *Fill the sedimentation cylinder to the 1000 mL mark and allow to sit overnight. Also fill a cylinder with DI water and a cylinder with 100 mL SHMP blank for rinsing/hydrometer verification.*

10.2.1.4 Record the ID of the hydrometer that you intend to use. *For each hydrometer, record and verify a reading of DI water at 1.000±0.0005. Select a different hydrometer if the readings are outside of this range.*

10.2.1.5 *For each hydrometer, record a reading of a 100 mL SHMP blank.*

10.2.1.6 *Record the start time and set the timer for elapsed time. Record the ambient temperature.*

10.2.1.7 Shake sedimentation cylinder by rotating the cylinder up and down for one minute approximating at least 60 turns (one turn upside down and then right side up constitutes two turns). Hydrometer reading times start after completing this rotation. *If significant foam develops, a spray of isopropyl alcohol may be used to reduce foaming.*

10.2.1.8 Take reading every 2, 5, 15, 30, 60, 240, and 1440 minutes. Record each reading on the bench sheet, and then transfer all this information into the appropriate cells of the spreadsheet. Remove and clean the hydrometer by twisting and dripping into a clean DI water bath between each reading.

10.2.1.8.1 To take a reading, gently insert the hydrometer into the cylinder then wait ~10 seconds. Read the hydrometer at the top of the meniscus to the nearest 0.0005. *Gently remove the hydrometer.*

10.2.1.9 *The sample is now ready to be washed in a #230 sieve. If any reading were missed the sample may now be reshaken and hydrometer reading taken t the appropriate time.*

10.3 Sieve Analysis

10.3.1 When the hydrometer test is complete, transfer the soil from the sedimentation cylinder to a #230 wet wash sieve.

10.3.1.1 Wash the soil through the #230 sieve until the water from the bottom of the sieve runs clear.

10.3.1.2 *Place the sieve in the oven. Dry at a temperature of 110°C, Remove the sieve from the oven and allow it to cool.*

10.3.1.3 Gently mix the dried contents of the *sieve* with a rubber–tipped pestle to break any soil aggregates that may have formed during the drying stage.

10.3.1.4 Transfer the dry sample into the sieve stack, ensuring that all material is transferred. Use hair or wire brushes to clean the sieve.

10.3.1.5 Place the sieve stack (see Section 6.0) on the Ro-tap machine and shake for ten minutes. *Ceramic beads maybe added to the sieve stack to facilitate disaggregation from the drying stage*

10.3.1.5.1 *Material larger than #4 maybe hand sieved.*

10.3.1.6 Transfer the material retained on each sieve to a tared weighing dish. Enter these weight measurements in the spreadsheet.

10.3.1.7 If appropriate, enter a brief description of the type of non-soil material (e.g. sticks, grass, wood, plastic) in the “Description of >#10 particles” of the EXCEL® spreadsheet in the cell labeled “Non-soil Material”.

11.0 Calculations / Data Reduction

11.1 Percent Solids

$$\frac{(dry\,\text{sample} + \text{pan}) - (\text{pan})}{(\text{wet sample})} \times 100$$

11.2 Sieve Analysis

Incremental Percent = IP = Mass retained on sieve / Dry Sample

3 inch Percent Finer = PF = 100% - 3 inch IP

2 inch through #230 Percent Finer: = PF of next largest sieve – IP of selected sieve size

11.3 Hydrometer Analysis

Particle size, Micron

1000* sqrt [930*viscosity/980*(SG-1) * (effective depth/time)]
Viscosity at sample temperature, poises
Effective Depth, cm = 16.29 -264.5*(actual hydrometer reading -1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8) + 10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt = square root
SG = specific gravity of soil
Viscosity = is the resistance of a liquid to flow.

11.4 Percent Finer (PF)

PF = constant*(actual hydrometer reading – hydrometer correction factor -1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used * sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1) - (high temp. reading-1) / (low temp.-high temp.))
Intercept = (low temp. reading-1) – (low temp. * slope)

12.0 Method Performance

Not applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Waste Disposal SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 Processed soil. Processed soil is disposed of in the Cubic Yard Box.

15.0 References / Cross-References

15.1 Standard Test Method for Particle Size Analysis of Soils, ASTM D422-63, Volume 04.08 Soil and Rock, American Society for Testing and Materials, Philadelphia, Pa., 1998.

15.2 Standard Test Methods for Particle-Size Distribution of Soils Using Sieve, ASTM D6913-04, American Society for Testing Materials, Philadelphia, Pa., 2009.

15.3 Standard Test Methods for Particle-Size Distribution of Fine-Grained Soils Using the Sedimentation Analysis, ASTM D7928-17, American Society for Testing and Materials, Philadelphia, Pa., 2017.

16.0 Method Modifications:

- #200 sieve has been replaced with #230 *for the final sieve*.
- *Hydrometers are read to the nearest ½ mark and not the ¼ mark.*
- Small variances in temperature throughout the hydrometer readings are not accounted for as they have a small impact on the recorded hydrometer reading.

17.0 Attachments

Attachment 1: Percent Solids Table

Attachment 2: Example Data Spreadsheet

Attachment 3: Example Particle Size Graph

18.0 Revision History

- *Revision 6, dated 10 October 2018*
  - *Updated approvers*
  - *Methods D7928 and D6913 have replaced D422*
  - *Defined particle sizes*
  - *Added #100 and #200 sieve*
  - *Added Isopropyl alcohol to reagents*
  - *Rewrote procedure to match D7928 and D6913*
  - *Updated references and method modifications*

- Revision 5, dated 14 October 2016
  - Added that methods have been withdrawn by ASTM in section 1
  - Replaced #200 sieve with #230 in section 2
  - Removed unneeded equipment
  - Removed Milipore requirement for ID water
  - Added Excel to the list of required software
  - Removed unused steps in section 10
  - Added processed soil to section 14.1.1

- Revision 4, dated 24 September 2014
  - Replaced #200 sieve with #230
  - Changed wording of SHMP prep
  - Removed Dry Prep section
  - Updated Wet Prep section to reflect the current method work flow and clarify procedures
  - Added Method Modifications for percent moisture, temperature correction, and #230 sieve
  - Updated Example Data Spreadsheet attachment
  - Removed Hydrometer reading table attachment

- Revision 3, dated 10 September 2012
  - Updated sections 5.0 and 5.2
  - Changed weighing procedures, Sections10.5.2 and 10.5.3

- Revision 2, dated 16 May 2011
  - Corrected typo in copyright section of page 1

- Revision 1, dated 16 April 2010
  - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
  - Added removal of expired standards Section 7.4.
  - Added daily balance check to Section 10.1.1.
  - Integration for TestAmerica Bothell and TestAmerica Seattle operations.

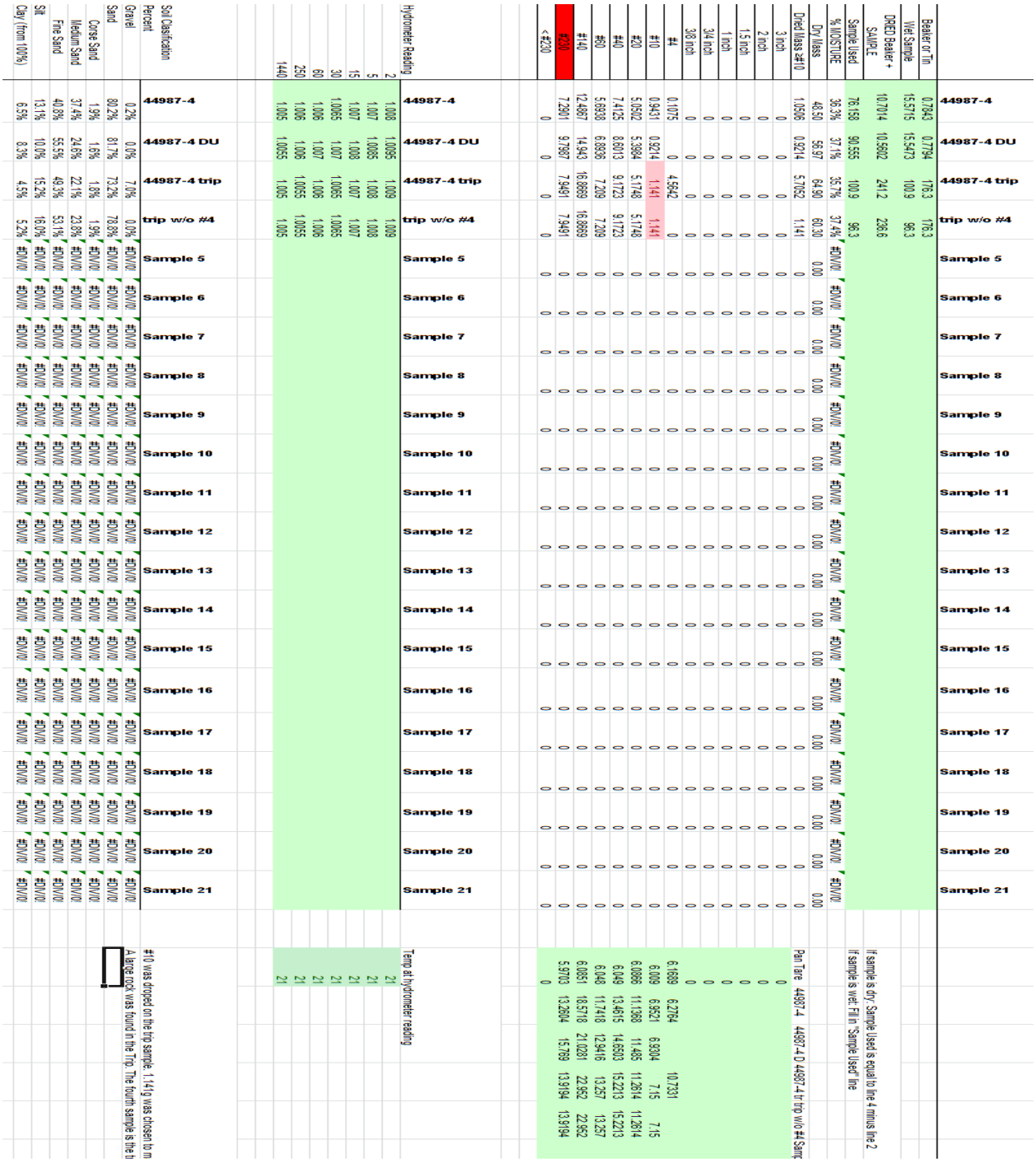
- Revision 0, dated 26 November 2008
  - Initial release.

Attachment 1. Mass of Sample (g) of Sample Needed for Particle Size Analysis based on Percent Solids Content

Instruction: Find the % solid value that corresponds to the % solid measurement in your sample. The value in the column next to the % solids specifies the mass of wet weight sample required to yield the required amount of dry weight needed for particle size analysis for each soil type. For example, if the % solids measurement for a sample is 55, then 91 g of wet weight sample is required to yield 50 g dry weight for silt/clay, 136 g of wet weight sample is required to yield 75 g of silt/sand, 182 g of wet weight is required to yield 100 g of sand, and 364 g of wet weight sample is required to yield 200 g of sand/gravel.

	Soil Type					Soil Type			
	Silt/Clay	Silty Sand	Sand	Sand/Gravel		Silt/Clay	Silty Sand	Sand	Sand/Gravel
% Solids	**50 g**	**75 g**	**100 g**	**200 g**	**% Solids**	**50 g**	**75 g**	**100 g**	**200 g**
1	5000	7500	10000	20000	51	98	147	196	392
2	2500	3750	5000	10000	52	96	144	192	385
3	1667	2500	3333	6667	53	94	142	189	377
4	1250	1875	2500	5000	54	93	139	185	370
5	1000	1500	2000	4000	55	91	136	182	364
6	833	1250	1667	3333	56	89	134	179	357
7	714	1071	1429	2857	57	88	132	175	351
8	625	938	1250	2500	58	86	129	172	345
9	556	833	1111	2222	59	85	127	169	339
10	500	750	1000	2000	60	83	125	167	333
11	455	682	909	1818	61	82	123	164	328
12	417	625	833	1667	62	81	121	161	323
13	385	577	769	1538	63	79	119	159	317
14	357	536	714	1429	64	78	117	156	313
15	333	500	667	1333	65	77	115	154	308
16	313	469	625	1250	66	76	114	152	303
17	294	441	588	1176	67	75	112	149	299
18	278	417	556	1111	68	74	110	147	294
19	263	395	526	1053	69	72	109	145	290
20	250	375	500	1000	70	71	107	143	286
21	238	357	476	952	71	70	106	141	282
22	227	341	455	909	72	69	104	139	278
23	217	326	435	870	73	68	103	137	274
24	208	313	417	833	74	68	101	135	270
25	200	300	400	800	75	67	100	133	267
26	192	288	385	769	76	66	99	132	263
27	185	278	370	741	77	65	97	130	260
28	179	268	357	714	78	64	96	128	256
29	172	259	345	690	79	63	95	127	253
30	167	250	333	667	80	63	94	125	250
31	161	242	323	645	81	62	93	123	247
32	156	234	313	625	82	61	91	122	244
33	152	227	303	606	83	60	90	120	241
34	147	221	294	588	84	60	89	119	238
35	143	214	286	571	85	59	88	118	235
36	139	208	278	556	86	58	87	116	233
37	135	203	270	541	87	57	86	115	230
38	132	197	263	526	88	57	85	114	227
39	128	192	256	513	89	56	84	112	225
40	125	188	250	500	90	56	83	111	222
41	122	183	244	488	91	55	82	110	220
42	119	179	238	476	92	54	82	109	217
43	116	174	233	465	93	54	81	108	215
44	114	170	227	455	94	53	80	106	213
45	111	167	222	444	95	53	79	105	211
46	109	163	217	435	96	52	78	104	208
47	106	160	213	426	97	52	77	103	206
48	104	156	208	417	98	51	77	102	204
49	102	153	204	408	99	51	76	101	202
50	100	150	200	400	100	50	75	100	200

Attachment 2. Example Data Spreadsheet



Beaker or Tin	0.7843	0.7794	176.3	176.3	44987-4
Wet Sample	15.5715	15.5473	100.9	96.3	44987-4 DU
DRED Beaker + SAMPLE	10.7014	10.5902	241.2	236.6	44987-4 trip
Sample Used	76.158	90.555	100.9	96.3	trip w/o #4
% MOISTURE	36.3%	37.1%	35.7%	37.4%	#DIV/0!
Dry Mass	48.50	56.97	64.90	60.30	#DIV/0!
Dried Mass a#10	1.0606	0.9214	5.7052	1.141	#DIV/0!
3 inch	0	0	0	0	0.00
2 inch	0	0	0	0	0.00
1.5 inch	0	0	0	0	0.00
1 inch	0	0	0	0	0.00
3/4 inch	0	0	0	0	0.00
3/8 inch	0	0	0	0	0.00
#4	0.1075	0	4.5642	0	#DIV/0!
#10	0.9431	0.9214	1.141	1.141	#DIV/0!
#20	5.0602	5.3984	5.1748	5.1748	#DIV/0!
#40	7.4125	8.6013	9.1723	9.1723	#DIV/0!
#60	5.6938	6.8836	7.209	7.209	#DIV/0!
#140	12.4987	14.943	16.8689	16.8689	#DIV/0!
#230	7.2901	9.7987	7.9491	7.9491	#DIV/0!
<#230	0	0	0	0	#DIV/0!

If sample is dry, Sample Used is equal to line 4 minus line 2.
If sample is wet, Fill in 'Sample Used' line

Pan Tare	44987-4	44987-4 DU	44987-4 trip	trip w/o #4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10	Sample 11	Sample 12	Sample 13	Sample 14	Sample 15	Sample 16	Sample 17	Sample 18	Sample 19	Sample 20	Sample 21
0	6.1689	6.2764																			
0	6.009	6.9521	6.9304	7.15	7.15																
0	6.0886	11.1368	11.485	11.2614	11.2614																
0	6.049	13.4615	14.6503	15.2213	15.2213																
0	6.048	11.7418	12.9416	13.257	13.257																
0	6.0851	18.5718	21.0281	22.952	22.952																
0	5.9703	13.2604	15.769	13.9194	13.9194																

Hydrometer Reading	44987-4	44987-4 DU	44987-4 trip	trip w/o #4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10	Sample 11	Sample 12	Sample 13	Sample 14	Sample 15	Sample 16	Sample 17	Sample 18	Sample 19	Sample 20	Sample 21
2	1.008	1.0095	1.009	1.009																	
5	1.007	1.0085	1.008	1.008																	
15	1.007	1.008	1.007	1.007																	
30	1.0065	1.007	1.0065	1.0065																	
60	1.006	1.006	1.006	1.006																	
250	1.006	1.006	1.0055	1.0055																	
1440	1.005	1.0055	1.005	1.005																	

Temp at hydrometer reading	
21	
21	
21	
21	
21	
21	
21	

Soil Classification	44987-4	44987-4 DU	44987-4 trip	trip w/o #4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10	Sample 11	Sample 12	Sample 13	Sample 14	Sample 15	Sample 16	Sample 17	Sample 18	Sample 19	Sample 20	Sample 21
Percent																					
Gravel	0.2%	0.0%	7.0%	0.0%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Sand	86.2%	81.7%	73.2%	78.8%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Corse Sand	1.9%	1.6%	1.8%	1.9%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Medium Sand	37.4%	24.6%	22.1%	23.8%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Fine Sand	40.8%	55.5%	49.3%	53.1%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Silt	13.1%	10.0%	15.2%	16.0%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Clay (from 100%)	6.5%	8.3%	4.5%	5.2%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

#10 was dropped on the trip sample. 1.141g was chosen to m

A large rock was found in the Trip. The fourth sample is the l

Attachment 3. Example Particle Size Graph

44987-4

Largest Partical Size #4

Partical Size	Partical Size	Percent Finer	Incremental Percent
3 inch	75000	100.0%	0.0%
2 inch	50000	100.0%	0.0%
1.5 inch	37500	100.0%	0.0%
1 inch	25000	100.0%	0.0%
3/4 inch	19000	100.0%	0.0%
3/8 inch	9500	100.0%	0.0%
#4	4750	99.8%	0.2%
#10	2000	97.8%	1.9%
#20	850	87.4%	10.4%
#40	425	72.1%	15.3%
#60	250	60.4%	11.7%
#140	106	34.7%	25.7%
#230	63	19.6%	15.0%
Hydrometer	36	13.0%	6.7%
Hydrometer	23	9.7%	3.2%
Hydrometer	13	9.7%	0.0%
Hydrometer	9	8.1%	1.6%
Hydrometer	7	6.5%	1.6%
Hydrometer	3	6.5%	0.0%
Hydrometer	1	3.2%	3.2%

Soil Clasification Percent	44987-4
Gravel	0.2%
Sand	80.2%
Corse Sand	1.9%
Medium Sand	37.4%
Fine Sand	40.8%
Silt	13.1%
Clay (from 100%)	6.5%

Page 1