VERSION 4

UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION

NIKE ANTIAIRCRAFT MISSILE BATTERY BU-51/52 (NIKE BATTERY) LAUNCH AREA HAMBURG, ERIE COUNTY, NEW YORK FUDS PROJECT NUMBER: C02NY0079 P2 PROJECT NUMBER: 467299

SEPTEMBER 14, 2020

Contract No.: W912WJ-19-D-0004
Task Order No.: W912WJ19F0123

Prepared For:

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Introduction

I.1 The SERES-Arcadis Joint Venture (JV), Limited Liability Company (LLC)[1] (hereafter referred to as the SERES-Arcadis JV) has prepared this Quality Assurance Project Plan (QAPP) under Contract No.: W912WJ-19-D-0004, Task Order No.: W912WJ19F0123. The QAPP was prepared consistent with the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPPs) on behalf of the United States (U.S.) Army Corps of Engineers (USACE) to conduct a Comprehensive Environmental Response, Compensation, and Liability Act- (CERCLA-) compliant Remedial Investigation (RI) at the former Launch Area of the former Nike Antiaircraft Missile Battery BU-51/52 (Nike Battery), a Formerly Used Defense Site (FUDS), located in Hamburg, Erie County, New York. The USACE oversees the FUDS program. **Appendix A** contains figures showing the general site location on a regional map (**Figure 1**) and the site plan (**Figure 2**) of the former Nike Battery. As shown in the figures, the Nike Battery includes the non-contiguous former Launch Area and former Control Area. The focus of the RI effort is the former Launch Area.

I.2 This QAPP was prepared based on the following documents:

- EM 385-1-1, *Safety and Health Requirements Manual* (USACE 2014);
- United States Environmental Protection Agency (USEPA) guidance document 505-F-03-001, *Intergovernmental Data Quality Task Force, Uniform Federal Policy for Implementing Environmental Quality Systems* (2005a);
- USEPA's *Guidance for Quality Assurance Project Plans*, CIO 2106-G-05 (2012); and
- Other guidance documents as appropriate.

This QAPP was developed with the understanding that unanticipated conditions may dictate a change in the plan as currently written.

I.3 The primary RI goals for the former Nike Battery are to:

1. Assess whether a release to the environment associated with past DoD operations has occurred and, if so, what media are impacted;
2. If a release has occurred, determine the nature and extent of impacts;
3. Assess the potential risks and hazards to human health and the environment; and
4. Evaluate the site boundary based on the RI findings.

A secondary goal is to update the conceptual site model (CSM).

I.4 For the former Launch Area of the Nike Battery Site, the RI will build on existing data to assess and delineate the nature and extent of impacts in soil and groundwater. The RI will provide the data necessary to make sound decisions regarding remedial alternatives or to justify a determination of either No U.S. Department of Defense (DoD) Action Indicated or No Further Action. The RI will collect data sufficient to meet USACE and regulatory requirements and support remedial alternative decisions, if needed, for the former Nike Battery. The technical approach for the RI is designed to achieve the data quality objectives (DQOs), which are listed in **Worksheet #11**. The RI focuses on the areas where receptors (such as Town of Hamburg workers) could potentially encounter elevated levels of constituents of concern, if present,

[1] The SERES-Arcadis JV is composed of protégé firm SERES Engineering & Services, LLC (SERES) and its mentor Arcadis U.S., Inc. (Arcadis).

associated with DoD activities. Soil and groundwater sampling activities are planned as part of the RI field work.

I.5 The purpose of this QAPP is to document the planning process for collecting data, including the implementation of the quality assurance (QA) and quality control (QC) activities developed for this project. The objectives of this QAPP are to generate project data that are technically valid, legally defensible, and useful in meeting the project goals, as well as to integrate the technical and QC requirements for future investigation activities. This QAPP addresses four primary elements:

- Project management;
- Measurement and data acquisition;
- Assessment and oversight; and
- Data validation and usability.

I.6 The above elements incorporate QA/QC requirements cited within the following documents:

- USEPA *Requirements for Quality Assurance Project Plans*, USEPA QA/R-5 (2001);
- USEPA *Guidance on Systematic Planning Using the Data Quality Objectives Process*, QA/G-4 (2006);
- *Uniform Federal Policy for Quality Assurance Project Plans*, Final Version (2005);
- USEPA *Guidance on Quality Assurance Project Plans*, CIO-2106-G-05 (2012); and
- *Department of Defense Quality Systems Manual*, Version 5.3 (2019).

I.7 The optimized UFP-QAPP workbook format used for this QAPP implements the systematic planning process and was developed via collaboration between the USEPA, DoD, and the Department of Energy. In 2010, a subgroup composed of members from the participating agencies reviewed and optimized the UFP-QAPP workbook in close coordination with USEPA's 2012 update of QA/G5, *Guidance for Quality Assurance Project Plans.* The information contained in the worksheets captures the elements that would otherwise be included in related project planning documents, such as a Sampling and Analysis Plan and Field Sampling Plan. **Table I** is a crosswalk between the optimized UFP-QAPP worksheet numbers and titles and the USEPA's QAPP Guidance (2012).

I.8 The following appendices are provided to supplement the information presented in this QAPP:

Appendix A	Site Maps
Appendix B	Accident Prevention Plan
Appendix C	Human Health and Ecological Risk Assessment Work Plan
Appendix D	Project Schedule
Appendix E	Field Standard Operating Procedures
Appendix F	Field Forms
Appendix G	Laboratory Quality Manuals, Standard Operating Procedures, Certifications, and Control Limits
Appendix H	Community Air Monitoring Plan

I.9 Refer to the table of contents for the specific worksheets and appendices comprising this QAPP. In several cases, the appendices contain standalone documents; as such, reference to these documents, where applicable, is made in this QAPP.

Table I: Crosswalk: UFP-QAPP Workbook to 2106-G-05 QAPP

Worksheet Number(s)	Worksheet Title	CIO 2106-G-05 QAPP Guidance Section	
		Section	Title
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 Table	Chapter 3	QAPP Elements for Evaluating Existing Data
14, 16	Project Tasks and 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 Standard Operating Procedures	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
23	Analytical Standard Operating Procedures	2.3.4	Analytical Methods Requirements and Task Description

Worksheet Number(s)	Worksheet Title	CIO 2106-G-05 QAPP Guidance Section	
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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	Analytical Quality Control and Corrective Action
29	Project Documents and Records	2.3.8	Documentation and Records Requirements
31, 32, 33	Assessments and Corrective Action	2.4	Assessments and Data Review (Check)
		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
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Acronyms and Abbreviations

%	percent
°C	degrees Celsius
%D	percent drift
°F	degrees Fahrenheit
%R	percent recovery
µg/kg	microgram per kilogram
µg/L	microgram per liter
amsl	above mean sea level
AOC	Area of Concern
APP	accident prevention plan
ARAR	applicable or relevant and appropriate requirements
AST	aboveground storage tank
ASTM	American Society of Testing and Materials
BA	Bachelor of Arts
BCEE	Board Certified Environmental Engineer
BERA	baseline ecological risk assessment
bgs	below ground surface
BS	Bachelor of Science
BTEX	benzene, toluene, ethylbenzene and xylene
C&D	construction and demolition
CA	corrective action
CCV	continuing calibration verification
CENAE	United States Army Corps of Engineers, New England District
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CIH	Certified Industrial Hygienist
CLP	Contract Laboratory Program
cm	centimeter
COC	contaminant of concern
COPC	constituent of potential concern
COPEC	constituent of potential ecological concern
COR	Contracting Officer's Representative
CSM	conceptual site model
CSP	Certified Safety Professional
CX	Center of Expertise
DERP	Defense Environmental Restoration Program
DL	detection limit
DO	dissolved oxygen
DoD	U.S. Department of Defense
DOT	U.S. Department of Transportation
DQCR	daily quality control report
DQI	data quality indicator
DQO	data quality objective
DU	decision unit
EcoSSL	ecological soil screening level
ELAP	Environmental Laboratory Accreditation Program

256	EM	engineer manual
257	EPC	exposure point concentration
258	ESL	ecological screening level
259	ESV	ecological screening value
260	FOIL	Freedom of Information Law
261	ft	foot or feet
262	ft/sec	feet per second
263	FUDS	Formerly Used Defense Site
264	FUDSChem	Formerly Used Defense Site Chemical (database)
265	GPS	global positioning system
266	GSA	General Services Administration
267	GZA	GZA Environmental
268	HAZWOPER	Hazardous Waste Operations and Emergency Response
269	H&S	health and safety
270	HHRA	human health risk assessment
271	HMW	high molecular weight
272	HQ	hazard quotient
273	HSDB	Hazardous Substances Data Bank
274	HTRW	hazardous, toxic, and radioactive waste
275	ICAL	initial calibration
276	ICP-AES	inductively coupled plasma – atomic emission spectrometry
277	ICS	interference check sample
278	ICV	initial calibration verification
279	ID	Identification
280	Inc.	Incorporated
281	INPR	inventory project report
282	IPaC	Information for Planning and Consultation
283	ISM	incremental sampling methodology
284	JV	joint venture
285	LCS	laboratory control sample
286	LCSD	laboratory control sample duplicate
287	LEP	Licensed Environmental Professional
288	LLC	Limited Liability Company
289	LMW	low molecular weight
290	LOD	limit of detection
291	LOQ	level of quantitation
292	M&E	Metcalf & Eddy
293	MBA	Master of Business Administration
294	MCL	maximum contaminant level
295	MDL	method detection limit
296	mg/kg	milligram per kilogram
297	mg/L	milligram per liter
298	ml	milliliter
299	MPC	measurement performance criteria
300	MQO	management quality objective
301	MS	matrix spike
302	MSD	matrix spike duplicate

NA	not applicable/not available
NFA	no further action
Nike Battery	Nike Antiaircraft Missile Battery BU-51/52
NYCRR	New York Codes, Rules, and Regulations
NYS	New York State
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYSDOT	New York State Department of Transportation
ORP	oxidation-reduction potential
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbon
PAL	project action limit
PCB	polychlorinated biphenyl
PE	Professional Engineer
PFAS	per- and polyfluoroalkyl substances
PG	Professional Geologist
PID	photoionization detector
PM	Project Manager
POL	petroleum, oil, and lubricants
PPE	personal protective equipment
PQL	practical quantitation limit
PRP	Potentially Responsible Party
PVC	polyvinyl chloride
QA	quality assurance
QAPP	quality assurance project plan
QA/QC	quality assurance /quality control
QC	quality control
QSM	quality systems manual
RAGS	Risk Assessment Guidance for Superfund
RCA	root cause analysis
RI	remedial investigation
RME	reasonable maximum exposure
ROE	right of entry
RPD	relative percent difference
RQD	rock quality designation
RRT	relative retention time
RSD	relative standard deviation
RSL	regional screening level
RT	retention time
SCO	soil cleanup objective
SEDD	staged electronic data deliverable
SJB	SJB Services, Inc.
SLERA	screening level ecological risk assessment
SMDP	scientific management decision point
SOP	standard operating procedure
SSHP	Site Safety and Health Plan
SVOC	semi-volatile organic compound

SWAT	special weapons and tactics
TAL	target analyte list
TBD	to be determined
TCL	target compound list
TOC	total organic carbon
TPH	total petroleum hydrocarbon
TPP	technical project planning
UDMH	1,1-dimethylhydrazine
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
U.S.	United States
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
USCS	Unified Soil Classification System
USGS	United States Geological Survey
UST	underground storage tank
VISL	vapor intrusion screening level
VOC	volatile organic compound

QAPP Worksheets #1 & #2: Title and Approval Pages

(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

This worksheet identifies the principal points of contact for all organizations having decision authority in the project and documents their commitment to implement the UFP-QAPP. Signatories usually include the Project Manager (PM) and QA Manager, and individuals with approval or oversight authority. Signatures indicate that officials have reviewed the UFP-QAPP and concur with the implementation as written. It is the lead organization's responsibility to make sure all signatures are in place before work begins.

1. Project Identifying Information:
    a. Site name/project name: Nike Antiaircraft Missile Battery BU-51/52 Launch Area, Remedial Investigation
    b. Site location/number: Hamburg, Erie County, New York / FUDS Project Number: C02NY0079
    c. Contract/work assignment number: W912WJ-19-D-0004, Task Order W912WJ-19-F-0123
    d. Lead Organization: USACE
    e. Geographical Corps District: New England District
2. Department of Defense Organization(s):
    a. USACE, New England District (CENAE)
        i. Name: Erin Kirby, Professional Geologist (PG), Licensed Environmental Professional (LEP)
        ii. Title: USACE PM
        iii. Signature: _____ Date: _____

3. Contractor: SERES-Arcadis JV
    a. SERES-Arcadis JV PM
        i. Name: Andy Vitolins, PG
        ii. Title: SERES-Arcadis JV PM

        iii. Signature: _____ Date: _____

    b. SERES-Arcadis JV Corporate QA Manager
        i. Name: John Nocera, Professional Engineer (PE)
        ii. Corporate Quality Manager

        iii. Signature: _____ Date: _____

4. Federal Regulatory Agency: Not Applicable (NA)
5. State/Territory Regulatory Agency:
    a. New York State Department of Environmental Conservation (NYSDEC)
    b. New York State Department of Health (NYSDOH)
6. Other Stakeholders:
    a. Town of Hamburg, New York
    b. Local Residents and Businesses

410 7. Plans and reports from previous investigations and previous scoping session and dates:

Plans and Reports from Previous Investigations Relevant to this Project:	*1989*	*Final Engineering Report, Defense Environmental Restoration Act Confirmation Study and Contamination Evaluation at the Former Nike Battery Site, BU-51/52, Hamburg, New York.*
	1999	*Soil and Groundwater Quality Investigations of Lakeview Properties Bordering the Town of Hamburg Landfill and former Nike Base.*
	1999	*Results of Groundwater Sampling of Neighboring Wells.*
	2000	*Data Report, Site Investigation, Former Nike Battery BU-51/52, Hamburg, New York.*
	2000	*Screening Level Human Health Risk Assessment, Former Nike Battery BU-51/52, Hamburg, New York.*
	2003	*Memorandum for Commander, North Atlantic Division and Commander, U.S. Army Engineer District Omaha, Subject: DERP-FUDS Inventory Project Report for Site No. C02NY0079, Former Nike Battery BU-51/52, Hamburg, New York (Amended). Prepared by the Department of the Army, New York District, Corps of Engineers, New York, NY. 15 October. [FUDS Document No. C02NY007902_01.08_1008_a].*
	2003	*Nike Missile Battery Environmental Conditions Assessment Guide. Defense Environmental Restoration Program for Formerly Used Defense Sites, Final Report. Prepared by the USACE. July 2003.*
	2018	*Technical Memorandum, Subject: Records Review and Site Visit, Nike Antiaircraft Missile Battery BU-51/52, New York, Formerly Used Defense Site #C02NY0079.*
	2018	*Draft Remedial Investigation Work Plan, Nike Antiaircraft Missile Battery BU-51/52, New York, Formerly Used Defense Site #C02NY0079.*
Scoping Sessions and Dates:	USACE Kickoff Meeting: 27 August 2019 Technical Project Planning (TPP) Meeting #1: To Be Determined (TBD)	

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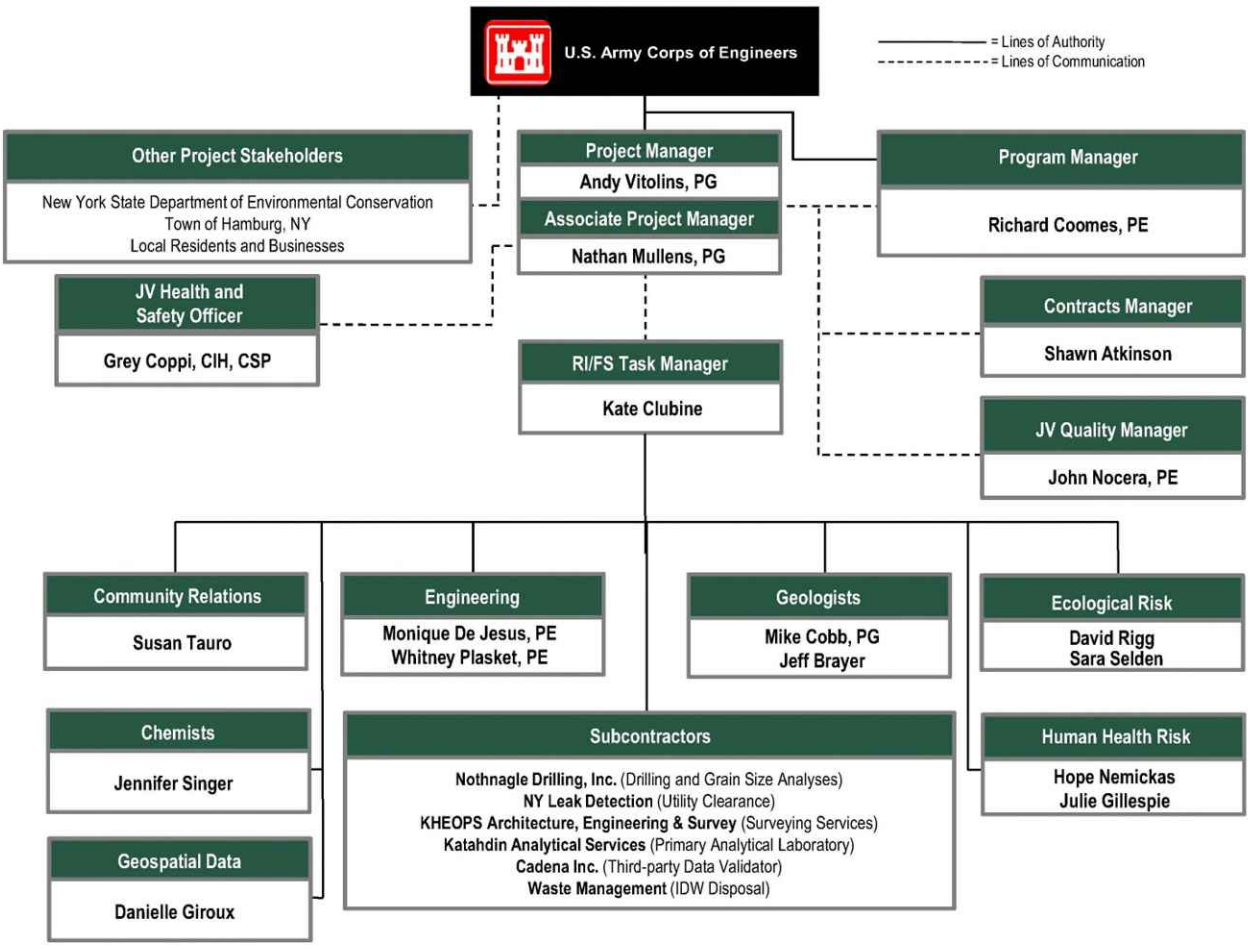
QAPP Worksheets #3 & #5: Project Organization and QAPP Distribution

(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)

This worksheet identifies key project personnel, as well as lines of authority and lines of communication among the DoD organization, prime contractor, subcontractors, and regulatory agencies. A list of QAPP recipients along with their contact information is provided in the table below.



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Project Delivery Team and QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
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Cheryl Montgomery	Ecological Risk	CENAE	978-318-8088 (Office) 781-530-8317 (Mobile)	Cheryl.R.Montgomery@usace.army.mil
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Nathan Mullens, PG	Associate PM	SERES-Arcadis JV	843-478-0366	nrmullens@seres-es.com
Shawn Atkinson	Contracts Manager	SERES-Arcadis JV	303-471-3420 (Office)	Shawn.Atkinson@arcadis.com

Project Delivery Team and QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
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Grey Coppi, Certified Industrial Hygienist (CIH), Certified Safety Professional (CSP)	Safety and Health Manager	SERES-Arcadis JV	732-225-5061 (Office) 908-917-6948 (Mobile)	grey.coppi@arcadis.com
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David Rigg	Ecological Risk Assessor	SERES-Arcadis JV	518-250-7379 (Office)	david.rigg@arcadis.com
Sara Selden	Ecological Risk Assessor	SERES-Arcadis JV	312-575-3713 (Office)	sara.selden@arcadis.com
Hope Nemickas	Human Health Risk Assessor	SERES-Arcadis JV	631-391-5237 (Office)	hope.nemickas@arcadis.com
Julie Gillespie	Human Health Risk Assessor	SERES-Arcadis JV	845-218-0749 (Office)	Julie.Gillespie@arcadis.com
Jennifer Singer	Chemist	SERES-Arcadis JV	716-667-6664 (Office)	jennifer.singer@arcadis.com
Mike Cobb, PG	Geologist	SERES-Arcadis JV	207-613-8351 (Office) 207-712-8843 (Mobile)	michael.cobb@arcadis.com
Jim Tomalia	Data Validator	Cadena, Inc.	517-819-0356	jtomalia@cadenaco.com

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QAPP Worksheets #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)

This worksheet identifies the key project personnel for the contractor performing tasks defined in this QAPP, which includes the prime contractor and laboratories.

ORGANIZATION: SERES-Arcadis JV

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications for the Project Title/Role[1]	Signature/Date[2]
Richard Coomes, PE	Program Manager	Bachelor of Science (BS), Engineering Science, Master of Science, Civil Engineering, 29 years of experience in federal and commercial environmental consulting; Program Manager for Federal IDIQ contracts, Senior Engineer or Principal on CERCLA projects including FUDS sites for USACE.	• Occupational Safety and Health Administration (OSHA) Initial 40 Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) • USACE Construction Quality Management for Contractors	Signature on file
Monique DeJesus, PE	Deputy Program Manager	BS/Master of Engineering (M Eng), Civil and Environmental Engineering. Project environmental engineer working on munitions response projects for the Army and Navy, including Active projects in California and FUDS projects in Florida.	• OSHA Initial 40 Hour HAZWOPER • OSHA 8-hour HAZWOPER Refresher	Signature on file
Andy Vitolins, PG	PM	Bachelor of Arts (BA), Geological Sciences, Master of Science, Environmental Science, 24 years of experience. Experienced in the management of numerous comprehensive investigation and remediation projects throughout New York State and the eastern United States under federal and state environmental programs.	• OSHA Initial 40-Hour HAZWOPER • OSHA 8-Hour HAZWOPER Refresher • Construction Safety Awareness and Site Supervisor Safety Training • Arcadis Certified Federal PM	Signature on file

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications for the Project Title/Role[1]	Signature/Date[2]
John Nocera, PE, BCEE	Corporate Quality Manager	Bachelor of Engineering (BE) and Master of Engineering (ME), 31 years of experience in environmental investigation and remedial design for USACE including a number of Nike Missile Silo investigations and closure designs throughout New York, New Jersey, and Pennsylvania.	• OSHA Initial 40 Hour HAZWOPER • USACE Construction Quality Management for Contractors	Signature on file
Shawn Atkinson	Federal Contracts Manager	BS, Management, Master of Business Administration (MBA), 12 years of experience managing both cost and fixed-price contracts for the DoD, civilian agencies, and federal contractors. Solid understanding of the Federal Acquisition Regulation, various contract types and vehicles, and the entire acquisition process.	• Certified Professional Contracts Manager • Previously held Defense Acquisition Workforce Improvement Act Level III certification	Signature on file
Grey Coppi, CSP, CIH	Corporate Safety Professional	BS, Health Science, Master of Science, Environmental Health Science, 25 years of facilities and environmental health and safety consulting experience.		Signature on file
Jennifer Singer	Project Chemist	BS, Biochemistry, Master of Science, Environmental Pollution Control, 21 years of project chemistry experience in coordination with the Air Force Civil Engineer Center, USACE, and USEPA.		Signature on file
Whitney Plasket, PE (Maine)	Senior Environmental Engineer	BS, Environmental Engineering, 10 years of experience, specializing in preparing site conceptual models and evaluating remedial technologies. Diverse background in both the remediation of contaminated soil and groundwater.	• OSHA Initial 40-Hour HAZWOPER • OSHA 8-Hour HAZWOPER Refresher	Signature on File

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications for the Project Title/Role[1]	Signature/Date[2]
Mike Cobb, PG	Geologist	BS/Master of Science, Geology, 15 years of professional experience with a range of geological/hydrogeologic investigations, with an emphasis in dense non-aqueous phase liquid sites, fractured rock and karst. Experience developing conceptual site models in an array of hydrogeologic settings, ranging from bedrock, and glacial terrains, to made-land waterfront settings. Has coordinated major subsurface investigations located throughout the eastern and central United States.	• OSHA Initial 40-Hour HAZWOPER • OSHA 8-Hour HAZWOPER Refresher	Signature on file
David Rigg	Ecological Risk Assessor	BS, Environmental Science; Master of Science, Environmental Modeling and Risk Analysis, 30 years of experience in aquatic and terrestrial ecology, specializing in fish and wildlife impact assessments, ecological risk assessments, biological surveys, and aquatic and terrestrial community characterization. Has developed food web models to evaluate potential uptake and assess risks to various wildlife species.	• OSHA Initial 40 Hour HAZWOPER • OSHA 8-hour HAZWOPER Refresher • United States Fish and Wildlife Service (USFWS) Electrofishing Certification	Signature on file
Sara Selden	Ecological Risk Assessor	BS, Applied Ecology and Environmental Science, 13 years of experience conducting site investigation and terrestrial and aquatic ecological risk assessments, including data analysis, conceptual site models, and wildlife exposure dose modeling.	• OSHA Initial 40 Hour HAZWOPER • OSHA 8-hour HAZWOPER Refresher	Signature on file

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications for the Project Title/Role[1]	Signature/Date[2]
Hope Nemickas	Human Health Risk Assessor	BS, Natural Resources, 23 years of experience conducting human health and ecological risk assessments; provided risk assessment expertise to U.S. Army, Air Force, and Navy sites, including FUDS projects; helped develop CSMs and risk-related input for Phase II Army Operational Range Assessments. Knowledge of conducting human health evaluations and fish and wildlife impact analyses in New York State.	• OSHA Initial 40 Hour HAZWOPER • OSHA 8-hour HAZWOPER Refresher	Signature on file
Julie Gillespie	Human Health Risk Assessor	BS, Natural Resources Management, Master of Science, Environmental Policy, 15 years of experience conducting CERCLA baseline human health risk assessments and screening-level ecological risk assessments; provided risk assessment expertise to U.S. Army, Air Force, and Navy sites, including FUDS projects. Knowledge of conducting human health evaluations and fish and wildlife impact analyses in New York State.	• OSHA Initial 40 Hour HAZWOPER • OSHA 8-hour HAZWOPER Refresher	Signature on file

Notes:
[1] Training listed is required for the project title/role only and not the personnel holding that position.
[2] Signatures indicate personnel have read and agree to implement this QAPP as written (signatures required for final submittal only).

ORGANIZATION: Katahdin Analytical Services (primary laboratory)

Name	Project Title/Role	Education/Experience	Signature/Date
Heather Manz	Laboratory PM	Representative for project laboratory.	Signature on file
Leslie Dimond	Laboratory QA Manager	Representative for project laboratory.	Signature on file

ORGANIZATION: Cadena Inc. (Data Validator)

Name	Project Title/Role	Education/Experience	Signature/Date
Jim Tomalia	Data Validation PM	BS Environmental Chemistry – Oakland University. Graduate Certificate in Hazardous Materials Management – Wayne State University. Environmental laboratory experience – 18 years. Data verification / validation and data management experience – 15 years. Software Training - Formal: FUDSChem DoD on-line tools. Equis 5 Tools and datachecker. ADR software. Cadena CLMS project management and data verification tools. CHMM certification.	Signature on file

Note:
Signatures indicate personnel have read and agree to implement this QAPP as written (signatures required for final submittal only).

PERSONNEL QUALIFICATIONS CERTIFICATION LETTER

Note: After the field schedule and personnel availability are confirmed, a letter will be submitted to the Contracting Officer for review of qualifications and approval of personnel proposed for the RI. The SERES-Arcadis JV will not be authorized to begin field work until the Contractor's personnel are accepted by the Government. The acceptance letter will be placed here when finalized.

QAPP Worksheet #6: Communication Pathways

(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

This worksheet documents specific issues (communication drivers) that will trigger the need to communicate with other project personnel or stakeholders. It ensures there are procedures in place for providing the appropriate notifications and generating the appropriate documentation when handling important communications, including those involving regulatory interfaces, unexpected events, emergencies, non-conformances, and stop-work orders. An organizational chart is included in Worksheets #3 & #5. Appendix B contains the Project Accident Prevention Plan (APP).

Communication Driver	Organization	Name/Role	Phone Number	Procedure (Timing, Pathways, Documentation, etc.)
Program issues and modifications	CENAE	Erin Kirby *CENAE PM*	978-318-8147 (Office)	The CENAE PM will communicate project management decisions and QAPP modifications to the USACE Contracting Officer's Representative (COR) and the SERES-Arcadis JV PM, as necessary. All approved QAPP modifications will be included in the amendments to the QAPP by the SERES-Arcadis JV and signed within 7 working days.
Project issues	SERES-Arcadis JV	Andy Vitolins *SERES-Arcadis JV PM*	518-250-7359 (Office) 518-461-3145 (Mobile)	The SERES-Arcadis JV PM will notify the USACE COR and PM of project issues within seven days by telephone or email.
Regulatory agency interface (*e.g.*, NYSDEC)	SERES-Arcadis JV	Andy Vitolins *SERES-Arcadis JV PM* Erin Kirby *CENAE PM*	518-250-7359 (Office) 518-461-3145 (Mobile) 978-318-8180 (Office) 508-713-3718 (Mobile)	Communication with the NYSDEC will include USACE representatives. At the direction of the USACE COR or PM, the SERES-Arcadis JV PM may communicate directly with regulatory agency representatives by phone or email. If this occurs, the USACE COR and PM will be copied on any communications and a summary of discussion points will be provided.
Field progress reports	SERES-Arcadis JV	Andy Vitolins *SERES-Arcadis JV PM*	518-250-7359 (Office) 518-461-3145 (Mobile)	The SERES-Arcadis JV PM will send weekly field status reports to the USACE COR and/or PM by email. Daily email updates will also be provided.

Communication Driver	Organization	Name/Role	Phone Number	Procedure (Timing, Pathways, Documentation, etc.)
Stop work due to safety issues	SERES-Arcadis JV	Grey Coppi, *SERES-Arcadis JV Federal Safety and Health Manager* Andy Vitolins, *SERES-Arcadis JV PM*	732-225-5061 (Office) 908-917-6948 (Mobile) 518-250-7359 (Office) 518-461-3145 (Mobile)	Anyone may stop work at any time for any safety concern. Refer to the APP included as **Appendix B** to this QAPP for specifics related to health and safety (H&S). Persons other than the responsible entity may also stop work for safety concerns. All stop work issues will be recorded in the daily QC report (DQCR). The SERES-Arcadis JV PM will notify the USACE COR and PM, by phone within 24 hours, if there is a stop work situation. Email documentation will follow within 24 hours.
QAPP changes prior to field work	SERES-Arcadis JV	Andy Vitolins, *SERES-Arcadis JV PM*	518-250-7359 (Office) 518-461-3145 (Mobile)	The SERES-Arcadis JV PM will submit documented amendments within 10 working days to the USACE COR and PM for approval.
QAPP changes during project execution	SERES-Arcadis JV	Andy Vitolins, *SERES-Arcadis JV PM*	518-250-7359 (Office) 518-461-3145 (Mobile)	The SERES-Arcadis JV PM will notify and obtain approval for QAPP modifications from the USACE COR and PM. The SERES-Arcadis JV will include all approved QAPP modifications in the amendments to the QAPP and sign them within 7 working days.
Field corrective actions	SERES-Arcadis JV	Andy Vitolins, *SERES-Arcadis JV PM*	518-250-7359 (Office) 518-461-3145 (Mobile)	The SERES-Arcadis JV PM will communicate field corrective actions within 24 hours to the USACE COR and PM by phone and email. Documentation of the corrective action will occur within seven working days.
Sample receipt variances	Katahdin Analytical Services	Heather Manz *Katahdin Analytical Services PM*	207-536-1904 (Office)	The laboratory PM will report all project field sample variance issues to the SERES-Arcadis JV Project Chemist within 24 hours of identification of the technical concern. The SERES-Arcadis JV PM will report all field sample variance issues to the USACE COR and PM within 24 hours (by phone followed by a confirming email) of notification from the laboratory.

Communication Driver	Organization	Name/Role	Phone Number	Procedure (Timing, Pathways, Documentation, etc.)
Laboratory quality control variances	Katahdin Analytical Services	Heather Manz *Katahdin Analytical Services PM*	207-536-1904 (Office)	The laboratory PM will report all QC issues with project field samples to the SERES-Arcadis JV PM and Project Chemist within 24 hours of identification of the technical concern. The SERES-Arcadis JV PM will report all QC issues with project field samples to the USACE COR and PM within 24 hours (by phone followed by a confirming email) of notification by the laboratory.
FUDSChem	SERES-Arcadis JV and	Jennifer Singer *SERES-Arcadis JV Project Chemist*	716-667-6664 (Office)	The SERES-Arcadis JV Project Chemist will oversee all FUDSChem activities including event planning, eQAPP development, lab SEDD file uploading, and data validation.
Data verification issues (for example, incomplete records)	SERES-Arcadis JV and/or Cadena Inc.	Jennifer Singer *SERES-Arcadis JV Project Chemist* Jim Tomalia *Cadena Inc. Data Validator*	716-667-6664 (Office) 517-819-0356.	The SERES-Arcadis JV Project Chemist or data validator (Cadena) will report all verification issues to the SERES-Arcadis JV PM via email within 24 hours of identification of the technical concern. The SERES-Arcadis JV PM will report verification issues to the USACE COR and PM via email within 24 hours (by phone followed by a confirming email) of notification.
Data validation issues (for example, non-compliance with procedures)	SERES-Arcadis JV and/or Cadena Inc.	Jennifer Singer SERES-Arcadis JV *Project Chemist* Jim Tomalia *Cadena Inc. Data Validator*	716-667-6664 517-819-0356.	The Cadena validator will report all validation issues to the SERES-Arcadis JV PM and Project Chemist via email within 24 hours of identification of the technical concern. The SERES-Arcadis JV PM will report all validation issues to the USACE COR and PM within 24 hours (by phone followed by a confirming email) of notification.
Data review corrective actions	SERES-Arcadis JV and/or Cadena Inc.	Jennifer Singer SERES-Arcadis JV *Project Chemist* Jim Tomalia *Cadena Inc. Data Validator*	716-667-6664 517-819-0356.	The SERES-Arcadis JV Project Chemist and/or Cadena validator, as appropriate, will determine the need for data review corrective actions and document the actions in a memorandum to the SERES-Arcadis JV PM. The SERES-Arcadis JV PM will report data review corrective actions to the USACE COR and PM within 24 hours (by phone followed by a confirming email) of notification.

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Points of Contact Information

Organization/Agency and Title/Project Function	Name	Contact Information
CENAE		
Project PM	Erin Kirby, PG, LEP	978-318-8147 (Office) Erin.Kirby@usace.army.mil
Project Geologist	Paul Young	978-318-8597 (Office) paul.j.young@usace.army.mil
Project Chemist	Yixian Zhang	978-318-8730 (Office) yixian.zhang@usace.army.mil
Project Ecological Risk	Cheryl Montgomery	978-318-8088 (Office) Cheryl.R.Montgomery@usace.army.mil
Project Human Health Risk	Cynthia Auld	978-318-8042 (Office) cynthia.a.auld@usace.army.mil
Town of Hamburg		
Town Engineer	Mike Quinn	716-392-1758 (Office) michael.quinn@ghd.com
Highway Superintendent	Ted Casey	716-649-7700 (Office) TedCasey@townofhamburgny.com
Deputy Highway Superintendent	Darren Smith	TBD
NYSDEC		
PM	Melissa Sweet	518-402-9614 (Office) melissa.sweet@dec.ny.gov
Section Chief	John Swartwout	518-402-9620 (Office) john.swartwout@dec.ny.gov
NYSDOH		
Public Health Specialist 3	Jacquelyn Nealon	518-402-7860 (Office) Jacquelyn.nealon@health.ny.gov
SERES-Arcadis JV		
Program Manager	Richard Coomes, PE	502-777-3734 (Office) racoomes@seres-es.com
Deputy Program Manager	Monique DeJesus, PE	781-213-4922 (Office) 510-449-8250 (Mobile) monique.dejesus@arcadis.com
PM	Andy Vitolins, PG	518-250-7359 (Office) 518-461-3145 (Mobile) andy.vitolins@arcadis.com
Associate PM	Nathan Mullens, PG	843-478-0366 (Office) nrmullens@seres-es.com

Organization/Agency and Title/Project Function	Name	Contact Information
Contracts Manager	Shawn Atkinson	303-471-3420 (Office) Shawn.Atkinson@arcadis.com
Corporate Quality Manager	John Nocera, PE, BCEE	251-405-4560 (Office) 251-689-7760 (Mobile) john.nocera@arcadis.com
Safety & Health Manager	Grey Coppi, CIH, CSP	732-225-5061 (Office) 908-917-6948 (mobile) grey.coppi@arcadis.com
Senior Environmental Engineer	Whitney Plasket, PE	207-613-8447 (Office) whitney.plasket@arcadis.com
Ecological Risk Assessor	David Rigg	518-250-7379 (Office) david.rigg@arcadis.com
Ecological Risk Assessor	Sara Selden	312-575-3713 (Office) sara.selden@arcadis.com
Human Health Risk Assessor	Hope Nemickas	631-391-5237 (Office) hope.nemickas@arcadis.com
Human Health Risk Assessor	Julie Gillespie	845-218-0749 (Office) julie.gillespie@arcadis.com
Chemist	Jennifer Singer	716-667-6664 (Office) jennifer.singer@arcadis.com
Geologist	Mike Cobb, PG	207-613-8351 (Office) 207-712-8843 (Mobile) michael.cobb@arcadis.com
Cadena, Inc.		
Data Validator	Jim Tomalia	517-819-0356 jtomalia@cadenaco.com
EMERGENCY NUMBERS		
Town of Hamburg Police Department	Not applicable (N/A)	911 (716) 648-5111 (non-emergency)
Erie County Sheriff's Department	N/A	911 (716) 858-2903 (non-emergency)
Fire Department	N/A	911
Ambulance Service	N/A	911
Work Care (SERES-Arcadis JV)	N/A	800-455-6155

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QAPP Worksheet #9: Project Planning Session Summary

(UFP-QAPP Manual Section 2.5.1 and Figures 9-12)
(EPA 2106-G-05 Section 2.2.5)

A Kickoff meeting was held for the RI for the former Launch Site of the Nike Battery BU-51/52 at the USACE New England District Office in Concord, Massachusetts on 27 August 2019. The purpose of the meeting was to:

- Confirm project stakeholders;
- Discuss communication tools and protocols;
- Review the RI objectives;
- Discuss the project schedule;
- Review site information and data gaps; and
- Plan a site visit.

A list of attendees and meeting minutes are provided below.

Technical project planning sessions may be scheduled, as needed, as the project progresses.

Meeting Minutes
Nike BU 51/52 RI-DD
Contract W912WJ-19-D-0004; Task Order W912WJ-19-F-0123
August 27, 2019 @ 0900 EDT
USACE NAE Office, Concord, MA

USACE New England:	Erin Kirby – Technical Lead
	Rose Schmidt – Geologist
	Cheryl Montgomery – Ecological Risk Assessor
	Cindy Auld – Human Health Risk Assessor
SERES-Arcadis JV:	Alex Lo – JV Managing Partner
	Jennifer Mayers – JV Programmatic Support
	Jeffery Smoak – JV Programmatic Support
	Andy Vitolins – Project Manager
	Nathan Mullens – Deputy Project Manager
Absent:	Yixian Zhang (USACE NAE Chemist)

Communication

The meeting commenced with Erin Kirby leading introductions of the USACE NAE and SERES-Arcadis JV team. Ms. Kirby discussed the proper procedures for communicating both internally with the project team and externally with other stakeholders such as property owners and regulators. Ms. Kirby stated that she preferred to make initial contact with these stakeholders, but subsequent communications could be initiated by SERES-Arcadis JV team members in the future. She mentioned Mike Quinn is the Town Engineer and John Swartwout out of Albany will be the NYSDEC project manager. The ROE has been executed and she will send to the SERES-Arcadis JV.

Invoicing

Ms. Kirby requested draft invoices be sent to her for review. She would review the drafts within 1 to 2 days. Milestones will need to be established for the project. She would allow monthly billing of Project Management time, but monthly billing was not required. Invoicing frequency was at the discretion of the SERES-Arcadis JV. Invoices under $500 are not preferred. The monthly report must state if no invoice would be submitted during that period.

Project Schedule

Ms. Kirby expressed the critical nature of the project schedule. She requested a two-month schedule outlook with milestones clearly identified. Along with this, the SERES-Arcadis JV needs to setup Microsoft Outlook appointments for when milestones/deadlines will occur and add reminders to those items that occur four days prior. This will help the USACE team stay on schedule with items such as document reviews. The USACE NAE team would be performing document reviews by the PDT, ITR and

likely the USACE Centers of Expertise (CX). Ms. Kirby also stated that correspondence with the CX needed to come from her. She requested updates via phone instead of solely by email especially if there are issues with the project. Her cell phone is: 603-365-6036. Ms. Kirby requested the SERES-Arcadis JV setup recurring project status calls. The SERES-Arcadis JV will only invite her to the calls at this stage of the project. The first call will be September 3 at 1200. The following calls will be at 0900. Lastly, Ms. Kirby requested planning documents be submitted to her team as they are completed instead of completing all planning documents before submitting any. Documents should be submitted in both Microsoft Word and Adobe PDF formats.

Technical Presentation of Site Background and Data Gaps

Next on the agenda was the technical presentation prepared and presented by the SERES-Arcadis JV. Andy Vitolins presented 21 slides reviewing Site Background including historic and current site use, sources of contamination, and historic site investigations including a human health risk assessment (HHRA) performed in 2000.

Subsequent slides illustrated former soil and groundwater sample results from three separate investigations. One investigation occurred in 1989 and two in 1999. Primary Contaminants of Concern (COCs) indicated by these investigations were arsenic and chromium in surface soil, arsenic, chromium, TPH and hydrazine in subsurface soils, and VOCs, SVOCs, metals in groundwater.

Later slides identified data gaps which lead into objectives for the project. Mr. Vitolins explained the sequence of proposed field efforts that would address the data gaps and then walked through the early stages of the project schedule.

Items noted during the discussion are listed below:

- The FUDS program is CERCLA led, but NAE will seek concurrence from the NYSDEC and will give them a forum for discussion of their concerns.
- A challenge will be source differentiation, particularly with the landfill.
- Emerging contaminants (PFAS and 1,4-dioxane) were discussed. Sampling for these analytes is not planned at this time.
- Selection of an appropriate background sampling location will be critical due to the activities that have taken place on the site since the Nike facility was decommissioned.
- NYSDEC regulatory levels can be listed in data tables for information purposes, but RSLs will be driver for decision-making.
- The last risk assessment did not address drinking water but did include a model that was useful.
- A TPP meeting may be held in Albany or Hamburg. Ms. Kirby will discuss the need with NYSDEC.

Following the slide presentation, Ms. Auld noted that the prior Risk Assessment focused on Maximum Contaminant Levels (MCL) and not Regional Screening Levels (RSL). She noted that any risk assessments that were part of this project would need to meet RSLs not MCLs. This led into a brief discussion of other SERES-Arcadis JV team members, after which, Ms. Kirby requested resumes of those personnel.

Ms. Schmidt indicated that the current topographic data available for the site was not high quality which was unfortunate because there were drainage and other ground surface features that impact water movement. She mentioned groundwater flow direction needs to be clearly defined. It was unlikely flow direction was uniform due to mitigation ponds and other features including the wetlands that lie southwest of the landfill and the landfill itself. Ms. Montgomery added that the SERES-Arcadis JV needs to be cognizant of the public's view of surface waters which typically are the highest visibility to the public regardless of the potential connectivity to DoD-related chemicals. Ms. Schmidt later mentioned that the Army maintains an Army Geospatial Center (AGC) that could provide the project team with higher quality topographic data.

USACE NAE mentioned the request has been made for this data but the data was not expected until January 2020.

The USACE NAE team suggested that the Work Plan mention incremental sampling may occur on the site in the future as a second phase but currently, there are too many data gaps to adequately identify decision units (DUs). The Work Plan also needs to be clear that the SERES-Arcadis JV will be collecting biased, discrete samples based on field screening.

Ms. Montgomery recommended revising the Conceptual Site Model (CSM) as the project progressed. As data is collected, exposure pathways could be amended and likely eliminated. The CSM needs to show all receptor categories and then the SERES-Arcadis JV needs to explain why each is part of this project or why it is not. Both human health and ecological CSMs need to be included in the Work Plan and they need to be comprehensive. Although ecological risk may not be carried forward through the RI, the Work Plan needs to provide a detailed discussion. This includes a T&E inventory.

FUDSCHEM and the eQAPP was discussed. It is important to start early and ensure the lab understands the requirements. The SERES-Arcadis JV has selected Katahdin as the lab, which USACE has worked with before.

Site Visit

The USACE NAE team also mentioned that the Site Walk, scheduled for September 10, would be heavily attended. The New York Department of Health (NYDOH) was expected to attend and it was anticipated that they would inquire about addressing Vapor Intrusion. Following the Site Walk, Ms. Kirby asked that the JV coordinate with property stakeholders for future site visits. Ms. Kirby provided existing access agreements for the site. At the site walk, Ms. Kirby suggested gauging attendee's interest in a Restoration Advisory Board and a project website. A repository has not yet been set up. A location can be discussed on the site visit.

The importance of Health and Safety was discussed and all parties agreed that it is. A SSHP is needed prior to the site visit allowing time for review.

585 **Action Items**

586 The following action items were discussed prior to ending the meeting.

Action Item	Assigned To	Scheduled Completion Date
1. USACE will provide the executed ROE to the SERES-Arcadis JV team	Erin Kirby	Completed 8/27/19
2. Provide 2-month Microsoft Outlook schedule with milestones - Use Outlook appointments with multiple day alerts before 4 days before they due	Andy Vitolins	9/5/19
3. Setup recurring Project Status Calls	Andy Vitolins	Completed 8/30/19
4. Submit SERES-Arcadis JV Project Team Resumes to Ms. Kirby	Andy Vitolins	9/5/19
5. Submit a Site-Specific Health and Safety Plan along with Activity Hazard Analyses (AHA) for the Site Walk	Nathan Mullens	9/5/19
6. Email the USACE NAE a list of Site Walk attendees	Andy Vitolins	9/5/19

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QAPP Worksheet #10: Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

10.1 Site Background

During the Cold War, the U.S. Army developed the Nike anti-aircraft missile to protect population centers and important industrial and military installations (Hamburg Historical Society 2019). The Nike Anti-aircraft Missile Battery BU-51/52 (Nike Battery), located in Hamburg, New York, operated from 1956 until its closure in 1961 (USACE 1991). During that period, the former battery consisted of a Launch Area, a Control Area, and an Easement Area. The Launch Area contained the facilities and equipment required to assemble, test, and maintain the missiles and launchers. The Control Area contained the electronic equipment for target tracking, missile guidance, and missile fire control. In 1968, a 57-acre plot, which contained the former Nike BU-51/52 Battery area, was turned over to the Town of Hamburg, which is the current owner of the Nike Battery (USACE 1991). The former Launch Area is currently used by the Town of Hamburg's Highway Department for office space, truck garage space, and storage of gravel piles and road salt. The Town of Hamburg police department uses the former enlisted men's barracks along the southwestern property boundary for the special weapons and tactics (SWAT) team and a firearm training center with earthen backstop berm in the northwestern corner of the former Launch Area for target practice. The former Control Area and Easement Area are currently used as the Town of Hamburg's Lakeview Recreational Area, including a playground, sledding hill, ball fields, and a BMX track (Bluestone 2019). A closed and capped landfill, formerly operated by the Town, is located adjacent to the former Launch Area.

Based on previous studies, the operation of the Nike BU-51/52 Battery from 1956 through 1961 resulted in historical impacts to the ground and subsurface. The Defense Environmental Restoration Program (DERP), which is managed by the USACE, has identified the Launch Area as a FUDS Area of Concern (AOC) (USACE 1991). The AOC was selected based on review of information contained in the FUDS Site Management Information System, USACE Investigation Project Reports, and Technical Project Planning meetings. The FUDS-eligible hazardous, toxic, and radioactive waste (HTRW) project consists of the former Launch Area. The former Control Area and Easement Area are not the subject of current investigations.

The DERP was established to address contamination caused by DoD activities on sites no longer owned by the DoD as of October 17, 1986. The DoD conducts environmental restorations in accordance with the CERCLA, also known as Superfund. As part of the Superfund process, the RI phase determines the nature and extent of impacts at the former Launch Area.

10.1.1 Site Location

The Nike Battery is located on Lakeview Road in Hamburg, New York **(Appendix A: Figures 1 and 2)**. The former Launch Area is bound on the north by Interstate I-90; on the west by the Easement Area; on the east by residential areas; and on the south by residential areas, Lakeview Road, and Eighteen Mile Creek. The closest residential housing is located approximately 500 feet to the south, on Lakeview Road.

10.1.2 Site History

Between December 1955 and May 1956, the DoD acquired approximately 57 acres by various deeds and condemnation proceedings, and 95 acres in easements to develop the Nike Battery. On June 1, 1965, DoD transferred the property to the General Services Administration (GSA) (USACE 1991). In 1968, GSA conveyed the 57 acres to the Town of Hamburg, New York (USACE 1991). The former Nike Battery was composed of the former Launch Area, the former Control Area, and Easement Area, as shown on **Appendix A: Figure 2**.

The former Launch Area, the focus of this QAPP, is currently used by the Town of Hamburg's Highway Department for office space, truck garage space, and storage of gravel piles and road salt. A closed and capped landfill, formerly operated by the Town, is located north of and adjacent to the former Launch Area. (**Appendix A: Figure 2)** provides historical site features of the former Launch Area from the period of DoD ownership. **Appendix A: Figure 3** provides current features of the Nike Battery under ownership of the Town of Hamburg. The former Launch Area is the FUDS-eligible HTRW project.

The Control Area generally included equipment for target tracking and missile guidance. The Control Area was formerly investigated and remediated before the current use. The purpose of the Easement Area was to maintain line of sight distance between the Control Area and Launch Area. Historical activities resulting in environmental contamination of the Easement Area are therefore unlikely. The Control Area and Easement Area are located northeast of the former Launch Area and are currently used as the Town of Hamburg's Lakeview Recreational Area including a playground, sledding hill, ball fields and a BMX track (Bluestone 2019). Neither the Control Area nor the Easement Area are FUDS-eligible.

The former Launch Area contained a total of 60 Nike Ajax supersonic missiles, which were stored horizontally in six underground silos (also known as magazines) (Hamburg Historical Society 1991). Above ground, there were four launchers per silo. The missiles were brought to the surface by means of an elevator and then attached, via a system of rails, to a launcher (Hamburg Historical Society 2019). The aboveground components of the silos have been demolished, and the subsurface elements of the silos were recently filled with asphalt road filings by the Town, per a site visit with Darren Smith, the Deputy Highway Superintendent on 10 September 2019.

The DoD used a former sewage treatment plant, which was located on the southern side of North Creek Road (**Appendix A: Figure 2**). According to an internal USACE memo dated 27 March 2000, the Town of Hamburg never used this facility. Information regarding the abandonment or demolition of the sewage treatment plant was not found among the historical documents. The inventory project report (INPR), dated 01 February 1991, states that the Town requested it be demolished, and that there was no evidence of hazardous/toxic waste, unsafe debris, or unexpected ordinances remaining from DOD use. However, during utility location in July 2020, sewage treatment infrastructure was determined to be in place, and fresh sewage was observed. On July 31, 2020, JV confirmed with Rick Nowak of the Town Buildings and Grounds Department that the treatment system is active and has been used since the Town acquired the property from the DoD. As such, is not possible to distinguish impacts from the DoD versus the Town, and the sewage treatment plant is not assessed in this QAPP.

The Town of Hamburg began operating the Hamburg Landfill located north of the former Launch Area in 1970, on land transferred to the Town from GSA in 1968 (USACE 1991). The landfill is not FUDS-eligible since the landfill is outside of the Nike Battery boundaries and was constructed and operated by the Town of

Hamburg after the Nike Battery closure. The Hamburg Landfill is identified as Site 915097 in the State of New York's Superfund Program. The landfill was permitted "under Part 360 for trash, rubbish, construction and demolition (C&D) material, and other non-putrescible, non-industrial wastes" (NYSDEC 2017). Empty and partially empty drums may have been illegally dumped at the landfill, some of which contained potentially hazardous wastes (NUS 1987). However, those drums were removed from the landfill site and properly disposed (NYSDEC 2017). The landfill was closed in 1984 and covered with approximately 2 feet of soil. According to NYSDEC, the landfill is not a health risk for residents because access is restricted; however, Town of Hamburg employees could be exposed to contaminated groundwater and soil if the landfill is disturbed. Contaminated groundwater does not impact drinking water wells near the site (NYSDEC 2017). Improvements were made to the landfill cap in 2008 (GZA 2018).

10.2 Known or Suspected DoD Related Chemicals or Classes of Chemicals

Solvents, corrosion preventives, and petroleum, oil, and lubricants (POL) were commonly used during the operations of a typical Nike Missile Battery Launch Area and may have been used in and around these buildings. Table 10.2, below, summarizes the potential sources of environmental contamination and associated classes of constituents based on historical DoD activities at the former Launch Area.

Table 10.2: Potential Sources and Classes of Constituents

Potential Source Products [1]	Potential Source Historical Infrastructure [2]	Associated Classes of Constituents [3]
• Missile components • Liquid fuels • Oils (hydraulic & motor) • Chlorinated solvents • Missile starter fluid • Rocket propellants • Paint	• Missile assembly and test building • Drum storage area • Generator building • Acid storage shed • Acid fueling stations • Acid neutralization pits • Missile silos	• VOCs • SVOCs • Hydrazine • Polychlorinated biphenyls (PCBs) • Petroleum compounds • Metals

Notes:

1. Based on general historical disposal practices across Nike sites, as documented in historical interviews with former Nike workers and Nike operation manuals.
2. As shown on **Appendix A: Figure 3**.
3. As identified by previous investigations and assessments.

10.2.1 Previous Site Investigations

USACE, New York District, conducted a site survey of the Nike Battery to assess the presence of unsafe debris, hazardous waste impacts and unexploded ordnance and completed an INPR (USACE 1991). An amendment to the INPR was issued in October 2003, recommending a Potentially Responsible Party (PRP)/ /HTRW project to "investigate the use of the underground storage tanks (USTs) at the property [within the Control Area] subsequent to DoD ownership…" Subsequently, the USTs were removed; all required cleanup was completed, and the HTRW project closed. Currently, the former Launch Area, used by DoD for

missile storage, assembly, testing, and maintenance from 1956 to 1965, is the only HTRW FUDS-eligible area of concern at the former Nike Battery (USACE 1991).

Three main field investigations have been conducted to date at the former Launch Area (Metcalf & Eddy [M&E] 1989; GZA Environmental 1999; Battelle 2000a). These investigations and their findings are summarized in Bluestone 2019. Section 10.2.2 summarizes the comparison of analytical results for soil and groundwater samples to regulatory benchmarks.

In 1999, the NYSDOH sampled nine residential sumps and wells near the former Launch Area (NYSDOH 1999). Samples were analyzed for VOCs, SVOCs, and metals. Some VOC and metal concentrations were detected above regulatory limits or applicable screening levels at the Lakeview residences of Dr. Donald Hellert and Mr. Robert Martin. Groundwater VOC concentrations were above regulatory limits or applicable screening levels at sample location BW-1. Groundwater metal concentrations were above regulatory limits or applicable screening levels at sample locations BW-1 (Robert Martin property) and DW-1 (Donald Hellert property). The owners of the property contacted a hydrogeologist consultant to investigate soil and water quality on these two Lakeview Road properties (Hellert 1999). Results of the groundwater analysis indicated the presence of ethylbenzene and xylenes, 2-butanone, and acetone above NYSDEC Class GA criteria in groundwater. Metals detected in groundwater above NYSDEC criteria included arsenic, beryllium, cadmium, chromium, lead, nickel, and zinc. The two surface soil samples had arsenic, beryllium, cadmium, chromium, lead, nickel, and zinc concentrations above the NYSDEC criteria for soil.

The Human Health Risk Assessment performed by Battelle (2000b) evaluated potential risks to Lakeview Road residents south of the former Launch Area from exposure to DoD-related chemicals in soil, groundwater, and sump water. Results of five independent investigations previously conducted by the State of New York and independent entities were used for the public health risk assessment. Constituents of potential concern (COPCs) in soil included cadmium, nickel, thallium, zinc, hydrazine, and TPH. All volatile chemicals detected in soil, groundwater, and sump water were retained as COPCs for the vapor migration to indoor air evaluation; only the adult indoor air exposure through vapor intrusion was evaluated (e.g., inhalation of indoor air for children and shower inhalation were not evaluated). Total cancer risks for residential exposures to soil (dermal contact, incidental ingestion, and vegetable consumption), based on the maximum and average detected concentrations, were less than the USEPA's acceptable risk range of 1×10^{-6} to 1×10^{-4}, and non-cancer hazard indices were below 1. Inhalation risks and hazards for adult residential vapor intrusion exposure to volatile constituents in soil, groundwater and sump water were also less than 1×10^{-6} and 1, respectively. The assessment concluded that the risks and hazards were within acceptable ranges, indicating adverse health effects to residents near the former Launch Area were unlikely.

In 1999, the NYSDEC opened spill number 987548 due to sheen observed in soil during excavation of two USTs from the Nike Battery by the Town of Hamburg. The Town removed a 550-gallon UST from the former Launch Area and an 8,000- to 9,000-gallon UST from the former Control Area (GZA 1999). Both USTs historically contained petroleum products, and petroleum-impacted soil from the excavation area was disposed offsite. In 2003, four additional USTs were removed from the former Control Area (two 2,000-gallon USTs and two 4,000-gallon USTs) under NYSDEC spill number 0375052. Petroleum impacted soils from the excavation area and the USTs were disposed off site (USACE 2003).

10.2.2 Evaluation of Known or Suspected DoD Related Chemicals or Classes of Chemicals

Existing soil data were compared to the USEPA RSLs for residential soil. The RSLs are based on a target cancer risk of 1×10^{-6} or a target non-cancer hazard quotient (HQ) of 0.1. Existing soil data were also compared to the NYSDEC 6 New York Codes, Rules, and Regulations (NYCRR) Part 375 residential soil cleanup objectives (SCOs) for informational purposes. The surface soil data were also compared to ecological screening values (ESVs) for soil using the following hierarchy:

- USEPA Ecological Soil Screening levels (EcoSSLs; USEPA 2007);
- USEPA Region 4 Ecological Soil Screening Values (USEPA 2018); and
- Department of Energy Laboratories i.e., Los Alamos National Laboratory (LANL 2017).

Existing groundwater data were compared to the USEPA tapwater RSLs, based on a target cancer risk of 1×10^{-6} or a target non-cancer HQ of 0.1. The existing groundwater data were also compared to the Safe Drinking Water Act MCLs and Class GA Standards provided in NYSDEC Technical Operational Guidance Series Memorandum 1.1.1 for informational purposes (NYSDEC 1998).

The DoD-related chemicals with detected concentrations above screening levels and regulatory benchmarks are identified in the tables below. Separate columns were used for the various benchmarks to clearly indicate which were exceeded. Only the DoD-related chemicals identified as exceeding the benchmarks are included; additional DoD-related chemicals were analyzed and are not shown. A full list of the constituents being sampled for during the RI is included in **Worksheet #15**.

Table 10.2.2(a): Constituents with Soil Concentrations Exceeding Regulatory Benchmarks

Constituent [1]	Soil (2 feet bgs) (M&E 1989)		Surface Soil (0 to 1 feet bgs) (GZA 1999)			Subsurface Soil (approximately 4 feet bgs) (GZA 1999)	Subsurface Soil (> 2 feet bgs) (Battelle 2000a)
	USEPA Residential Soil RSLs	Ecological ESVs [2]	USEPA Residential Soil RSLs	Part 375 Residential SCOs	Ecological ESVs [2]	USEPA Residential Soil RSLs	USEPA Residential Soil RSLs
	Semi-Volatile Organic Compounds						
HMW PAHs					✓ a		
TPH							✓
Hydrazine							✓
	Inorganics						
Aluminum			✓			✓	✓
Antimony			✓		✓ a	✓	
Arsenic	✓		✓			✓	✓
Cadmium		✓ a			✓ a		
Chromium	✓		✓			✓	✓
Cobalt			✓			✓	✓
Copper					✓ a		
Iron			✓			✓	✓
Lead		✓ a			✓ a		
Manganese			✓		✓ a	✓	✓
Mercury					✓ b		
Nickel							
Selenium		✓ a	✓	✓	✓ a		
Thallium			✓		✓ b		✓
Vanadium					✓ a		
Zinc					✓ b		

Notes:

✓ – Maximum detected concentration exceeds benchmark.

> – greater than

feet bgs – feet below ground surface

HMW PAHs – high molecular weight polycyclic aromatic hydrocarbons

[1] Only the DoD-related chemicals identified as exceeding regulatory benchmarks are included in Table 10.2.2(a). Additional DoD-related chemicals were analyzed, as noted in the historical reports and summarized below:

- M&E 1989: VOCs, total recoverable metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium and silver).
- GZA 1999: Phase 1 surface soil sample analyses included TCL VOCs/SVOCs/PCBs/pesticides, hydrazine, and TAL metals plus cyanide. Phase 2 surface soil samples were analyzed for only arsenic at the request of the NYSDOH. Subsurface soil sample analyses included TCL VOCs/SVOCs/PCBs/pesticides, hydrazine, and TAL metals plus cyanide.
- Battelle 2000a: VOCs, SVOCs, TPH, metals, cyanide, pesticides, hydrazine, and nitrate/nitrites.

[2] USEPA EcoSSLs [a] and USEPA Region 4 (2018) ESVs [b].

Table 10.2.2(b): Constituents with Groundwater Concentrations Exceeding Regulatory Benchmarks

Constituent [1]	Groundwater (M&E 1989)			Groundwater (GZA 1999)			Groundwater (Battelle) 2000a		
	USEPA Tapwater RSL	MCL	NYSDEC Class GA Limits	USEPA Tapwater RSL	MCL	NYSDEC Class GA Limits	USEPA Tapwater RSLs	MCL	NYSDEC Class GA Limits
Volatile Organic Compounds									
Acetone						✓			
Chloroform							✓		
Benzene				✓	✓	✓	✓	✓	✓
Ethylbenzene				✓			✓		
Toluene						✓			✓
m-Xylene						✓			
p-Xylene						✓			
o-Xylene				✓		✓			
Semi-Volatile Organic Compounds									
bis(2-ethylhexylphthalate)				✓		✓	✓		✓
Benzo(a)pyrene							✓	✓	
Benzo(b)fluoranthene			✓						
Chrysene			✓						
Dibenzofuran			✓			✓			✓
Fluoranthene									✓
Indeno(1,2,3-cd)pyrene							✓		
Inorganics									
Aluminum			✓	✓		✓	✓		
Arsenic	✓	✓							
Barium	✓	✓					✓		
Beryllium									✓
Cadmium	✓	✓		✓	✓				
Chromium	✓	✓		✓					
Cobalt							✓		
Copper			✓				✓		
Iron			✓	✓		✓	✓		✓
Lead	✓	✓							
Magnesium			✓			✓			✓
Manganese			✓	✓		✓	✓		✓
Mercury	✓	✓							
Nickel				✓			✓		
Silver				✓					
Sodium			✓			✓			✓
Thallium							✓		
Cyanide				✓			✓		

Notes:

✓ – Maximum detected concentration exceeds regulatory benchmark.

[1] Only the DoD-related chemicals identified as exceeding regulatory benchmarks are included in Table 10.2.2(b). Additional DoD-related chemicals were analyzed, as noted in the historical reports and summarized below:

- M&E 1989: VOCs, petroleum hydrocarbons, total recoverable metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium and silver).
- GZA 1999: TCL VOCs/SVOCs/PCBs/pesticides, hydrazine, nitrates, and TAL metals plus cyanide.
- Battelle 2000a: VOCs, SVOCs (full scan and SIM), TPH, TAL-metals and cyanide, nitrate/nitrite, hydrazine, and metals.

10.2.3 Primary Release Mechanism / Fate and Transport Consideration

Source Area/Media:

DoD-related impacts are potentially present in the surface and subsurface soil, and groundwater at the former Launch Area.

Release Mechanism/Activity:

A variety of metals have been detected above comparison criteria in the environmental media at the former Launch Area (Bluestone 2019). Historical DoD activities may be the source of detected chromium and lead concentrations (M&E 1989). Chromium is a constituent of cleaning reagents that are used to remove corrosion from metal. Chromium and lead are both constituents of paint that was used on military installations. However, the specific source(s) of the arsenic, barium, and cadmium at the former Launch Area has not been clearly identified (M&E 1989).

Metals such as arsenic, cadmium, chromium, lead, manganese, mercury, nickel, and zinc occur naturally in the earth's crust. The landfill may also be a potential upgradient contributor of metals to Nike Battery groundwater. The desorption and mobility of metals is dependent on the solubility of the individual chemical. In soil, chromium VI has high oxidizing potential, solubility, and mobility and chromium III has low solubility and low mobility (Hazardous Substances Data Bank [HSDB] 2016a). Lead compounds have limited mobility when released or deposited on soil (HSDB 2016b). Zinc is strongly adsorbed to soil at a pH of 5 or greater, thus is not very mobile.

VOCs (primarily benzene, toluene, ethylbenzene and xylene [BTEX] compounds) were detected in groundwater at the former Launch Area (Bluestone 2019). Their presence may be due to historical releases from fuel tanks now removed. When released to water, the VOCs detected at the former Launch Area are unlikely to adsorb to sediment or suspended solids; thus, they are less likely to persist in the environment. BTEX compounds are very water soluble, resulting in high mobility.

Various PAHs have been detected in former Launch Area soils and groundwater (Bluestone 2019). PAHs may be present due in part to petroleum residuals related to UST or aboveground storage tank (AST) leaks, fueling spills, or spraying motor oil as a dust suppressant, which although it was a common practice, cannot be confirmed for this site. Although this investigation focuses on potential environmental impacts attributed to DoD activities, there may also have been PAHs introduced to the former Launch Area when the missile silo vaults caps were removed and the silos were filled with asphalt millings by the Town of Hamburg in approximately 2001 (per the site visit with Darren Smith, the Deputy Highway Superintendent on 10 September 2019). This investigation will, to the extent possible, differentiate potential contributions to environmental impacts from non-DoD activities, such as the landfill and asphalt millings in the silos. PAHs tend to sorb to soil, sediment, and other organic materials. PAHs are released to the environment naturally (e.g., forest fires) and anthropogenically (e.g., fuel combustion and waste incineration). Both high molecular weight (HMW) PAHs, composed of four or more benzene rings, and low molecular weight (LMW) PAHs were detected on the Nike Battery. HMW PAHs are less water soluble than LMW PAHs, and more likely to adsorb to suspended particles. Although likely to bind to soil particles, PAHs could migrate via advective transport (bulk movement) or desorption (leaching) with the flow of surface water or groundwater toward adjacent water bodies.

Hydrazine is a common component of rocket fuel. UDMH (1,1-dimethylhydrazine) was used as a starting fluid for Ajax missiles, which were housed in the silos at the former Launch Area. Hydrazine compounds have very high mobility in soil but degrade due to biotic and abiotic processes. The degradation of hydrazines is also more rapid in soil than in water. When released to water, hydrazines are not expected to adsorb to solids or sediment and are unlikely to bioaccumulate in aquatic organisms.

Before being banned in the United States in 1978, PCBs were used as dielectric fluids in transformers and capacitors, in lubricating and cutting oils, and as plasticizers in paints, caulks, and adhesives. Oils containing PCBs may have been used in the transformer located on the pad adjacent to the Generator Building (Bluestone 2019) (see **Appendix A: Figure 3**). PCBs were not detected in soil or groundwater samples collected during previous site investigations; however, sampling was not focused around the former transformer pad (Bluestone 2019). PCBs are relatively insoluble and persistent in the environment.

10.3 Potential Receptors and Exposure Pathways

Appendix C provides the Human Health and Ecological Risk Assessment Work Plan, which presents the approach and methodology for performing the risk assessments that will be conducted to support the RI. The work plan contains detailed human health and ecological CSMs for the former Launch Area; the human health CSM is reproduced in **Section 10.3.1**, below. The CSMs are based on the current and most likely future land use of the former Launch Area and identify potential receptors and the pathways through which exposure to DoD-related chemicals could occur. The human health and ecological CSMs may be revised based on information collected during the RI. A preliminary understanding of potential receptors and exposure pathways is presented below.

10.3.1 Human Receptors and Exposure Pathways

The current and foreseeable future land use for the former Launch Area is industrial. The property is owned by the Town of Hamburg and utilized by the Highway Department for storage, office space, and maintenance. The Town of Hamburg police department uses the former enlisted men's barracks along the southwestern property boundary for the SWAT team and a firearm training center with earthen backstop berm in the northwestern corner of the former Launch Area for target practice.

The Town of Hamburg Master Plan depicts the Nike Battery Site, including the former Launch Area, as public land and indicates this general area of town contains rural residential development with open space, parks, and undeveloped lands (Wendel Duchscherer Architects & Engineers 2008). The former Launch Area is zoned for residential-agricultural use (Town of Hamburg 2018). Residential properties are located approximately 500 feet south of the former Launch Area, along Lakeview Road. However, future residential use of the Launch Area is not considered a realistic or reasonable scenario based on the current use and land ownership, proximity to the Hamburg Landfill, and the presence of the underground silos. Per a conversation with the Town Engineer on 21 November 2019, the former Launch Area will never realistically be redeveloped for future residential use (Personal communication with Mike Quinn, 2019). Therefore, future residential exposure and risk will not be evaluated.

Potential human receptors under the current and most likely future land use include on-site commercial/ industrial indoor workers, on-site outdoor maintenance workers, on-site construction/utility workers, and off-site residents.

Figure 10.3, below, is a graphical representation of the preliminary human health CSM for the former Launch Area. The exposure media of concern for human health include surface soil (0 to 1-foot bgs), subsurface soil (1-foot bgs to the depth of saturated soils), and groundwater. Surface water and sediment are not considered potential exposure media, as there are no permanent surface water bodies or intermittent streams at the former Launch Area, and a hydrologic connection between groundwater underlying the former Launch Area and Eighteen Mile Creek is not confirmed. In general, an exposure pathway consists of the following components:

- Source and mechanism of chemical release to the environment;
- Environmental transport medium (e.g., surface soil);
- Point of contact with the impacted medium (exposure point; e.g., workplace); and
- Exposure route at the exposure point (e.g., dermal contact with surface soil).

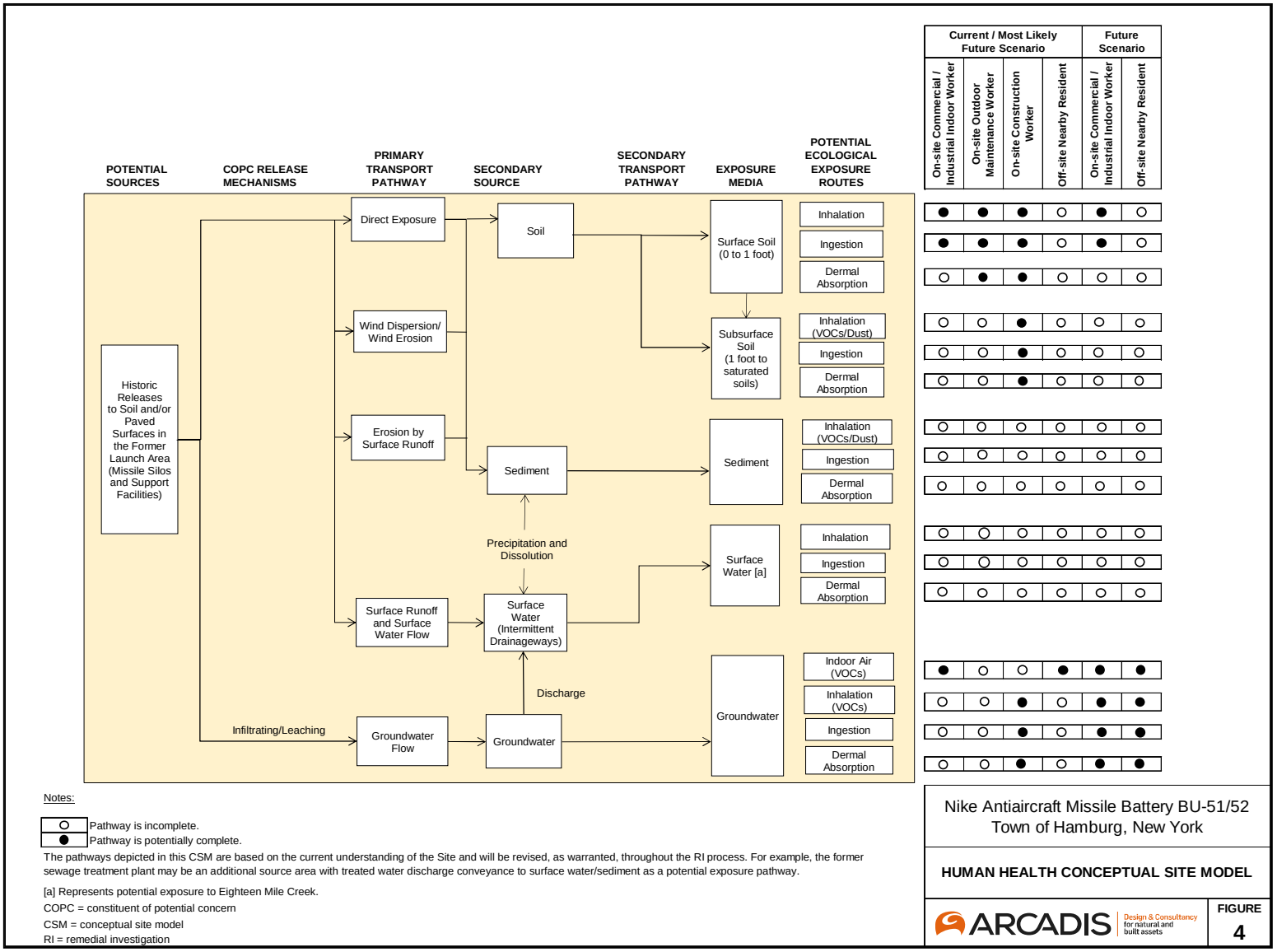
If all four components are present (or potentially present), the pathway is considered complete (or potentially complete). Each pathway defines a unique mechanism by which potential receptors are directly or indirectly exposed to constituents of concern.

Generally, human exposure to DoD-related impacts in soil may occur through incidental ingestion, dermal contact, and inhalation (particulates and volatile constituents). Under the current and most likely future land use, direct exposure to DoD-related impacts in groundwater may occur through incidental ingestion, dermal contact, and inhalation (mostly for VOCs), if shallow groundwater is encountered during site excavation. Indirect exposure to volatile constituents in shallow groundwater could occur through inhalation of indoor air, if an occupied building overlies affected groundwater.

Groundwater at the former Launch Area is not used as a drinking water source. Potable water is supplied to the former Launch Area and nearby residents by the Erie County Water Authority, which receives surface water from Lake Erie and the Niagara River. Therefore, use of groundwater as drinking water is currently an incomplete exposure pathway. However, no restrictions would limit future use of the surficial aquifer as a drinking water source. On-site commercial/industrial indoor workers and off-site nearby residents may potentially use groundwater as drinking water in the future. Therefore, these groundwater exposure pathways are potentially complete under hypothetical future exposure scenarios. These pathways could include preferential flow from the overburden groundwater to topographically low areas of the bedrock surface, and subsequently to the bedrock groundwater through fracture or bedding-plane pathways. If existing downgradient residential drinking wells are identified during the well survey, SERES-Arcadis JV will discuss with USACE if sampling of these wells will occur, and if a QAPP amendment is needed.

A preliminary search of public databases, including the Safe Drinking Water Information System (USEPA 2020a), United States Geological Survey (USGS) National Water Information System (USGS 2020) and NYSDEC Water Wells Database (NYSDEC 2020), was performed in November 2019 to identify water supply wells within 1 mile of the address for the former Launch Area. Based on this search and what is currently known about surrounding land uses (which are primarily residential), irrigation wells and industrial wells were not identified. Therefore, use of groundwater for irrigation or as industrial process water was not identified as a potential exposure scenario.

903 **Figure 10.3: Nike Battery BU-51/52 Preliminary Human Health Conceptual Site Model**



904

10.3.2 Ecological Receptors and Exposure Pathways

Ecological receptors include terrestrial plants, soil invertebrates, and birds and mammals using the lands for nesting, foraging, and burrowing. However, the on-site habitat is developed area characterized by asphalt- and gravel-paved areas and building structures. Due to the proximity of human activities and the degree to which the area has been reworked, ecological receptors using these areas of the site are likely limited. Representative ecological receptors potentially using the former Launch Area are discussed further below and in **Appendix C**.

For those areas of the former Launch Area that provide viable ecological habitat, ecological receptors may be exposed to on-site surface soil (0 to 1-foot bgs) through ingestion, dermal contact (during feeding or preening activities), and (for bioaccumulative constituents) uptake through the food web. Potential exposure for ecological receptors to subsurface soils would be limited to instances of ecological receptors' burrowing activities. Exposure to groundwater is not expected because the depth to groundwater is approximately 4 to 7 feet bgs, unless groundwater seeps are identified at the former Launch Area.

The intermittent drainage ditches that exist in the former Launch Area are not expected to provide aquatic habitat, and sediment and surface water sampling is not proposed at this time. However, if the habitat assessment identifies the occurrence of on-site aquatic habitat (e.g., vernal pools, permanent or semi-permanent water in the drainage ditches or swales) that may be affected by site conditions, then surface water and/or sediment sampling may be considered.

Other surface water bodies in the vicinity of the former Launch Area include the mitigation pond (approximately 0.5 mile north of the site), the twin pond area (approximately 1 mile north of the site), and Eighteen Mile Creek (approximately 1 mile southeast of the site). These areas provide aquatic habitat for ecological receptors. However, preliminary data indicate that there are no complete transport mechanisms for site-related constituents to reach these areas, and therefore it is expected that they will not be evaluated in the screening level ecological risk assessment (SLERA). If the RI data indicate the potential for transport, then these areas may be considered in the risk assessment process.

Based on the available information regarding potential contaminant transport and site use by ecological receptors, potentially complete ecological exposure pathways are exposure of plant communities, terrestrial invertebrates, and wildlife (primarily birds and mammals) to DoD-related impacts present in soil and ingestion of bioaccumulative DoD-related impacts via the food chain by birds and mammals that may reside or forage in the habitats present on site. Representative ecological receptors and potentially complete pathways for the former Launch Area are discussed further in Section 3.2.2 of **Appendix C** and listed below:

- Herbivorous birds and mammals: mourning dove (*Zenaida macroura*) and meadow vole (*Microtus pennsylvanicus*)
- Invertivorous/omnivorous birds and mammals: American robin (*Turdus migratorius*) and short-tailed shrew (*Blarina brevicauda*). American robin and short-tailed shrew will be used to represent both invertivorous and omnivorous birds and mammals by modifying the diet assumptions to represent each feeding guild.
- Carnivorous birds and mammals: red fox (*Vulpes vulpes*) red-tailed hawk (*Buteo jamaicensis*)

The federally-listed threatened northern long eared bat (insectivorous mammal) may be present within Erie County, New York. However, due to the lack of water bodies at the former Launch Area, this species is not expected to utilize the site habitat. Section 10.4 discusses the northern long-eared bat in more detail.

10.4 Regional Sensitive Ecological Resources

A search of the USFWS Information for Planning and Consultation (IPaC) website (https://ecos.fws.gov/ipac/) in September 2019, found one federally-listed threatened mammal species (northern long eared bat [*Myotis septentrionalis]*) which may be present within Erie County, New York. A search of the NYSDEC Nature Explorer website (available at link: http://www.dec.ny.gov/natureexplorer/app/) conducted in September 2019 confirmed the potential presence of the federal- and state-threatened northern long-eared bat in the vicinity of the former Launch Area. The SLERA will evaluate the potential site usage of the northern long-eared bat, and (if appropriate) the potential for ecological risk to this specific receptor species.

10.5 Land Use Considerations

The former Nike Antiaircraft Missile Battery BU-51/52 originally consisted of approximately 150 acres in a roughly rectangular shape trending southwest to northeast on Lakeview Road in Erie County, Hamburg, New York, approximately 5 miles southeast of Lake Erie (**Appendix A: Figure 2**). The former Launch Area located at the south end of the former Nike Battery is currently used by the Town of Hamburg's Highway Department for office space, truck garage space, and storage of gravel piles and road salt. The Town of Hamburg police department uses the former enlisted men's barracks along the southwestern property boundary for the SWAT team and a firearm training center with earthen backstop berm in the northwestern corner of the former Launch Area for target practice (**Appendix A: Figure 3**). From 1970 to 1984, the Town of Hamburg operated a landfill north of the former Launch Area. The former sewage treatment plant located on the southern side of North Creek Road was used by DoD. Based on the INPR 01 February 1991, it states that the Town requested it to be demolished, and that there was no evidence of hazardous/toxic waste, unsafe debris, or unexpected ordinances remaining from DOD use. However, during utility location in July 2020, sewage treatment infrastructure was determined to be in place, and fresh sewage was observed. On July 31, 2020, JV confirmed with Rick Nowak of the Town Buildings and Grounds Department that the treatment system is active and has been used since the Town acquired the property from the DoD. As such, is not possible to distinguish impacts from the DoD versus the Town, and the former sewage treatment plant is not assessed in this QAPP. The former Launch Area is bounded on the north by Interstate I-90; on the west by the Easement Area; on the east by residential areas; and on the south by residential areas, Lakeview Road, and Eighteen Mile Creek. The closest residential housing is located approximately 500 feet to the south, on Lakeview Road. There are no state or federally designated wildlife refuges or recreational areas on or adjacent to the Nike former Launch Area.

10.6 Key Physical Aspects of the Site

10.6.1 Climate

Erie County is located on the eastern shore of Lake Erie and is characterized by moderately warm summers and bitterly cold winters. Cold winds in the winter typically prevail from the northwest in the Great Lakes region, producing dramatic lake-effect snowfalls on the southern and eastern shores of the Great Lakes. Total annual rainfall precipitation averages 41 inches for Erie County. The average annual snowfall is

approximately 92 inches with the heaviest accumulations typically in the period from December to March. In the warmer months of July and August, 80 degrees Fahrenheit (°F) is the normal high temperature. The colder months of January and February have a normal low temperature of 16°F.

10.6.2 Site Hydrology

The below ground silos are in an area currently used by the Hamburg Highway Department for staging of materials. The silo area drains toward Eighteen Mile Creek located south of the former Launch Area. The Town's former landfill (Hamburg Landfill) is located north of the former silo area with more natural and less disturbed areas between the landfill and the former Launch Area (**Appendix A: Figure 2**). The former Launch Area is highly disturbed with little native vegetation. There is a small manmade drainage swale along the southeastern border of the former Launch Area that may collect runoff during periods of heavy rainfall and snowmelt (**Appendix A: Figure 4**). The drainage swale extends approximately 450 feet into a small patch of phragmites (Bluestone 2019).

Surface water at the former Launch Area flows into a drainage ditch that drains the perimeter of the former Town landfill, then traces the southwest and southeast sides of the silo area (**Appendix A: Figure 4**). This ditch drains east to a second ditch that discharges to Eighteen Mile Creek, located along the southern boundary of the former Launch Area. Eighteen Mile Creek flows westward approximately 6 miles to Lake Erie (Foster 1999). Natural surface water drainage at the former Launch Area emanates from an area northeast of the landfill and flows along the western landfill boundary. All other surface water drainage is controlled by ditches. The Highway Department constructed a bermed wetlands mitigation pond within the last 5 years approximately 800 feet northeast of the former Launch Area (**Appendix A: Figure 2**). Much of the northern portion of the former Launch Area is covered with wetlands; specifically, freshwater forested/shrub and freshwater emergent with two freshwater ponds. 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.

Groundwater at the former Launch Area is approximately 4 to 7 feet bgs in the overburden and flows southward from the former Launch Area toward Eighteen Mile Creek (M&E, 1989). Percolation testing conducted in 1997 indicated that the hydraulic conductivity of the proglacial lake deposits, measured at a depth of 10 feet at two locations on former Launch Area, was 1.58×10^{-8} feet per second (ft/sec) and 2.82×10^{-8} ft/sec (SJB 1997). Groundwater velocity in the fractured shale was calculated at the former Launch Area at approximately 20 feet per year (SJB 1997). Groundwater flow maps provided by Battelle (2000a) show no difference in depth to groundwater measurements between overburden and bedrock wells suggesting hydrogeologic communication between the two units.

10.6.3 Site Geology

The former Launch Area is approximately 5 miles southeast of Lake Erie (**Appendix A: Figure 1**). The area surrounding the former Launch Area is characterized by a relatively flat land surface that slopes gently toward Lake Erie. Elevation ranges from 756.5 feet above mean sea level (amsl) to 761.6 feet amsl (M&E 1989).

Previous investigations at the Nike Battery show that the subsurface in the area of the former Launch Area is composed of interlaminated silt and clay derived from proglacial lake deposits (Rickard and Fischer 1970, Niagara Sheet). The site thickness ranges from 8 to 18 feet (GZA 1999). A grain size distribution test

determined that the overburden unit consisted of 20.2% gravel, 24.1% sand, 33.4% silt, and 22.3% clay (SJB 1997).

The proglacial lake sediments were deposited over shales of the Upper Devonian West Falls Group (Rickard and Fisher 1970). West Falls shale in the vicinity of the former Launch Area consists of two members: the Angola Member, which overlies the Rhinehart Member. Both members are described as a black and gray shale and light gray siltstone and sandstone with a thickness from 400 to 520 feet (LaSala 1968). The unit dips gently southward. Based on the morphology of streams flowing over this unit, there appears to be northeast/southwest trending joint system, which is typical of western and central New York. A weathered shale zone with a thickness of 2 to 8 feet lies below the proglacial lake deposits (GZA 1999); fractured shale is found below that depth (Battelle 2000a).

10.6.4 Sources of Drinking Water

Public water is supplied to the Town of Hamburg by the Erie County Water Department. Raw water is drawn from Lake Erie and the Niagara River at the Sturgeon Point and Van de Water Treatment Plants, respectively (available at link: https://www.ecwa.org/my-water/treatment-process/). Residents are not required to connect to the public water supply. A Freedom of Information Law (FOIL) request submitted to Erie County Water Authority revealed four residential parcels within a 1-mile radius of the Nike Battery with potable water wells (Bluestone, 2019). Bluestone (2019) noted three additional registered water wells were listed in the NYSDEC online water well registration database for the Town of Hamburg in Erie County (NYSDEC 2018a).

10.6.5 Cultural Sites

Previous investigations at the Nike Battery did not indicate evidence that any culturally-sensitive sites are located within the area of the former Launch Area being investigated as part of the RI.

10.7 Data Gaps

Before developing the QAPP, the SERES-Arcadis JV reviewed existing data and performed a site visit on September 10, 2019. Data gaps are identified in **Table 10.7** below, along with the planned approach for addressing each data gap.

The exposure media of concern for human health include surface soil (0 to 1-foot bgs), subsurface soil (1-foot bgs to the depth of saturated soils or 10 feet whichever is first), and groundwater. **Figure 10.3** illustrates the potential exposure media and pathways for each identified human receptor population. **Worksheet #11** discusses the soil and groundwater data needed to complete the human health and ecological risk assessments. Additionally, information on ecological habitats and receptors will be gathered during the RI fieldwork, and a well search will be performed to identify residential wells downgradient of the former Launch Area. If concentrations in the samples exceed the regulatory benchmarks or risk-based screening levels, then additional delineation samples may be collected following the completion of an initial risk evaluation and potential consultation with the USACE and stakeholders. The discrete soil sampling analytical results will be used to develop DUs for implementing a soil sampling plan based on incremental sampling methodology (ISM) and evaluating risk, if necessary. Sample methodology for groundwater investigation and the soil investigation is discussed in **Worksheet #17**. Sample information, such as nomenclature, media, depth, type, analytes, and rationale, is included in **Worksheet #18**.

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Data Gap	Goal	Approach to Address Data Gap
Existing groundwater monitoring wells are approximately 20-30 yrs. old.	Use new groundwater monitoring wells to eliminate well conditions that would invalidate data.	• 3 wells were identified during the September 2019 site visit. • These wells will be abandoned, and new wells will be installed.
Incomplete horizontal and vertical delineation of the groundwater flow and impacts from potential source areas.	Determine groundwater flow direction and assess whether overburden and bedrock groundwater are in communication.	• 21 new monitoring well locations are proposed, and the direction of groundwater flow in both the overburden and shallow bedrock will be evaluated. A bedrock surface map will also be prepared to evaluate potential low areas where preferential transport of contaminants could occur. • The degree of communication between the overburden and bedrock groundwater will be evaluated using the information from 16 overburden and 5 bedrock wells. • Rock core will be used to evaluate the bedrock lithology and shallow bedrock properties.
	Determine the presence and extent of DoD-related chemicals at or above levels of concern in groundwater in areas with incomplete data.	• Analytical results from two rounds of groundwater samples from each newly installed monitoring well will be compared to regulatory benchmarks, risk-based screening levels, and data from the upgradient overburden monitoring well.
	Assess potential human health risks based on current and reasonably anticipated future uses.	• Analytical results from the RI will be used to select human health COPCs. • Exposure units and exposure point concentrations (EPCs) for on-site groundwater COPCs will be based on evaluation of the groundwater analytical data from the proposed RI samples and any subsequent groundwater sampling events, if warranted. • If residential wells downgradient of the former Launch Area are identified, groundwater samples will be collected from these wells and used in the evaluation of potential risks to off-site residents at individual properties.
Groundwater data are outdated, and some detection limits are above regulatory standards or applicable screening levels.	Obtain additional groundwater data so that present day conditions are represented in the data set.	• The new monitoring wells will be installed using performance-based well development techniques to ensure adequate development. • 1 groundwater sample will be collected from each well using USEPA low-flow sampling methods during each of two groundwater monitoring events. • Water quality parameters will be evaluated during groundwater sampling using a calibrated water quality meter. • Hydraulic conductivity data will be updated using rising- and/or falling-head conductivity tests (as appropriate) with transducers/electronic monitoring techniques.
Surface and subsurface soil data are limited.	Determine the presence and extent of DoD-related chemicals at or above levels of concern in soil in areas with incomplete data.	• Continuous split spoon samples will be collected while drilling overburden monitoring wells. Each boring will have up to 3 soil samples, 1 surface (top of native material to 1-foot below) and up to 2 subsurface, will be collected during installation of each of the 16 overburden wells. Based on the anticipated depth to groundwater, the deepest subsurface soil sample from each boring will be collected at 10 feet or above. • Analytical results from soil samples will be compared to regulatory benchmarks, risk-based screening levels, and site-specific reference sample data that may reflect background conditions (i.e., reference soil sample data).
	Investigate bedrock fractures to assist in characterizing site-specific hydrogeology.	• A log will be generated for each location, and for the bedrock portion of wells, site specific rock lithology will be ascertained, and rock quality will be characterized and evaluated for the quantity and magnitude of fractures in accordance with USACE EM 1110-1-1804 guidance.
	Assess potential human health risks based on current and reasonably anticipated future uses.	• Analytical results from the RI will be used to select human health COPCs. • Exposure units and EPCs for soil COPCs will be based on evaluation of the soil sample locations and analytical data from the proposed RI samples and any delineation samples, if warranted.

Data Gap	Goal	Approach to Address Data Gap
	Assess potential ecological risks.	• Analytical results from the RI will be used to select constituents of potential ecological concern (COPECs). • Exposure units and EPCs for soil COPECs will be based on evaluation of the analytical data from the proposed RI samples and any delineation samples, if warranted, and on the results of the habitat assessment.
Insufficient historical surface soil data is available to assess human health and ecological screening at the site.	Collect a sample set for statistical analysis of the 95% upper confidence limit.	• 16 surface soil samples will be collected at the same location as groundwater samples. 12 additional surface soil sample locations were selected based on historic and current use of the former Launch Area or were biased toward drainageways where constituent concentrations are potentially concentrated. In addition, up to 5 discretionary surface soil samples will be collected based on the results of habitat assessment.
Background level data are limited.	Collect a sample set for statistical analysis of the 95% upper confidence limit.	• 16 surface soil (top of native material to 1-foot below) and 16 subsurface soil samples will be collected for the purpose of gathering site-specific reference soil data for comparison against sample concentrations in the former Launch Area. Reference soil sample locations were selected in consultation with the USACE and are further described in Worksheet #17. Sampling of the former Launch Area will occur before reference soil sample collection, and soil from the reference sample will be collected after confirming that reference sample depth and soil classification are consistent with samples from the former Launch Area.
Some historical data has detection limits above regulatory limits or applicable screening levels.	Set detection levels equal to or lower than regulatory limits or applicable screening levels, to the extent possible, to allow for comparison.	• Through the QAPP process, it will be confirmed that the laboratory can meet all necessary detection limits for regulatory limits or applicable screening levels.
PCB samples were not collected in the area of the former transformer pad.	Determine the presence and extent of PCBs in the area of the former transformer pad.	• 3 surface soil (top of native material to 1-foot below) samples will be collected from beneath the current gravel surface cover layer for PCB analysis. • Initial sampling is intended to determine the presence or absence of PCBs. Subsequent delineation sampling will be conducted if total PCB concentrations in any of the samples are greater than 1 ppm. In the event delineation samples are warranted, it is anticipated a sufficient number of soil samples will be collected so the PCB data can be used in the risk assessments.
Ecological habitats require additional characterization.	Identify potential ecological habitat for the purpose of determining ecological receptors.	• As part of the SLERA, site figures and aerial photographs will be reviewed to identify areas with potential ecological habitat in the vicinity of the former Launch Area. • Habitat characteristics will be further documented during a site visit to identify vegetative cover-types.
Ecological receptors require additional evaluation.	Characterize potential health risks to ecological receptors within the former Launch Area.	• As part of the SLERA a site visit will be conducted to document potential ecological receptor species observed or expected to occur at the former Launch Area based on available habitat.
Public and residential well information is limited.	Identify downgradient residents with existing wells or potential to install wells, which are potential sensitive receptors.	• A search of public databases will be performed to identify commercial and public water supply wells within a 1-mile radius of the former Launch Area. • Information on the location, ownership, and screened interval depth (if available) of residential wells in the vicinity of the former Launch Area will also be obtained from local sources.

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QAPP Worksheet #11: Project/Data Quality Objectives

(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

11.1 Objective of the Project

The DQO process per the USEPA (1993) was used to develop a sampling design for the former Launch Area.

The seven steps in the DQO process are to:

1. State the Problem;
2. Identify the Decisions and Goals of the Study;
3. Identify Inputs to the Decision;
4. Define the Study Boundaries;
5. Develop a Decision Rule/Analytic Approach;
6. Specify Limits on Decision Errors; and
7. Optimize the Design for Obtaining Data

Each of these steps is described below.

1. State the Problem

DoD-related chemicals may have been released to surface soil, subsurface soil, and groundwater because of historical activities related to DoD activities in the former Launch Area. Previous investigations and historical records review have been performed, but additional data are needed to quantify and qualify potential environmental impacts in the former Launch Area and to determine the risk to ecological and human receptors. The identified data gaps are outlined in **Table 10.7**.

2. Identify the Decisions/Goals of the Study

The objective of the project is to collect sufficient data to determine the presence and extent of DoD-related chemicals above applicable reference concentrations and levels of concern in soil and/or groundwater, to characterize the exposure potential for human receptors such as on-site workers and off-site downgradient residents, and to assess potential risks to human health and ecological receptors related to the former Launch Area based on reasonably anticipated future uses. The study boundary will be evaluated based on the RI findings. A secondary goal is to update the conceptual site model (CSM). Goals related to each data gap are identified in **Table 10.7.**

3. Identify Inputs to the Decision

Inputs for each data gap are identified in **Table 10.7**. The following resources and data will be used as information inputs:

- Reports documenting prior investigations;
- Preliminary human health and ecological CSMs;
- Public database searches;
- Future RI surface and subsurface data, such as:
    - Field measurements and observations (soil, groundwater, geology);
    - Laboratory analysis of samples in area of concern (soil, groundwater, geology);
    - Laboratory analysis of reference samples (soil metals and PAHs); and
- Survey of residential and commercial wells located within and adjacent to the former Launch Area

4. Define the Study Boundaries

The initial spatial study boundary is the boundary of the former Launch Area of the Nike Battery Site (**Appendix: Figures 1 and 2**). The former Launch Area is bound on the north by Interstate I-90; on the west by the Easement Area; on the east by residential areas; and on the south by residential areas, Lakeview Road, and Eighteen Mile Creek. The vertical study boundary will initially be the first/upper water-bearing zone in the shallow bedrock. The actual horizontal and vertical study boundaries will be determined by the results of the RI. The temporal boundary of this study is detailed in the schedule located in **Worksheets #14 and #16**.

5. Develop a Decision Rule/Analytic Approach

Regulatory benchmarks and risk-based screening levels for data are specified in **Worksheet #15** and include the USEPA RSLs for Residential Soil and USEPA RSLs for Tapwater (USEPA 2019) for human health. The RSLs protective at a cancer risk level of 1×10^{-6} or a noncancer hazard quotient of 0.1 will be used. For ecological receptors, screening levels provided in **Worksheet #15** are based on (in order of preference) the USEPA EcoSSLs (2007), USEPA Region IV soil ESVs for ecological receptors (USEPA 2018), or Department of Energy Laboratories (LANL 2017).

If the DoD-related chemical concentrations in all the samples from the investigation area are less than the screening levels identified in **Worksheet #15**, then no further action will be indicated for the former Launch Area.

If metals or PAH concentrations in the samples from the investigation area exceed the RSLs identified in **Worksheet #15**, a comparison to site-specific background threshold values calculated from the reference soil sample data and a comparison of estimates of central tendency based on box plots and two-sample hypothesis testing will be made. Random discrete reference soil samples will be used to calculate the background threshold values. Constituents with concentrations that are statistically similar to reference levels will be eliminated from further quantitative evaluation (USACE 2011).

If sample results for constituents exceed the screening levels identified in **Worksheet #15** (and exceed reference levels for metals and PAHs), then a baseline human health risk assessment (HHRA) and screening level ecological risk assessment (SLERA) and, if necessary, a SLERA refinement step (Step 3a) will be performed to assess potential risk to human and ecological receptors. The risk assessments will document the results of the data comparisons for metals and PAHs in the discussion of reference soil sample data and in the discussion of uncertainties.

The HHRA and SLERA will be performed in accordance with USEPA Risk Assessment Guidance (1989, 2001, 2004, 2009) and USACE EM 200-1-4 Volumes 1 and 2 Risk Assessment Guidance (1999).

Step 7 ("How much data are needed") summarizes the analytes, parameters, media, and sample quantities, and **Worksheet #17** contains information on the sampling design and rationale.

6. Specify Limits on Decision Errors

Field precision will be controlled by using experienced field personnel, properly calibrated field instruments/equipment, and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system, including sampling, handling, shipping, storage, preparation, and analysis. Laboratory data precision for organic analyses will be monitored by using matrix spike duplicates (MSDs), laboratory duplicates, and field duplicates.

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated instruments and equipment, and adherence to established protocols and standard business practices. The accuracy of field instruments will be assessed by review of calibration and maintenance logs. Laboratory accuracy will be assessed via the use of matrix spikes (MSs), surrogate spikes, and internal standards.

Data validation will consist of a review of QC data and raw data to verify that the laboratory was operating within required limits, analytical results are correctly transcribed from the instrument, and which, if any, environmental samples are related to any out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements. Data validation will consist of data screening, checking, reviewing, editing, and interpreting to document analytical data quality and determine if the quality is sufficient to meet DQOs.

7. Optimize the Design for Obtaining Data

The project team organization was selected to create an integrated team that has collaborated on numerous projects and includes local, cost effective field staff with experience performing site assessments safely and efficiently. The project team qualifications are listed in **Worksheets # 4, 7, and 8**.

Before designing the sampling program, data gaps were identified. Approaches for addressing the data gaps have been incorporated into the sampling program and are identified in **Worksheet #10**. To increase efficiency, subsurface soil samples and surface soil samples will be collected at the same location as groundwater samples. Additional surface soil samples will be collected to ensure a statistically valid data set. Sampling locations have been selected based on historical data, historical activities, and current activities onsite. **Worksheet #17** describes the design for data collection, and documents Step 7 of the DQO process.

Who will use the data and what will the data be used for?

CENAE will use the data collected from the former Launch Area site characterization to determine if areas within the former Launch Area present unacceptable risks to human health or the environment under current and reasonably anticipated future use scenarios due to the presence of elevated concentrations of constituents due to DoD activities. The data may also be used to inform remedial decision-making should that be required based on the results of the RI.

What types of data are needed?

Soil and groundwater samples will be collected and analyzed during the RI field work. Surface water, sediment, porewater, or biological tissue samples are not proposed at this time. Subsurface geologic information necessary to define the nature and extent of contamination (if present) will also be required. This will include soil classification using the Unified Soil Classification System (USCS), identification of fine-grained soil units that could retain contamination, and overburden and shallow bedrock groundwater samples to support decision-making for any additional RI phases and/or remedial decision-making. The future need for these types of samples (if any) will be based on whether a release is confirmed to these media and whether these media occur on the former Launch Area.

How much data are needed?

Data will be collected to fill the identified data gaps, including groundwater and soil samples from the former Launch Area, and to characterize potential human health and ecological receptors and exposure pathways.

The current and most likely future land use for the former Launch Area is industrial. Potential human receptors under the current and most likely future land use include on-site commercial/industrial indoor workers, on-site outdoor maintenance workers, on-site construction/utility workers, and off-site nearby residents. The exposure media of concern for human health include surface soil (0 to 1-foot bgs), subsurface soil (1-foot bgs to the depth of saturated soils or 10 feet whichever is first), and groundwater. **Figure 10.3** illustrates the potential exposure media and pathways for each identified human receptor population. The soil and groundwater data described below are needed to complete the human health and ecological risk assessments. Additionally, information on ecological habitats and receptors will be gathered during the RI fieldwork, and a well search will be performed to identify residential wells downgradient of the former Launch Area.

- Soil Samples:
    - Thirty-two surface and subsurface soil samples (**Appendix A: Figure 7A and Figure 7B**) will be collected from 16 locations and analyzed for metals and PAHs for the purpose of gathering site-specific reference sample data for comparison against sample concentrations in the former Launch Area. The locations were selected from an adjacent area outside of the investigation area that is used by the Town for recreational purposes.
    - Up to 53 surface soil samples will be collected for varying analyses. Surface soil samples (**Appendix A: Figure 7A and Figure 7B**) will be collected from the top of native material to 1-foot below using either a split spoon sampler (where collocated with a drilling location) or stainless-steel hand auger.

- Three surface soil samples will be collected for PCB analysis beneath the gravel layer adjacent to the former transformer pad. Previous investigations did not include collection of soil samples around the former transformer pad. Therefore, samples will be collected for PCB analysis to assess if a release occurred. One sample will be collected from the north, south, and east sides of the transformer pad, totaling three samples. A sample will not be collected from the west side of the transformer pad because the building extends west of the former transformer room.
- Twenty-nine surface soil samples will be collected for target analyte list (TAL) metals, target compound list (TCL) VOCs, TCL SVOCs, and hydrazines to evaluate the potential risks associated with surface soil exposure to site users. These lists of constituents are a broad list of analytes since there is insufficient data to target specific compounds. Based on historical review, metals, VOCs, and SVOCs have been associated with Launch Area operations. The sample locations were selected based on historic and current use of the former Launch Area or were biased toward drainageways where constituent concentrations are potentially concentrated.
  - Eight soil samples will be collected from beneath the gravel layer near the former silos;
  - Four samples from grass-covered areas near the former generator building;
  - Three samples at the former transformer pad (one sample on each of three sides);
  - One sample on the south side of a building currently used by the Highway Department;
  - One sample from the drainage swale from the landfill area;
  - One sample at the former sewage treatment plant; and
  - Eleven samples from drainageways around the former Launch Area.
- Up to five discretionary surface soil samples will be collected from locations identified in the field, based on the results of habitat assessment that will be conducted to support the SLERA. The soil sample locations will consider areas of viable habitat that are not covered by existing historic or proposed surface soil sample locations. In addition to being analyzed for TAL metals and hexavalent chromium, TCL VOCs, TCL SVOCs, and hydrazines, the discretionary samples will be analyzed for grain size by ASTM D422, total organic carbon (TOC), moisture content, and pH.
- Sixteen reference surface soil samples will be collected and analyzed for TAL metals, hexavalent chromium, and PAHs.

o Up to 50 subsurface soil samples will be collected for analysis. Soil samples will be characterized in accordance with the USCS and, where appropriate/relevant, fine-grained soil types will be considered when selecting aliquots for laboratory analysis. Subsurface soil sampling will include:

- Up to 34 continuous split spoon subsurface soil samples will be collected during installation of 16 overburden wells from the top of native material to the water table. One soil sample will be collected from the 1 to 3 feet interval below native material at all locations. Field screening of the split spoon samples will be conducted using visual, olfactory, and photoionization detector (PID) methods. The second soil sample will be collected from the zone above the water table that exhibits the greatest impacts based on field screening. If no indications of impacts are evident, the second soil sample will be collected from the 2-foot

interval above the water table. Soil samples will be analyzed for TAL metals and hexavalent chromium, TCL VOCs, TCL SVOCs, hydrazines, grain size by ASTM D422, TOC, and moisture content.

- Sixteen reference subsurface soil samples will be collected concurrently with reference surface soil samples and analyzed for TAL metals, hexavalent chromium, and PAHs.

- If concentrations in the samples exceed the regulatory benchmarks or risk-based screening levels, then additional delineation samples may be collected following the completion of an initial risk evaluation and potential consultation with the USACE and stakeholders. Additional factors considered in the initial risk evaluation may include the comparison of metals and PAH concentrations to reference concentrations, the results of hypothesis tests used to compare the site and reference data sets for metals and PAHs, and possibly the comparison of 95% upper confidence limits or arithmetic average concentrations to regulatory benchmarks or risk-based screening levels.

- The discrete soil sampling analytical results will be used to develop DUs for implementing a soil sampling plan based on ISM and evaluating risk, if necessary.

- Groundwater Samples:

  - Twenty-one new monitoring wells (16 overburden and five bedrock wells) are proposed to evaluate the nature and extent of any groundwater impacts and for delineation of groundwater flow (**Appendix A: Figure 6**). The quantity and locations of proposed wells were determined based on review of areas previously sampled during historical investigations and areas where additional horizontal and vertical delineation of groundwater flow and impacts from potential sources areas is needed. The five bedrock wells will be located adjacent to five of the 16 overburden wells. Groundwater samples will be analyzed for TAL metals (USEPA Methods 6010/6020), TCL VOCs (USEPA Method 8260C), TCL SVOCs (USEPA Method 8270D), and hardness. These lists of constituents are a broad list of analytes since there is insufficient data to target specific compounds. Based on historical review, metals, VOCs, and SVOCs have been associated with Launch Area operations. The hardness parameter was selected due to the salt storage onsite and the presence of the landfill. Salinity of the groundwater will be evaluated using an on-site flow-through cell and salinity meter during groundwater sampling activities.

  - One groundwater sample will be collected from each of the 21wells during each of two groundwater monitoring events. The first event is anticipated to occur during late spring during high water table conditions. The second event is anticipated to occur during the late summer, during low water table conditions. Prior to sampling, water levels will be recorded using an oil-water interface probe. Wells will be developed prior to sampling using the methodology outlined in USACE guidance document EM 1110-1-4000 Monitoring Well Design, Installation, and Documentation at Hazardous Toxic, and Radioactive Waste Sites (1998). The water will be containerized per the waste management protocol identified in **Section 17.3**. A low flow purging/sampling method (using a bladder or peristaltic pump) will be used to collect the samples. Water quality parameters, including temperature, conductivity, turbidity, pH, oxidation-reduction potential (ORP), salinity, and dissolved oxygen (DO), will be measured during monitoring well development and low-flow purge activities.

  - Slug tests will be used to evaluate hydraulic conductivity. Five overburden wells will undergo slug testing, with two tests per well, consisting of one rising head test and one falling head test. The

locations of slug tests will be distributed across the investigation area to be representative of the site as a whole. The slug testing will be performed after the first groundwater monitoring event. The data will be corrected as necessary based on screen and filter pack saturation at the time of the testing.

- Human health and ecological receptors/pathways:
  - Ecological habitat characterization – As part of the SLERA, site figures and aerial photographs will be reviewed to identify areas with potential ecological habitat in the vicinity of the former Launch Area. Habitat characteristics will be further documented during a site visit to identify vegetative cover types.
  - Ecological receptors – As part of the SLERA, a site visit will be conducted to document potential ecological receptor species observed or expected to occur at the former Launch Area based on available habitat.
  - Well search – A search of public databases will be performed to identify commercial and public water supply wells within a 1-mile radius of the former Launch Area. Information on the location, ownership, and screened interval depth (if available) of residential wells in the vicinity of the former Launch Area will also be obtained from local sources.

How good do data need to be?

The data must be of sufficient quality to establish ongoing constituent concentration trends and support remedial decision-making purposes – this includes field measurements (i.e., field meters and survey equipment) as well as those performed by an analytical laboratory. Field measurements quality will be maintained using proper equipment maintenance and/or calibration and adherence to field data collection SOPs. All definitive data will be verified for completeness to ensure that all the requested data have been received and that the laboratory reports contain all elements required to be included in the data package per the applicable USEPA analytical methods and in accordance with the DoD Quality Systems Manual (QSM), Version 5.3 (2019), criteria. In addition:

- Analytical data will undergo third-party data validation by Cadena and will be validated to the stage(s) defined in **Worksheet #12**.
- Usability of the data will be determined based on criteria specified in this QAPP.

Who will collect and generate the data? Where, when, and how should the data be collected/generated?

The SERES-Arcadis JV and our subcontractors (i.e., surveyor and analytical laboratory) will collect the data needed to prepare an RI.

- Proposed sampling locations are shown on shown on **Appendix A: Figures 6** and **7**.
- The data will be collected in accordance with the schedule presented in **Worksheets #14/16 and #17**.
- Standard operating procedures (SOPs) will be followed during collection, transport, and analyses of field and QC samples, as well as for the maintenance, calibration, and use of field measurement and survey equipment.

- Field duplicate samples will be collected at a rate of one per every 10 environmental samples for each media, not including QC samples.
- MS/MSD samples will be collected at a rate of one per every 20 environmental samples for each media, not including QC samples. Triplicate volumes will be collected for soil and water MS/MSDs.
- Equipment blanks will be collected at a rate of one per day for the duration of the field sampling activities when non-dedicated sampling equipment is used, including the drill rig bit, soil sampling tools, and water meters.
- Trip blanks will be placed in coolers containing VOC samples.

Katahdin Analytical Services (Katahdin) will generate the analytical data. The laboratory is accredited by the DoD Environmental Laboratory Program (ELAP) and is expected to meet the Measurement Performance Criteria (MPC) specified in DoD QSM Version 5.3 (2019).

How will the data be reported?

The SERES-Arcadis JV will prepare an RI report to present the results of the RI field work. The data will be presented in tables to show detected analytes, exceedances of screening and/or reference levels, and risk assessment results.

How will the data be archived?

All data will be provided to USACE for archival/retention with the project files to be maintained following USACE requirements.

Prior to field sampling, an eQAPP within FUDSChem will be created; the eQAPP will be fully-compliant with the project UFP-QAPP, incorporating methods, sample types, and quality specifications (Limit of Detection [LOD] Limit of Quantitation [LOQ], Holding Time, QC Sample Acceptance Limits, etc.). The pre-population of sampling event information (location information, sample type, matrix, methods, etc.) will also be performed to ensure accurate database field matching and status tracking within FUDSChem.

The analytical laboratory will generate portable document format (PDF) reports and will upload the Staged Electronic Data Deliverable (SEDD) 2a into FUDSChem for review against the approved eQAPP. Analytical data will be validated using the Automated Data Review (ADR) functions within FUDSChem. Following the ADR run, the data validator will review the FUDSChem ADR output and first reviewer's checklist against the laboratory report and supplement the FUDSChem ADR with a manual review, as necessary. A second data validation reviewer will perform the second review of the files to verify the project data quality requirements defined by the eQAPP and this UFP-QAPP have been satisfied. A data review report summarizing the findings from the data validation will be generated and upload it to the FUDSChem library.

Analytical data will be managed in the FUDSChem per the requirements specified in the New England District Data Management Plan to ensure data deliverables are in compliance with Performance Works Statement data requirements. Other types of data that will be uploaded to FUDSChem include: spatial information pertaining to sample locations (northing, easting, and elevation), hydrogeological information and physical parameters (pH, temperature, conductivity, and turbidity for groundwater samples), and monitoring well information (such as screen intervals, depth to water, and well construction data).

QAPP Worksheet #12a: Measurement Performance Criteria (VOCs in Soil/Sediment by 8260C)

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

MPC for field QC sampling results are used to evaluate project data quality indicators (DQIs) such as accuracy/bias and precision. The following tables provide definition of different laboratory and field blanks that will be used as quality control measures to evaluate the site-specific DQIs.

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 5035/8260C
Laboratory SOP: CA-202, CA-214

DQIs	QC Sample of Measurement Performance Activity	MPCMPC
Precision – Overall	Field Duplicate	All target analytes relative percent difference (RPD) ≤50% (soil).
Contamination	Trip Blank, Equipment Blank, and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	Limit of Detection (LOD) / Limit of Quantitation (LOQ)	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	Laboratory Control Sample (LCS) containing all analytes to be prepared in the same manner as field samples	Percent recovery (%R) per DoD QSM Version. 5.3 (2019) Table C-23.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version 5.3 (2019) Table C-23; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12b: MPC (VOCs in Groundwater by 8260C)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 5030B/8260C
Laboratory SOP: Katahdin CA-202

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤35% (water).
Contamination	Trip Blank, Equipment Blank, and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Table C-24.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version. 5.3 (2019) Table C-24; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12c: MPC (SVOCs in Soil/Sediment by 8270D and 8270D-SIM)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 3540C or 3550C/8270D/8270D-SIM
Laboratory SOP: Katahdin CA-213, CA-226, CA-512, CA-526

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤50% (soil).
Contamination	Equipment Blank, and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-25 and C-27.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version 5.3 (2019) Tables C-25 and C-27; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12d: MPC (SVOCs in Groundwater by 8270D and 8270D-SIM)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 3510C or 3520C/8270D/8270D-SIM
Laboratory SOP: Katahdin C-213, CA-226, CA-502

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤35% (water).
Contamination	Equipment Blank, and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-26 and C-28.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version. 5.3 (2019) Tables C-26 and C-28; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12e: MPC (PCBs in Soil/Sediment by 8082A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 3540C, 3545A or 3550C/8082A
Laboratory SOP: Katahdin CA-329, CA-500, CA-524, CA-537

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤50% (soil).
Contamination	Equipment Blank, and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Table C-17.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version 5.3 (2019) Table C-17; RPD of all analytes ≤30%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**

QAPP Worksheet #12f: MPC (Hydrazines in Soil/Sediment by 8315A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 modified 8315A
Laboratory SOP: Eurofins Lancaster Laboratories Environmental (ELLE) WI9431

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤50% (soil).
Contamination	Equipment Blank, and Method Blanks	No target analyte > LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R lab generated control limits: 1,1-Dimethylhydrazine 86-124% Hydrazine 54-128% Methylhydrazine 29-187%
Accuracy/Bias/Precision	MS/MSD	%R lab generated control limits: 1,1-Dimethylhydrazine 86-124% Hydrazine 54-128% Methylhydrazine 29-187% RPD of all analytes ≤30%

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12g: MPC (Metals in Soil/Sediment by SW846 6010C/7471B)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 3050B, 6010C/7471A
Laboratory SOP: Katahdin CA-605, CA-608, CA-611

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤50% (soil).
Contamination	Equipment Blank and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-3 and C-11.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version 5.3 (2019) Tables C-3 and C-11; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12h: MPC (Metals in Groundwater by SW846 6020A/7470A)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 3010A, 6020A/7470A
Laboratory SOP: Katahdin CA-604, CA-627, CA-615

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤35%.
Contamination	Equipment Blank and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-6 and C-12.
Accuracy/Bias/Precision	MS/MSD	%R per DoD QSM Version 5.3 (2019) Tables C-6 and C-12; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12i: MPC (Total Organic Carbon by EPA Lloyd Kahn)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: EPA Lloyd Kahn
Laboratory SOP: Katahdin CA-741

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	≤50% RPD.
Contamination	Equipment Blank and Method Blanks	No target analyte > LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS	%R 80 to 120%
Accuracy/Bias/Precision	MS/MSD	%R 75 to 125% RPD ≤30%

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12j: MPC (Hardness in Groundwater by SM2340B)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: Standard Methods (SM) 2340B (calculation)
Laboratory SOP: Katahdin CA-627

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision - Overall	Field Duplicate	RPD ≤35%.
Contamination	Method Blank	No target analyte > LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R 80 to 120%
Accuracy/Bias	MS to be prepared in the same manner as field samples	%R 75 to 125%
Precision	MSD or Matrix Duplicate	%R 75 to 125% MSD or matrix duplicate: RPD ≤ 20%

Notes:

1. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12k: MPC (Hexavalent Chromium in Soil/Sediment by SW846 7196A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 7196A
Laboratory SOP: Katahdin CA-625

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision – Overall	Field Duplicate	All target analytes RPD ≤50% (soil).
Contamination	Equipment Blank and Method Blanks	No target analyte > ½ LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-9.
Accuracy/Bias	MS to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-9.
Precision	MSD or Matrix Duplicate	%R per DoD QSM Version 5.3 (2019) Tables C-9; RPD of all analytes ≤20%.

Notes**:**

1. The laboratory is DoD ELAP accredited for the test method and is expected to meet the MPC specified in DoD QSM Version 5.3 (2019).
2. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #12l: MPC (Hexavalent Chromium in Groundwater by SW846 7196A)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 7196A
Laboratory SOP: Katahdin CA-772

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision - Overall	Field Duplicate	RPD ≤35%.
Contamination	Equipment Blank and Method Blanks	No target analyte > LOQ. See QAPP **Worksheet #15** for project specific LOQs.
Sensitivity	LOD / LOQ	Sufficiently low to support project specified screening criteria as specified in QAPP **Worksheet #15.**
Accuracy/Bias	LCS containing all analytes to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-10.
Accuracy/Bias	MS to be prepared in the same manner as field samples	%R per DoD QSM Version 5.3 (2019) Tables C-10.
Precision	MSD or Matrix Duplicate	%R per DoD QSM Version 5.3 (2019) Tables C-10; RPD of all analytes ≤20%.

Notes:

1. The laboratory SOPs are provided in **Appendix G**.

QAPP Worksheet #13: Secondary Data Uses and Limitations

(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements for Evaluating Existing Data)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Previous Investigation Sampling Results	*Final Engineering Report, Defense Environmental Restoration Act Confirmation Study and Contamination Evaluation at the Former Nike Battery Site, BU-51/52, Hamburg, New York.* 1989	M&E. Prepared for USACE, Huntsville.	Background information on nature and distribution of potential constituents of concern.	The historical analytical data are considered valid and there are no known limitations regarding their use.
Previous Investigation Sampling Results	*Results of Groundwater Sampling of Neighboring Wells. 1999.*	NYSDOH.	Background information on nature and distribution of potential constituents of concern.	The historical analytical data are considered valid and there are no known limitations regarding their use.
INPR Assessment	*Memorandum for Commander, North Atlantic Division and Commander, U.S. Army Engineer District Omaha, Subject: DERP-FUDS Inventory Project Report for Site No. C02NY0079, Former Nike Battery BU-51/52, Hamburg, New York (Amended). 15 October 2003. [FUDS Document No. C02NY007902 _01.08_1008_a].*	USACE, New York District.	Background information on history of site.	There are no known limitations regarding use of the historical information and findings in this report.
Guidance Document	*Nike Missile Battery Environmental Conditions Assessment Guide. Defense Environmental Restoration Program for Formerly Used Defense Sites, Final Report. July 2003.*	USACE HTRW-CX.	Guide for assessing Nike Missile Sites	There are no known limitations regarding use of the information in this document.

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Previous Investigation Sampling Results	*Data Report, Site Investigation, Former Nike Battery BU-51/52, Hamburg, New York. 2000*	Battelle	Background information on nature and distribution of potential constituents of concern.	The historical analytical data are considered valid and there are no known limitations regarding their use.
Previous Risk Assessment	*Screening Level Human Health Risk Assessment, Former Nike Battery BU-51/52, Hamburg, New York. 2000.*	Battelle	Background information on nature and distribution of potential constituents of concern.	There are no known limitations regarding use of the historical information and findings in this report.
Previous Site Assessment	*Technical Memorandum, Subject: Records Review and Site Visit, Nike Antiaircraft Missile Battery BU-51/52, New York, Formerly Used Defense Site #C02NY0079, 31 August 2018.*	Bluestone. Prepared for USACE New England District.	Background information on nature and distribution of potential constituents of concern.	There are no known limitations regarding use of the historical information and findings in this report.
CSM Data	*Revised Draft Remedial Investigation Work Plan, Nike Antiaircraft Missile Battery BU-51/52, New York, Formerly Used Defense Site #C02NY0079, February 2019*	Bluestone. Prepared for USACE New England District.	Historical data and background information on nature and distribution of potential constituents of concern.	There are no known limitations regarding use of the historical information and findings in this report.

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QAPP Worksheet #14 & 16: Project Tasks and Schedule

(UFP-QAPP Manual Section 2.8.2)
(EPA 2106-G-05 Section 2.2.4)

The project schedule, including the specific task/activity and the person or group responsible for execution is provided in the following table. The complete schedule for this project timeline, including planning document preparation and field activities, report preparation, and meeting support, is presented in **Appendix D** of this QAPP. Note that the schedule is subject to updates and revisions based upon field conditions.

Activity	Responsible party	Deliverable(s)	Deliverable due date
DFW 1: Pre-Mobilization Activities			
Final QAPP, including Final APP	Andy Vitolins *SERES-Arcadis JV PM*	Draft and Draft Final QAPP Submittals	One week after resolution of Draft Final QAPP comments.
DFW 2: Mobilization/Site Preparation			
Mobilization	Kate Clubine SERES-Arcadis *JV Field Supervisor*	Field notes, DQCR	Weekly submittals due Friday of the week following for which the activity was performed.
DFW 3: Environmental Data Collection			
RI Field Activities	Andy Vitolins *SERES-Arcadis JV PM*	Data Package, including data validation summary	Eight weeks after completion of field activities.
DFW 4: Final Report			
Draft RI Report	Andy Vitolins *SERES-Arcadis JV PM*	Draft RI Report	Ten weeks after completion of field activities
Draft Final/Final RI Report	Andy Vitolins *SERES-Arcadis JV PM*	Draft Final/Final RI Report	Two weeks after receipt of Draft/Draft Final comment resolution.

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

(UFP-QAPP Manual Section 2.6.2.3)
(EPA 2106-G-05 Section 2.2.6)

The following **Worksheet #15** tables identify the project action limits (PALs) and practical quantitation limits (PQLs), and provide a comparison of the PALs and PQLs to analytical laboratory reference limits (i.e., LODs and LOQs) for soil and groundwater, per analytical method. The objective is for the laboratory to achieve LOQs low enough to measure analytes at concentrations less than the PALs to obtain a dataset of known quality and sufficient sensitivity to meet project DQOs. The PALs represent the lowest of the relevant human health and ecological screening levels and other applicable criteria that may be used in the RI and later stages of the CERCLA process. The USEPA residential RSLs, based on a target cancer risk of 1×10^{-6} or a target non-cancer HQ of 0.1, were used as the human health PALs for soil (USEPA, 2019). The lowest of the USEPA tapwater RSLs (USEPA, 2019), MCLs where available, and for volatile constituents, the USEPA groundwater Vapor Intrusion Screening Levels (VISLs; USEPA 2020b) were used as human health PALs for groundwater. For ecological receptors, the lowest of available USEPA (2007) EcoSSLs for protection of plants, soil invertebrates, mammals and birds was preferentially used. Where EcoSSLs are not available, the USEPA (2018) Region IV ESVs for soil were used. The laboratory reference limits are provided by the selected laboratory, Katahdin Analytical Services, of Scarborough, Maine.

Conservative assumptions were made when selecting screening levels for use as PALs for purposes of achieving an appropriate level of data quality. Site-specific refinements may be made during application of screening levels for use in the evaluation of analytical data. The PALs are not intended to be used as cleanup levels. Concentrations above the PALs would not automatically trigger a response action but would suggest further Site-specific consideration is appropriate.

The analytical laboratory reference limits presented in **Worksheet #15** tables are as follows:

- Limits of detection (LOD) – The smallest concentration of a substance that must be present in a sample in order to be detected at the detection limit (DL) with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
- Limit of quantitation (LOQ) – The lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias.
- Practical quantitation Limit (PQL) – The desired limit of quantitation set at some fraction of the PAL to account for the potential for error around the actual result.

PQLs were selected per analyte/analytical method/matrix using the following hierarchy of rules:

1. If no PAL is available, the PQL = 5 times the LOQ;
2. If the LOQ ≤ 1/3 the PAL, the PQL = 1/3 PAL;
3. If the LOQ > 1/3 the PAL, the PQL = LOQ.

LODs and LOQs > the PAL are highlighted in the tables. The analytes for which the LOD and/or LOQ > PAL are also discussed further below.

VOCs

- Bromomethane in Soil – The LOQ (0.01 mg/kg) and LOD (0.005 mg/kg) are less than the human health PAL (0.68 mg/kg) but greater than the ecological PAL (0.002 mg/kg). The ecological PAL is the USEPA Region 4 soil ESV and is protective of adverse effects in soil invertebrates. An ESV protective of mammals is also available from the Region 4 guidance document (USEPA, 2018). The mammalian ESV (0.24 mg/kg) is two orders of magnitude greater than the ecological PAL and is greater than the LOQ and LOD for bromomethane in soil. Therefore, the laboratory limits for bromomethane in soil are considered appropriate for meeting project DQOs.
- 1,1,2,2-Tetrachloroethane, 1,1,2-trichloroethane, 1,2-dibromo-3-chloropropane, 1,2-dibromoethane, 1,2-dichloroethane, bromodichloromethane, chloroform, and vinyl chloride in Groundwater – The LOQ and LOD are greater than the associated PALs, which are USEPA residential tapwater RSLs based on a target cancer risk of 1×10^{-6} or for 1,1,2-trichloroethane, a non-cancer HQ of 0.1. The LODs for 1,1,2,2-tetrachloroethane, 1,2-dichloroethane, bromodichloromethane and chloroform do not exceed tapwater RSLs adjusted for a target cancer risk of 1×10^{-5}. The LODs for 1,2-dibromoethane and vinyl chloride do not exceed tapwater RSLs adjusted for a target cancer risk of 1×10^{-4}. The LOD and LOQ for 1,1,2-trichloroethane do not exceed the tapwater RSL adjusted for a non-cancer HQ of 10. The LOQ and LOD are less than or equal to MCLs for 1,1,2-trichloroethane, 1,2-dichloroethane, vinyl chloride and trihalomethanes (applies to bromodichloromethane and chloroform). Based on historical Site activities and the analytical results from previous soil and groundwater samples, 1,2-dibromo-3-chloropropane is not likely a DoD-related constituent. Therefore, the laboratory limits for the VOCs listed above are considered appropriate for meeting project DQOs.

SVOCs

Analysis using Selected Ion Monitoring (SIM) will be conducted to obtain lower LOQs and LODs for a select group of SVOC analytes (i.e., PAHs) with PALs that are less than the full scan limits. Analytes requiring SIM were selected based on the likelihood that the analyte is DoD-related and professional judgement as to the analytes that are more likely to drive potential risk in a risk assessment. SIM is the most sensitive option available from commercial laboratories for multi-compound analysis of SVOCs that retains the specificity of GC/MS analysis.

- Hexachlorobenzene and hexachlorocyclopentadiene in Soil – The LOQ (0.33 mg/kg) and LOD (0.248 mg/kg) are greater than the associated human health and ecological PALs. The human health PALs are USEPA residential soil RSLs based on a target cancer risk of 1×10^{-6} (for hexachlorobenzene) or a non-cancer HQ of 0.1 (for hexachlorocyclopentadiene). The LOQ and LOD do not exceed residential soil RSLs adjusted for a target cancer risk of 1×10^{-5} (2.1 mg/kg for hexachlorobenzene) or a non-cancer HQ of 1 (1.8 mg/kg for hexachlorocyclopentadiene), nor do they exceed industrial soil RSLs (0.96 mg/kg based on a target cancer risk of 1×10^{-6} for hexachlorobenzene and 0.75 mg/kg based on a non-cancer HQ of 0.1 for hexachlorocyclopentadiene) which are more applicable to the current and future Site use. The ecological PAL for hexachlorobenzene (0.079 mg/kg) is the USEPA (2018) Region 4 soil ESV and is protective of adverse effects in birds. ESVs protective of plants (10 mg/kg), soil invertebrates (10 mg/kg) and mammals (0.2 mg/kg) are also available from USEPA (2018). The LOQ and LOD do not exceed these ESVs for plants and soil invertebrates. The ecological PAL

for hexachlorocyclopentadiene (0.001 mg/kg) is the USEPA (2018) Region 4 soil ESV and is based on effects in soil invertebrates. ESVs protective of plants (10 mg/kg) and mammals (0.755 mg/kg) are also available from USEPA (2018). The LOQ and LOD do not exceed the ESVs for these other receptor groups. Therefore, the laboratory limits are considered appropriate for meeting project DQOs.

- N-nitroso-di-n-propylamine in Soil – The LOQ (0.33 mg/kg) and LOD (0.248 mg/kg) are greater than the human health PAL (0.078 mg/kg), which is the USEPA residential soil RSL based on a target cancer risk of 1×10^{-6}. The LOQ and LOD do not exceed the residential soil RSL adjusted for a target cancer risk of 1×10^{-5} (0.78 mg/kg), nor do they exceed the industrial soil RSL (0.33 mg/kg, based on a target cancer risk of 1×10^{-6}) which is more applicable to the current and future Site use. Therefore, the laboratory limits for n-nitroso-di-n-propylamine in soil are considered appropriate for meeting project DQOs.

- Pentachlorophenol in Soil – The LOQ (0.82 mg/kg) and LOD (0.615 mg/kg) are greater than the human health PAL (0.23 mg/kg), which is the USEPA residential soil RSL based on a target cancer risk of 1×10^{-6}. The LOQ and LOD do not exceed the residential soil RSL adjusted for a target cancer risk of 1×10^{-5} (2.3 mg/kg), nor do they exceed the industrial soil RSL (4.0 mg/kg, based on a target cancer risk of 1×10^{-6}) which is more applicable to the current and future Site use. Therefore, the laboratory limits for pentachlorophenol in soil are considered appropriate for meeting project DQOs.

- 2,4-Dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 2-chlorophenol, 2-methylphenol, 2-nitroaniline, 3,3-dichlorobenzidine, atrazine, bis(2-ethylhexyl)phthalate, carbazole, di-n-butylphthalate, hexachlorobutadiene, and hexachloroethane in Soil – The LOQ and LOD are less than human health PALs but greater than the associated ecological PALs, which are the USEPA (2018) Region 4 soil ESVs. The selected ESV for each constituent is the lowest of available screening levels protective of adverse effects in plants, soil invertebrates, mammals and birds (USEPA, 2018). For all the above listed SVOCs except 2,4-dimethylphenol, atrazine and hexachlorobutadiene, ESVs that are greater than the LOQ and LOD are available for the other receptor groups in the USEPA (2018) Region 4 guidance document. While there is some uncertainty where the ecological PALs are less than the LOQ and LOD, the laboratory limits are considered appropriate for meeting project DQOs.

- 1,1'-Biphenyl, 1,2,4,5-tetrachlorobenzene, 1,4-dioxane, 2,4,6-trichlorophenol, 2,4-dinitrotoluene, 2,6-dinitrotoluene, 3,3'-dichlorobenzidine, 4,6-dinitro-2-methylphenol, 4-chloroaniline, atrazine, bis(2-chloroethyl)ether, dibenzofuran, hexachlorobenzene, hexachlorobutadiene, hexachlorocyclopentadiene, hexachloroethane, nitrobenzene, N-nitroso-di-n-propylamine, and pentachlorophenol in Groundwater – The LOQ and LOD are greater than the associated PALs, which are USEPA residential tapwater RSLs based on a target cancer risk of 1×10^{-6} or a non-cancer HQ of 0.1. The LODs do not exceed tapwater RSLs adjusted for a non-cancer HQ of 1 for 2,4,6-trichlorophenol and dibenzofuran, and they do not exceed tapwater RSLs adjusted for a non-cancer HQ of 10 for 1,1-biphenyl and 1,2,4,5-tetrachlorobenzene. The LOQs for 1,4-dioxane, 2,4-dinitrotoluene, 3,3'-dichlorobenzidine, 4-chloroaniline, atrazine, hexachlorobutadiene, hexachloroethane and nitrobenzene are in the same order of magnitude but do not exceed tapwater RSLs adjusted for a target cancer risk of 1×10^{-4}. The LOQ (10 µg/L) and LOD (7.5 µg/L) for hexachlorocyclopentadiene do not exceed the MCL (50 µg/L). MCLs are not available for the remaining listed SVOCs, except atrazine (3 µg/L) and hexachlorobenzene (1 µg/L). These laboratory limits are considered appropriate for meeting project DQOs as the use of the tapwater RSLs is conservative since drinking water is provided from a separate municipal supply.

- Benzo(a)anthracene, benzo(a)pyrene, and dibenzo(a,h)anthracene in Groundwater – The LOQ (0.2 µg/L) and LOD (0.1 µg/L) for the SIM analysis in groundwater are greater than the

associated PALs, which are USEPA residential tapwater RSLs based on a target cancer risk of 1×10^{-6}. The LOQ and LOD do not exceed tapwater RSLs adjusted for a target cancer risk of 1×10^{-5}. Additionally, the LOQ and LOD are equal to or less than the MCL (0.2 µg/L) for benzo(a)pyrene. Therefore, the laboratory limits are considered appropriate for meeting project DQOs.

Metals

- Thallium in Soil – The ICP/AES LOQ (1.5 mg/kg) and LOD (0.5 mg/kg) are greater than the human health PAL (0.078 mg/kg) and ecological PAL (0.05 mg/kg). The human health PAL is the USEPA residential soil RSL based on a non-cancer HQ of 0.1. The LOD does not exceed the residential soil RSL adjusted for a non-cancer HQ of 1 (0.78 mg/kg), or the industrial soil RSL (1.2 mg/kg, based on a non-cancer HQ of 0.1) which is more applicable to the current and future Site use. The ecological PAL is the USEPA Region 4 soil ESV based on effects in plants. ESVs for mammals (0.42 mg/kg) and birds (4.5 mg/kg) are also available from USEPA (2018). The LOQ and LOD do not exceed the avian ESV. Therefore, the laboratory limits for thallium in soil are considered appropriate for meeting project DQOs.

- Arsenic and thallium in Groundwater – The ICP/MS LOQ and LOD are greater than the associated PALs, which are USEPA residential tapwater RSLs based on a target cancer risk of 1×10^{-6} (for arsenic) or a non-cancer HQ of 0.1 (for thallium). The LOQ (5.0 µg/L) and LOD (4.0 µg/L) for arsenic do not exceed the tapwater RSL adjusted for a target cancer risk of 1×10^{-4} (5.2 µg/L). The LOQ (1.0 µg/L) and LOD (0.4 µg/L) for thallium do not exceed the tapwater RSL adjusted for a non-cancer HQ of 10 (2.0 µg/L). The LOQ and LOD also do not exceed applicable MCLs. Therefore, the laboratory limits are considered appropriate for meeting project DQOs.

The worksheets that follow include:

- Worksheet #15-1: Volatile Organic Compounds in Soil
- Worksheet #15-2: Volatile Organic Compounds in Groundwater
- Worksheet #15-3: Semivolatile Organic Compounds in Soil
- Worksheet #15-4: Semivolatile Organic Compounds in Groundwater
- Worksheet #15-5: Polychlorinated Biphenyls in Soil
- Worksheet #15-6: Hydrazines in Soil
- Worksheet #15-7: Metals in Soil
- Worksheet #15-8: Metals in Groundwater
- Worksheet #15-9: Total Organic Carbon in Soil
- Worksheet #15-10: Hardness in Groundwater

Worksheet #15-1: Volatile Organic Compounds in Soil

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Volatile Organic Compounds (8260C) (milligram per kilogram [mg/kg])									
1,1,1-Trichloroethane	71-55-6	810	EPA RSL[1]	0.04	EPA R4	0.013	0.005	0.0025	0.00042
1,1,2,2-Tetrachloroethane	79-34-5	0.6	EPA RSL[1]	0.127	EPA R4	0.042	0.005	0.0025	0.00084
1,1,2-Trichloroethane	79-00-5	0.15	EPA RSL[1]	0.32	EPA R4	0.05	0.005	0.0025	0.00097
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	670	EPA RSL[1]	NA	NA	223	0.005	0.0025	0.0009
1,1-Dichloroethane	75-34-3	3.6	EPA RSL[1]	0.14	EPA R4	0.047	0.005	0.0025	0.00167
1,1-Dichloroethene	75-35-4	23	EPA RSL[1]	0.04	EPA R4	0.013	0.005	0.0025	0.00093
1,2,3-Trichlorobenzene	87-61-6	6.3	EPA RSL[1]	20	EPA R4	2.1	0.005	0.0025	0.00076
1,2,4-Trichlorobenzene	120-82-1	5.8	EPA RSL[1]	0.27	EPA R4	0.090	0.005	0.0025	0.00079
1,2-Dibromo-3-chloropropane	96-12-8	0.0053	EPA RSL[1]	NA	NA	0.005	0.005	0.0025	0.00153
1,2-Dibromoethane	106-93-4	0.036	EPA RSL[1]	NA	NA	0.012	0.005	0.0025	0.0012
1,2-Dichlorobenzene	95-50-1	180	EPA RSL[1]	0.09	EPA R4	0.03	0.005	0.0025	0.00078
1,2-Dichloroethane	107-06-2	0.46	EPA RSL[1]	0.4	EPA R4	0.13	0.005	0.0025	0.00101
1,2-Dichloropropane	78-87-5	1.6	EPA RSL[1]	0.28	EPA R4	0.093	0.005	0.0025	0.00138
1,3-Dichlorobenzene	541-73-1	NA	NA	0.08	EPA R4	0.027	0.005	0.0025	0.00062
1,4-Dichlorobenzene	106-46-7	2.6	EPA RSL[1]	0.88	EPA R4	0.29	0.005	0.0025	0.00044
2-Butanone	78-93-3	2,700	EPA RSL[1]	1	EPA R4	0.33	0.025	0.0125	0.00593
2-Hexanone	591-78-6	20	EPA RSL[1]	0.36	EPA R4	0.12	0.025	0.0125	0.00477
4-Methyl-2-pentanone	108-10-1	3,300	EPA RSL[1]	NA	NA	1,100	0.025	0.0125	0.00585
Acetone	67-64-1	6,100	EPA RSL[1]	1.2	EPA R4	0.40	0.025	0.0125	0.0051
Benzene	71-43-2	1.2	EPA RSL[1]	0.12	EPA R4	0.04	0.005	0.0025	0.00092
Bromochloromethane	74-97-5	15	EPA RSL[1]	NA	NA	5	0.005	0.0025	0.00091

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Bromodichloromethane	75-27-4	0.29	EPA RSL[1]	NA	NA	0.097	0.005	0.0025	0.0006
Bromoform	75-25-2	19	EPA RSL[1]	0.07	EPA R4	0.023	0.005	0.0025	0.0007
Bromomethane	74-83-9	0.68	EPA RSL[1]	0.002	EPA R4	0.01	0.01	0.005	0.00108
Carbon Disulfide	75-15-0	77	EPA RSL[1]	0.005	EPA R4	0.005	0.005	0.0025	0.00078
Carbon Tetrachloride	56-23-5	0.65	EPA RSL[1]	0.05	EPA R4	0.017	0.005	0.0025	0.00126
Chlorobenzene	108-90-7	28	EPA RSL[1]	2.4	EPA R4	0.80	0.005	0.0025	0.00051
Chloroethane	75-00-3	1,400	EPA RSL[1]	NA	NA	467	0.01	0.005	0.00132
Chloroform	67-66-3	0.32	EPA RSL[1]	0.05	EPA R4	0.017	0.005	0.0025	0.00035
Chloromethane	74-87-3	11	EPA RSL[1]	NA	NA	3.7	0.01	0.005	0.00139
cis-1,2-Dichloroethene	156-59-2	16	EPA RSL[1]	0.04	EPA R4	0.013	0.005	0.0025	0.00091
cis-1,3-Dichloropropene	10061-01-5	1.8	EPA RSL[1]	NA	NA	0.6	0.005	0.0025	0.00072
Cyclohexane	110-82-7	650	EPA RSL[1]	NA	NA	217	0.005	0.0025	0.00138
Dibromochloromethane	124-48-1	8.3	EPA RSL[1]	NA	NA	2.8	0.005	0.0025	0.00103
Dichlorodifluoromethane	75-71-8	8.7	EPA RSL[1]	NA	NA	2.9	0.01	0.005	0.00092
Ethylbenzene	100-41-4	5.8	EPA RSL[1]	0.27	EPA R4	0.09	0.005	0.0025	0.00065
Isopropylbenzene	98-82-8	190	EPA RSL[1]	0.04	EPA R4	0.013	0.005	0.0025	0.00092
Methyl Acetate	79-20-9	7,800	EPA RSL[1]	NA	NA	2,600	0.005	0.003	0.00274
Methyl Tertiary Butyl Ether	1634-04-4	47	EPA RSL[1]	NA	NA	15.7	0.005	0.0025	0.00106
Methylcyclohexane	108-87-2	NA	NA	NA	NA	0.025	0.005	0.0025	0.00096
Methylene Chloride	75-09-2	35	EPA RSL[1]	0.21	EPA R4	0.07	0.025	0.0125	0.00793
Styrene	100-42-5	600	EPA RSL[1]	1.2	EPA R4	0.4	0.005	0.0025	0.00051
Tetrachloroethene	127-18-4	8.1	EPA RSL[1]	0.06	EPA R4	0.02	0.005	0.0025	0.00123
Toluene	108-88-3	490	EPA RSL[1]	0.15	EPA R4	0.05	0.005	0.0025	0.00136
trans-1,2-Dichloroethene	156-60-5	160	EPA RSL[1]	0.04	EPA R4	0.013	0.005	0.0025	0.00071
trans-1,3-Dichloropropene	10061-02-6	1.8	EPA RSL[1]	NA	NA	0.6	0.005	0.0025	0.00086

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Trichloroethene	79-01-6	0.41	EPA RSL[1]	0.06	EPA R4	0.02	0.005	0.0025	0.00059
Trichlorofluoromethane	75-69-4	2,300	EPA RSL[1]	16.4	EPA R4	5.5	0.01	0.005	0.00091
Vinyl Chloride	75-01-4	0.059	EPA RSL[1]	0.03	EPA R4	0.01	0.01	0.005	0.00087
m+p-Xylene	179601-23-1	55	EPA RSL[1]	0.1	EPA R4[5]	0.033	0.01	0.005	0.00167
o-Xylene	95-47-6	65	EPA RSL[1]	0.1	EPA R4[5]	0.033	0.005	0.0025	0.00128

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1657 **Worksheet #15-2: Volatile Organic Compounds in Groundwater**

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Volatile Organic Compounds (8260C) (µg/L)							
1,1,1-Trichloroethane	71-55-6	200	EPA MCL	66.7	1.0	0.5	0.20
1,1,2,2-Tetrachloroethane	79-34-5	0.076	EPA RSL[2]	1.0	1.0	0.5	0.38
1,1,2-Trichloroethane	79-00-5	0.041	EPA RSL[2]	1.0	1.0	0.5	0.33
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	24.2	EPA VISL[3]	8.07	1.0	0.5	0.31
1,1-Dichloroethane	75-34-3	2.8	EPA RSL[2]	1.0	1.0	0.5	0.21
1,1-Dichloroethene	75-35-4	7	EPA MCL	2.3	1.0	0.5	0.35
1,2,3-Trichlorobenzene	87-61-6	0.7	EPA RSL[2]	1.0	1.0	0.5	0.27
1,2,4-Trichlorobenzene	120-82-1	0.4	EPA RSL[2]	1.0	1.0	0.5	0.37
1,2-Dibromo-3-chloropropane	96-12-8	0.00033	EPA RSL[2]	1.0	1.0	0.75	0.50
1,2-Dibromoethane	106-93-4	0.0075	EPA RSL[2]	1.0	1.0	0.5	0.22

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
1,2-Dichlorobenzene	95-50-1	30	EPA RSL[2]	10	1.0	0.5	0.15
1,2-Dichloroethane	107-06-2	0.17	EPA RSL[2]	1.0	1.0	0.5	0.20
1,2-Dichloropropane	78-87-5	0.82	EPA RSL[2]	1.0	1.0	0.5	0.25
1,3-Dichlorobenzene	541-73-1	NA	NA	5.0	1.0	0.5	0.26
1,4-Dichlorobenzene	106-46-7	0.48	EPA RSL[2]	1.0	1.0	0.5	0.24
2-Butanone	78-93-3	560	EPA RSL[2]	187	5	2.5	1.31
2-Hexanone	591-78-6	3.8	EPA RSL[2]	5	5	2.5	1.70
4-Methyl-2-pentanone	108-10-1	630	EPA RSL[2]	210	5.0	2.5	1.32
Acetone	67-64-1	1,400	EPA RSL[2]	467	5.0	2.5	2.21
Benzene	71-43-2	0.46	EPA RSL[2]	1.0	1.0	0.5	0.26
Bromochloromethane	74-97-5	8.3	EPA RSL[2]	2.7	1.0	0.5	0.21
Bromodichloromethane	75-27-4	0.13	EPA RSL[2]	1.0	1.0	0.5	0.33
Bromoform	75-25-2	3.3	EPA RSL[2]	1.1	1.0	0.5	0.23
Bromomethane	74-83-9	0.75	EPA RSL[2]	2.0	2.0	1.0	0.49
Carbon Disulfide	75-15-0	81	EPA RSL[2]	27	1.0	0.5	0.25
Carbon Tetrachloride	56-23-5	0.415	EPA VISL[3]	1.0	1.0	0.5	0.22
Chlorobenzene	108-90-7	7.8	EPA RSL[2]	2.6	1.0	0.5	0.22
Chloroethane	75-00-3	2,100	EPA RSL[2]	700	2.0	1.0	0.55
Chloroform	67-66-3	0.22	EPA RSL[2]	1.0	1.0	0.5	0.32
Chloromethane	74-87-3	19	EPA RSL[2]	6.3	2.0	1.0	0.36
cis-1,2-Dichloroethene	156-59-2	3.6	EPA RSL[2]	1.2	1.0	0.5	0.21
cis-1,3-Dichloropropene	10061-01-5	0.47	EPA RSL[2]	1.0	1.0	0.5	0.19
Cyclohexane	110-82-7	102	EPA VISL[3]	34	1.0	0.5	0.31
Dibromochloromethane	124-48-1	0.87	EPA RSL[2]	1.0	1.0	0.5	0.30

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
Dichlorodifluoromethane	75-71-8	0.744	EPA VISL[3]	2.0	2.0	1.0	0.24
Ethylbenzene	100-41-4	1.5	EPA RSL[2]	1.0	1.0	0.5	0.21
Isopropylbenzene	98-82-8	45	EPA RSL[2]	15	1.0	0.5	0.23
Methyl Acetate	79-20-9	2,000	EPA RSL[2]	667	1.0	0.75	0.53
Methyl Tertiary Butyl Ether	1634-04-4	14	EPA RSL[2]	4.7	1.0	0.5	0.36
Methylcyclohexane	108-87-2	NA	NA	5.0	1.0	0.5	0.30
Methylene Chloride	75-09-2	5	EPA MCL	5.0	5.0	2.5	1.13
Styrene	100-42-5	100	EPA MCL	33.3	1.0	0.5	0.23
Tetrachloroethene	127-18-4	4.1	EPA RSL[2]	1.37	1.0	0.5	0.40
Toluene	108-88-3	110	EPA RSL[2]	36.7	1.0	0.5	0.27
trans-1,2-Dichloroethene	156-60-5	36	EPA RSL[2]	12	1.0	0.5	0.25
trans-1,3-Dichloropropene	10061-02-6	0.47	EPA RSL[2]	1.0	1.0	0.5	0.20
Trichloroethene	79-01-6	0.28	EPA RSL[2]	1.0	1.0	0.5	0.28
Trichlorofluoromethane	75-69-4	520	EPA RSL[2]	173	2.0	1.0	0.24
Vinyl Chloride	75-01-4	0.019	EPA RSL[2]	2.0	2.0	1.0	0.25
m+p-Xylene	108-38-3 / 106-42-3	19	EPA RSL[2]	6.3	2.0	1.0	0.59
o-Xylene	95-47-6	19	EPA RSL[2]	6.3	1.0	0.5	0.25

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1659 **Worksheet #15-3: Semivolatile Organic Compounds in Soil**

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Semivolatile Organic Compounds (8270D) (mg/kg)									
1,1'-Biphenyl	92-52-4	4.7	EPA RSL[1]	0.2	EPA R4	0.33	0.33	0.248	0.073
1,2,4,5-Tetrachlorobenzene	95-94-3	2.3	EPA RSL[1]	0.18	EPA R4	0.33	0.33	0.248	0.135
1,4-Dioxane	123-91-1	5.3	EPA RSL[1]	NA	NA	1.8	0.33	0.248	0.054
2,2'-oxybis(1-Chloropropane)	108-60-1	310	EPA RSL[1]	NA	NA	103	0.33	0.248	0.089
2,4,5-Trichlorophenol	95-95-4	630	EPA RSL[1]	4	EPA R4	1.3	0.82	0.615	0.155
2,4,6-Trichlorophenol	88-06-2	6.3	EPA RSL[1]	9.94	EPA R4	2.1	0.33	0.248	0.155
2,4-Dichlorophenol	120-83-2	19	EPA RSL[1]	0.05	EPA R4	0.33	0.33	0.248	0.150
2,4-Dimethylphenol	105-67-9	130	EPA RSL[1]	0.04	EPA R4	0.33	0.33	0.248	0.165
2,4-Dinitrophenol	51-28-5	13	EPA RSL[1]	0.061	EPA R4	0.82	0.82	0.615	0.377
2,4-Dinitrotoluene	121-14-2	1.7	EPA RSL[1]	6	EPA R4	0.57	0.33	0.248	0.085
2,6-Dinitrotoluene	606-20-2	0.36	EPA RSL[1]	4	EPA R4	0.33	0.33	0.248	0.079
2-Chloronaphthalene	91-58-7	480	EPA RSL[1]	NA	NA	160	0.33	0.248	0.087
2-Chlorophenol	95-57-8	39	EPA RSL[1]	0.06	EPA R4	0.33	0.33	0.248	0.164
2-Methylnaphthalene	91-57-6	24	EPA RSL[1]	See Total LMW PAH	EPA R4	8.0	0.33	0.248	0.092
2-Methylphenol	95-48-7	320	EPA RSL[1]	0.1	EPA R4	0.33	0.33	0.248	0.200
2-Nitroaniline	88-74-4	63	EPA RSL[1]	0.02	EPA R4	0.82	0.82	0.615	0.075
2-Nitrophenol	88-75-5	NA	NA	NA	NA	1.65	0.33	0.248	0.167
3,3'-Dichlorobenzidine	91-94-1	1.2	EPA RSL[1]	0.03	EPA R4	0.33	0.33	0.248	0.114
3-Nitroaniline	99-09-2	NA	NA	NA	NA	4.1	0.82	0.615	0.094
4,6-Dinitro-2-methylphenol	534-52-1	0.51	EPA RSL[1]	NA	NA	0.82	0.82	0.615	0.337

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
4-Bromophenyl-phenylether	101-55-3	NA	NA	NA	NA	1.65	0.33	0.248	0.085
4-Chloro-3-methylphenol	59-50-7	630	EPA RSL[1]	NA	NA	210	0.33	0.248	0.166
4-Chloroaniline	106-47-8	2.7	EPA RSL[1]	1	EPA R4	0.33	0.33	0.248	0.119
4-Chlorophenyl-phenylether	7005-72-3	NA	NA	NA	NA	1.65	0.33	0.248	0.078
3&4-Methylphenol	108-39-4 / 106-44-5	320	EPA RSL[1]	NA	NA	107	0.33	0.248	0.187
4-Nitroaniline	100-01-6	25	EPA RSL[1]	NA	NA	8.3	0.82	0.615	0.134
4-Nitrophenol	100-02-7	NA	NA	5.12	EPA R4	1.7	0.82	0.615	0.309
Acenaphthene	83-32-9	360	EPA RSL[1]	See Total LMW PAH	EPA R4	9.7	0.33	0.248	0.065
Acenaphthylene	208-96-8	NA	NA	See Total LMW PAH	EPA R4	9.7	0.33	0.248	0.070
Acetophenone	98-86-2	780	EPA RSL[1]	NA	NA	260	0.33	0.248	0.178
Anthracene	120-12-7	1,800	EPA RSL[1]	See Total LMW PAH	EPA R4	9.7	0.33	0.248	0.084
Atrazine	1912-24-9	2.4	EPA RSL[1]	0.00005	EPA R4	0.33	0.33	0.248	0.091
Benzaldehyde	100-52-7	170	EPA RSL[1]	NA	NA	57	0.33	0.248	0.120
Benzo(a)anthracene	56-55-3	1.1	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.086
Benzo(a)pyrene	50-32-8	0.11	EPA RSL[1]	See Total HMW PAH	EPA R4	0.33	0.33	0.248	0.093
Benzo(b)fluoranthene	205-99-2	1.1	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.134
Benzo(g,h,i)perylene	191-24-2	NA	NA	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.104

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Benzo(k)fluoranthene	207-08-9	11	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.083
bis(2-Chloroethoxy)methane	111-91-1	19	EPA RSL[1]	NA	NA	6.3	0.33	0.248	0.096
bis(2-Chloroethyl)ether	111-44-4	0.23	EPA RSL[1]	NA	NA	0.33	0.33	0.248	0.081
bis(2-Ethylhexyl)phthalate	117-81-7	39	EPA RSL[1]	0.02	EPA R4	0.33	0.33	0.248	0.098
Butylbenzylphthalate	85-68-7	290	EPA RSL[1]	0.59	EPA R4	0.33	0.33	0.248	0.093
Caprolactam	105-60-2	3,100	EPA RSL[1]	NA	NA	1033	0.33	0.248	0.144
Carbazole	86-74-8	NA	NA	0.07	EPA R4	0.33	0.33	0.248	0.111
Chrysene	218-01-9	110	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.095
Dibenzo(a,h)anthracene	53-70-3	0.11	EPA RSL[1]	See Total HMW PAH	EPA R4	0.33	0.33	0.248	0.128
Dibenzofuran	132-64-9	7.3	EPA RSL[1]	0.15	EPA R4	0.33	0.33	0.248	0.079
Diethylphthalate	84-66-2	5,100	EPA RSL[1]	0.25	EPA R4	0.33	0.33	0.248	0.080
Dimethylphthalate	131-11-3	NA	NA	0.35	EPA R4	0.33	0.33	0.248	0.078
Di-n-butylphthalate	84-74-2	630	EPA RSL[1]	0.011	EPA R4	0.33	0.33	0.248	0.101
Di-n-octylphthalate	117-84-0	63	EPA RSL[1]	0.91	EPA R4	0.33	0.33	0.248	0.211
Fluoranthene	206-44-0	240	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.106
Fluorene	86-73-7	240	EPA RSL[1]	See Total LMW PAH	EPA R4	9.7	0.33	0.248	0.081
Hexachlorobenzene	118-74-1	0.21	EPA RSL[1]	0.079	EPA R4	0.33	0.33	0.248	0.082
Hexachlorobutadiene	87-68-3	1.2	EPA RSL[1]	0.009	EPA R4	0.33	0.33	0.248	0.083
Hexachlorocyclopentadiene	77-47-4	0.18	EPA RSL[1]	0.001	EPA R4	0.33	0.33	0.248	0.082
Hexachloroethane	67-72-1	1.8	EPA RSL[1]	0.024	EPA R4	0.33	0.33	0.248	0.096

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Indeno(1,2,3-cd)pyrene	193-39-5	1.1	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.122
Isophorone	78-59-1	570	EPA RSL[1]	NA	NA	190	0.33	0.248	0.075
Naphthalene	91-20-3	3.8	EPA RSL[1]	See Total LMW PAH	EPA R4	1.3	0.33	0.248	0.087
Nitrobenzene	98-95-3	5.1	EPA RSL[1]	2.2	EPA R4	0.73	0.33	0.248	0.091
N-Nitrosodiphenylamine	86-30-6	110	EPA RSL[1]	0.545	EPA R4	0.33	0.33	0.248	0.219
N-Nitroso-di-n-propylamine	621-64-7	0.078	EPA RSL[1]	NA	NA	0.33	0.33	0.248	0.083
Pentachlorophenol	87-86-5	11.0	EPA RSL[1]	NA	NA	0.82	0.82	0.615	0.237
Phenanthrene	85-01-8	NA	NA	See Total LMW PAH	EPA R4	9.7	0.33	0.248	0.083
Phenol	108-95-2	1,900	EPA RSL[1]	0.79	EPA R4	0.33	0.33	0.248	0.156
Pyrene	129-00-0	180	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.33	0.248	0.101
Total High Molecular Weight PAHs				1.1	USEPA EcoSSL				
Total Low Molecular Weight PAHs				29	USEPA EcoSSL				
Analytical Group: Semivolatile Organic Compounds (8270D-SIM) (mg/kg)									
2-Methylnaphthalene	91-57-6	24	EPA RSL[1]	See Total LMW PAH	EPA R4	8	0.020	0.010	0.0022
Acenaphthene	83-32-9	360	EPA RSL[1]	See Total LMW PAH	EPA R4	9.7	0.020	0.010	0.0015
Acenaphthylene	208-96-8	NA	NA	See Total LMW PAH	EPA R4	9.7	0.020	0.010	0.0012
Anthracene	120-12-7	1,800	EPA RSL[1]	See Total LMW PAH	EPA R4	9.7	0.020	0.010	0.0012

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Benzo(a)anthracene	56-55-3	1.1	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0019
Benzo(a)pyrene	50-32-8	0.11	EPA RSL[1]	See Total HMW PAH	EPA R4	0.037	0.020	0.010	0.0033
Benzo(b)fluoranthene	205-99-2	1.1	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0024
Benzo(g,h,i)perylene	191-24-2	NA	NA	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0020
Benzo(k)fluoranthene	207-08-9	11	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0031
Chrysene	218-01-9	110	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0017
Dibenzo(a,h)anthracene	53-70-3	0.11	EPA RSL[1]	See Total HMW PAH	EPA R4	0.037	0.020	0.010	0.0018
Fluoranthene	206-44-0	240	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0018
Fluorene	86-73-7	240	EPA RSL[1]	See Total LMW PAH	EPA R4	9.7	0.020	0.010	0.0032
Indeno(1,2,3-cd)pyrene	193-39-5	1.1	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.0019
Naphthalene	91-20-3	3.8	EPA RSL[1]	See Total LMW PAH	EPA R4	1.3	0.020	0.010	0.0026
Phenanthrene	85-01-8	NA	NA	See Total LMW PAH	EPA R4	9.7	0.020	0.010	0.0018
Pyrene	129-00-0	180	EPA RSL[1]	See Total HMW PAH	EPA R4	0.37	0.020	0.010	0.021
Total High Molecular Weight PAHs	-			1.1	USEPA EcoSSL				
Total Low Molecular Weight PAHs	-			29	USEPA EcoSSL				

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Worksheet #15-4: Semivolatile Organic Compounds in Groundwater

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Semivolatile Organic Compounds (8270D) (µg/L)							
1,1'-Biphenyl	92-52-4	0.083	EPA RSL[2]	10	10	7.5	2.7
1,2,4,5-Tetrachlorobenzene	95-94-3	0.17	EPA RSL[2]	10	10	7.5	1.8
1,4-Dioxane	123-91-1	0.46	EPA RSL[2]	10	10	7.5	1.8
2,2'-oxybis(1-Chloropropane)	108-60-1	71	EPA RSL[2]	23.7	10	7.5	2.1
2,3,4,6-Tetrachlorophenol	58-90-2	24	EPA RSL[2]	10	10	7.5	2.7
2,4,5-Trichlorophenol	95-95-4	120	EPA RSL[2]	40	25	18.8	3.6
2,4,6-Trichlorophenol	88-06-2	1.2	EPA RSL[2]	10	10	7.5	2.7
2,4-Dichlorophenol	120-83-2	4.6	EPA RSL[2]	10	10	7.5	3.0
2,4-Dimethylphenol	105-67-9	36	EPA RSL[2]	12	10	7.5	4.4
2,4-Dinitrophenol	51-28-5	3.9	EPA RSL[2]	25	25	18.8	1
2,4-Dinitrotoluene	121-14-2	0.24	EPA RSL[2]	10	10	7.5	2.2
2,6-Dinitrotoluene	606-20-2	0.049	EPA RSL[2]	10	10	7.5	2.0
2-Chloronaphthalene	91-58-7	75	EPA RSL[2]	25	10	7.5	2.9
2-Chlorophenol	95-57-8	9.1	EPA RSL[2]	10	10	7.5	3.2
2-Methylphenol	95-48-7	93	EPA RSL[2]	31	10	7.5	3.8
2-Nitroaniline	88-74-4	19	EPA RSL[2]	25	25	18.8	1.8
2-Nitrophenol	88-75-5	NA	NA	50	10	7.5	2.7
3,3'-Dichlorobenzidine	91-94-1	0.13	EPA RSL[2]	10	10	7.5	1.1
3-Nitroaniline	99-09-2	NA	NA	125	25	18.8	1.5
4,6-Dinitro-2-methylphenol	534-52-1	0.15	EPA RSL[2]	25	25	18.8	2.0
4-Bromophenyl-phenylether	101-55-3	NA	NA	50	10	7.5	1.9
4-Chloro-3-methylphenol	59-50-7	140	EPA RSL[2]	46.7	10	7.5	3.6
4-Chloroaniline	106-47-8	0.37	EPA RSL[2]	10	10	7.5	1.9
4-Chlorophenyl-phenylether	7005-72-3	NA	NA	50	10	7.5	2.2

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
3&4-Methylphenol	108-39-4 / 106-44-5	93	EPA RSL[2]	31	10	7.5	5.6
4-Nitroaniline	100-01-6	3.8	EPA RSL[2]	25	25	18.8	1.6
4-Nitrophenol	100-02-7	NA	NA	125	25	18.8	1.8
Acetophenone	98-86-2	190	EPA RSL[2]	63	10	7.5	3.9
Atrazine	1912-24-9	0.3	EPA RSL[2]	10	10	7.5	3.3
Benzaldehyde	100-52-7	19	EPA RSL[2]	10	10	7.5	1.0
bis(2-Chloroethoxy)methane	111-91-1	5.9	EPA RSL[2]	10	10	7.5	2.1
bis(2-Chloroethyl)ether	111-44-4	0.014	EPA RSL[2]	10	10	7.5	2.0
bis(2-Ethylhexyl)phthalate	117-81-7	5.6	EPA RSL[2]	10	10	7.5	1.8
Butylbenzylphthalate	85-68-7	16	EPA RSL[2]	10	10	7.5	1.9
Caprolactum	105-60-2	990	EPA RSL[2]	330	10	7.5	0.4
Carbazole	86-74-8	NA	NA	50	10	7.5	2.1
Dibenzofuran	132-64-9	0.79	EPA RSL[2]	10	10	7.5	1.6
Diethylphthalate	84-66-2	1,500	EPA RSL[2]	500	10	7.5	2.0
Dimethylphthalate	131-11-3	NA	NA	50	10	7.5	2.01
Di-n-butylphthalate	84-74-2	90	EPA RSL[2]	30	10	7.5	2.5
Di-n-octylphthalate	117-84-0	20	EPA RSL[2]	10	10	7.5	1.8
Hexachlorobenzene	118-74-1	0.0098	EPA RSL[2]	10	10	7.5	2.1
Hexachlorobutadiene	87-68-3	0.14	EPA RSL[2]	10	10	7.5	1.8
Hexachlorocyclopentadiene	77-47-4	0.041	EPA RSL[2]	10	10	7.5	1.2
Hexachloroethane	67-72-1	0.33	EPA RSL[2]	10	10	7.5	2.3
Isophorone	78-59-1	78	EPA RSL[2]	26	10	7.5	1.7
Nitrobenzene	98-95-3	0.14	EPA RSL[2]	10	10	7.5	3.1
N-Nitrosodiphenylamine	86-30-6	12	EPA RSL[2]	10	10	7.5	3.7
N-Nitroso-di-n-propylamine	621-64-7	0.011	EPA RSL[2]	10	10	7.5	1.9
Pentachlorophenol	111-44-4	0.014	EPA RSL[2]	25	25	18.8	2.3

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
Phenol	108-95-2	580	EPA RSL[2]	193	10	7.5	1.8
Analytical Group: Semivolatile Organic Compounds (8270D-SIM) (µg/L)							
2-Methylnaphthalene	91-57-6	3.6	EPA RSL[2]	1.2	0.20	0.10	0.077
Acenaphthene	83-32-9	53	EPA RSL[2]	17.7	0.20	0.10	0.064
Acenaphthylene	208-96-8	NA	NA	1.0	0.20	0.10	0.054
Anthracene	120-12-7	180	EPA RSL[2]	60	0.20	0.10	0.044
Benzo(a)anthracene	56-55-3	0.03	EPA RSL[2]	0.20	0.20	0.10	0.046
Benzo(a)pyrene	50-32-8	0.025	EPA RSL[2]	0.20	0.20	0.10	0.066
Benzo(b)fluoranthene	205-99-2	0.25	EPA RSL[2]	0.20	0.20	0.10	0.089
Benzo(g,h,i)perylene	191-24-2	NA	NA	1.0	0.20	0.10	0.065
Benzo(k)fluoranthene	207-08-9	2.5	EPA RSL[2]	0.83	0.20	0.10	0.049
Chrysene	218-01-9	25	EPA RSL[2]	8.3	0.20	0.10	0.036
Dibenzo(a,h)anthracene	53-70-3	0.025	EPA RSL[2]	0.20	0.20	0.10	0.070
Fluoranthene	206-44-0	80	EPA RSL[2]	26.7	0.20	0.10	0.073
Fluorene	86-73-7	29	EPA RSL[2]	9.7	0.20	0.10	0.061
Indeno(1,2,3-cd)pyrene	193-39-5	0.25	EPA RSL[2]	0.20	0.20	0.10	0.052
Naphthalene	91-20-3	0.17	EPA RSL[2]	0.20	0.20	0.10	0.064
Phenanthrene	85-01-8	NA	NA	1.0	0.20	0.10	0.051
Pyrene	129-00-0	12	EPA RSL[2]	4	0.20	0.10	0.059

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1663 **Worksheet #15-5: Polychlorinated Biphenyls in Soil**

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Polychlorinated Biphenyls (PCBs) (8082A) (micrograms per kilogram [µg/kg])									
PCB-1016	12674-11-2	0.41	EPA RSL[1]	NA	NA	0.14	0.017	0.0085	0.0060
PCB-1221	11104-28-2	0.20	EPA RSL[1]	NA	NA	0.067	0.017	0.0085	0.0079
PCB-1232	11141-16-5	0.17	EPA RSL[1]	NA	NA	0.057	0.017	0.01	0.0093
PCB-1242	53469-21-9	0.23	EPA RSL[1]	NA	NA	0.077	0.017	0.0085	0.0058
PCB-1248	12672-29-6	0.23	EPA RSL[1]	NA	NA	0.077	0.017	0.0085	0.0061
PCB-1254	11097-69-1	0.12	EPA RSL[1]	NA	NA	0.04	0.017	0.0085	0.0047
PCB-1260	11096-82-5	0.24	EPA RSL[1]	NA	NA	0.08	0.017	0.0085	0.0060
Total PCBs	1336-36-3	0.23	EPA RSL[1]	0.041	EPA R4	0.017	0.017	0.01	0.0093

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1665 **Worksheet #15-6: Hydrazines in Soil**

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Hydrazines (8315A) (mg/kg)									
1,1-Dimethylhydrazine	57-14-7	0.0057	EPA RSL[1]	NA	NA	0.005	0.005	0.004	0.002
Hydrazine	302-01-2	0.032	EPA RSL[1]	NA	NA	0.011	0.005	0.002	0.001
Methylhydrazine	60-34-4	0.1	EPA RSL[1]	NA	NA	0.033	0.005	0.004	0.002

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Worksheet #15-7: Metals in Soil

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Metals (6010C/7471A) (mg/kg)									
Aluminum	7429-90-5	7,700	EPA RSL[1]	Note 6	USEPA EcoSSL	2,567	30	10	0.7
Antimony	7440-36-0	3.1	EPA RSL[1]	0.27	USEPA EcoSSL	0.8	0.8	0.5	0.1
Arsenic	7440-38-2	0.68	EPA RSL[1]	18	USEPA EcoSSL	0.8	0.8	0.50	0.1
Barium	7440-39-3	1,500	EPA RSL[1]	330	USEPA EcoSSL	110	0.5	0.3	0.0
Beryllium	7440-41-7	16	EPA RSL[1]	21	USEPA EcoSSL	5.3	0.5	0.1	0.0
Cadmium	7440-43-9	7.1	EPA RSL[1]	0.36	USEPA EcoSSL	1.0	1.0	0.3	0.0
Calcium	7440-70-2	NA	NA	NA	NA	50	10	8.0	1.8
Chromium	7440-47-3	12,000	EPA RSL[1]	23	EPA R4	7.7	1.5	0.4	0.026
Cobalt	7440-48-4	2.3	EPA RSL[1]	13	USEPA EcoSSL	3.0	3.0	0.4	0.029
Copper	7440-50-8	310	EPA RSL[1]	28	USEPA EcoSSL	9.3	2.5	1.0	0.16
Iron	7439-89-6	5,500	EPA RSL[1]	Note 6	USEPA EcoSSL	1,833	10	8	1.4
Lead	7439-92-1	400	EPA RSL[1]	11	USEPA EcoSSL	3.7	0.50	0.4	0.087
Magnesium	7439-95-4	NA	NA	NA	NA	50	10	8.0	0.68
Manganese	7439-96-5	180	EPA RSL[1]	220	USEPA EcoSSL	60	0.50	0.4	0.16
Nickel	7440-02-0	150	EPA RSL[1]	38	USEPA EcoSSL	12.7	4.0	0.4	0.044

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Potassium	7440-09-7	NA	NA	NA	NA	500	100	50	2.9
Selenium	7782-49-2	39	EPA RSL[1]	0.52	USEPA EcoSSL	1.0	1.0	0.7	0.17
Silver	7440-22-4	39	EPA RSL[1]	4.2	USEPA EcoSSL	1.5	1.5	0.4	0.027
Sodium	7440-23-5	NA	NA	NA	NA	500	100	50	1.5
Thallium	7440-28-0	0.078	EPA RSL[1]	0.05	EPA R4	1.5	1.5	0.5	0.087
Vanadium	7440-62-2	39	EPA RSL[1]	7.8	USEPA EcoSSL	2.6	2.5	0.4	0.037
Zinc	7440-66-6	2,300	EPA RSL[1]	46	USEPA EcoSSL	15	2.5	1	0.17
Mercury	7439-97-6	2.3	EPA RSL[1]	0.013	EPA R4	0.033	0.033	0.017	0.0052
Analytical Group: Hexavalent Chromium (7196A) (mg/kg)									
Hexavalent Chromium	18540-29-9	0.30	EPA RSL[1]	0.34	EPA R4	0.40	0.40	0.24	0.12

Worksheet #15-8: Metals in Groundwater

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Metals (6020A/7470A) (micrograms per liter [µg/L])							
Aluminum	7429-90-5	2,000	EPA RSL[2]	667	100	40	4.4
Antimony	7440-36-0	0.78	EPA RSL[2]	1.0	1.0	0.5	0.054
Arsenic	7440-38-2	0.052	EPA RSL[2]	5.0	5.0	4.0	2.2
Barium	7440-39-3	380	EPA RSL[2]	127	2.0	1.0	0.27

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory	Laboratory	Laboratory
					LOQ	LOD	DL
Beryllium	7440-41-7	2.5	EPA RSL[2]	1.0	1.0	0.2	0.034
Cadmium	7440-43-9	0.92	EPA RSL[2]	1.0	1.0	0.2	0.030
Calcium	7440-70-2	NA	NA	500	100	80	20
Chromium	7440-47-3	2,200	EPA RSL[2]	733	4.0	3.0	0.22
Cobalt	7440-48-4	0.6	EPA RSL[2]	1.0	1.0	0.3	0.060
Copper	7440-50-8	80	EPA RSL[2]	27	3.0	2.0	0.18
Iron	7439-89-6	1,400	EPA RSL[2]	467	100	60	13
Lead	7439-92-1	15	EPA RSL[2]	5	1.0	0.5	0.074
Magnesium	7439-95-4	NA	NA	500	100	80	7.8
Manganese	7439-96-5	43	EPA RSL[2]	14	2.0	1.0	0.35
Nickel	7440-02-0	39	EPA RSL[2]	13	2.0	1.2	0.15
Potassium	7440-09-7	NA	NA	5000	1000	400	31
Selenium	7782-49-2	10	EPA RSL[2]	5.0	5.0	3.0	0.19
Silver	7440-22-4	9.4	EPA RSL[2]	3.1	1.0	0.4	0.050
Sodium	7440-23-5	NA	NA	5000	1000	400	18
Thallium	7440-28-0	0.02	EPA RSL[2]	1.0	1.0	0.4	0.060
Vanadium	7440-62-2	8.6	EPA RSL[2]	5.0	5.0	4.0	0.51
Zinc	7440-66-6	600	EPA RSL[2]	200	10	8	3.9
Mercury	7439-97-6	0.063	EPA RSL[2]	0.21	0.20	0.10	0.013
Analytical Group: Hexavalent Chromium (7196A) (µg/L)							
Hexavalent Chromium	18540-29-9	0.035	EPA RSL[2]	25	25	13	0.76

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Worksheet #15-9: Total Organic Carbon in Soil

Analyte	CAS #	Human Project Action Limit (PAL)	Human PAL Reference	Ecological PAL	Ecological PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
							Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Total Organic Carbon (Lloyd Kahn) (mg/kg)									
TOC	7440-44-0	NA	NA	NA	NA	2.0	0.4	0.3	0.085

Worksheet #15-10: Hardness in Groundwater

Analyte	CAS #	Project Action Limit (PAL)	PAL Reference	Practical Quantitation Limit (PQL)	Laboratory Achievable Detection Limits		
					Laboratory LOQ	Laboratory LOD	Laboratory DL
Analytical Group: Hardness SM2340B (mg/L)							
Hardness	NA	NA	NA	100	20	20	NA

Notes:

1. USEPA Regional Screening Levels (RSLs) for Residential Soil, as of May 2019 (USEPA, 2019). The RSLs protective at a cancer risk level of 1×10^{-6} or noncancer hazard quotient (HQ) of 0.1 are presented.
2. USEPA RSLs for Tapwater, as of May 2019 (USEPA, 2019). The RSLs protective at a cancer risk level of 1×10^{-6} or noncancer HQ of 0.1 are presented.
3. USEPA VISLs (USEPA, 2020). Groundwater VISLs protective at a cancer risk level of 1×10^{-6} or noncancer HQ of 0.1 are presented.
4. The PQLs listed are based on 1/3 of the lowest screening level except where that value is less than the LOQ. In circumstances when 1/3 of the lowest screening level is less than the LOQ, the PQL is set at the lab's LOQ due to limitations in the analytical method. When no screening level is listed, the PQL is set at 5-times the LOQ. Shaded rows represent analytes where the screening level is less than the LOQ, LOD, and DL. The laboratory achievable detection limits are based on those for Katahdin analytical methods, with the exception of hydrazines, which will be analyzed by Eurofins. If another laboratory performs the analyses the detection limits may differ.
5. USEPA Region 4 Soil Screening Value for total xylenes.
6. Aluminum and iron are pH-dependent; refer to USEPA EcoSSL documents for additional details.

SIM = selective ion monitoring
PQL = practical quantitation limit
MDL = method detection limit
LOD = limit of detection
LOQ = limit of quantitation

RSL = Regional Screening Level
MCL = Maximum Contaminant Level (for drinking water)
mg/kg = milligrams per kilogram
mg/L = milligrams per liter
µg/kg = microgram per kilogram
µg/l = microgram per liter
EcoSSL = Ecological Soil Screening Level
HMW = high molecular weight
LMW = low molecular weight
PAH = polycyclic aromatic hydrocarbon
USEPA = United States Environmental Protection Agency
VISL = Vapor Intrusion Screening Level

QAPP Worksheet #17: Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

The RI field activities are intended to determine the nature and extent of impacts associated with the former Launch Area to support human health and ecological risk assessment.

17.1 Overall Investigation Approach Rationale

This worksheet describes the design for data collection, and documents Step 7 of the DQO process. For each field task, this section summarizes the data gap, the rationale for data collection, and the approach. The general process for the investigation will consist of identifying and delineating potentially impacted areas of the former Launch Area using groundwater sampling and surface and subsurface soil sampling. SOPs will be followed during collection, transport, and analyses of samples. The SOPs provide procedural guidance in general accordance with industry standards/guidelines and regulatory requirements. The SOPs are included in **Appendix E**.

17.2 Groundwater Investigation

Review of historical groundwater data has determined that there is incomplete horizontal and vertical delineation of the groundwater flow and impacts from potential source areas. Existing groundwater data are also outdated. Due to the uncertainty of the exact locations of the samples, the reporting limits of some constituents, some constituents not sampled, and potential contaminant transport / migration over the past 20 years, data from previous evaluations are being used for guidance purposes only. The existing monitoring well network has questionable integrity and, as local lithology is not well-known, the existing well network may not be located or screened properly. Therefore, 21 new monitoring wells are proposed, and delineation of groundwater flow will be determined using 16 overburden and five bedrock wells. Overburden wells will be drilled and installed using hollow-stem auger drilling methods (including continuous split spoon sampling) to the top of bedrock, which is anticipated to be reached between approximately 15 and 20 feet bgs. Bedrock well locations are adjacent to five of the 16 proposed overburden wells, were selected based on upgradient or downgradient relation to the former Launch Area, and are spread across the site to allow for determination of flow direction. The field tasks associated with the groundwater investigation are outlined in the table below.

Table 17.2: Groundwater Investigation Tasks

Groundwater Investigation Task	Supporting Documents
Utility Survey	QAPP Appendix E SOP
Monitoring Well Abandonment	NYSDEC CP-43: Monitoring Well Decommissioning Policy (2009)
Monitoring Well Installation	USACE EM 1110-1-4000 Monitoring Well Design, Installation, & Documentation (1998) QAPP Appendix E SOP: Monitoring Well Installation
Monitoring Well Development and Water-Level Monitoring	USACE EM 1110-1-4000 Monitoring Well Design, Installation, & Documentation (1998) QAPP Appendix E SOPs: – Monitoring Well Development – Manual Water-Level Monitoring
Groundwater Sampling	USACE EM 1110-1-4000 Monitoring Well Design, Installation, & Documentation (1998) QAPP Appendix E SOPs: – Groundwater and Soil Sampling Equipment Decontamination – Low-Flow Groundwater Purging and Sampling Procedures – In-Situ and Ex-Situ Water Quality Parameters – Sample Chain of Custody – Slug Testing – Investigation-Derived Waste Handling and Storage Arcadis Hazmat Shipping Handbook (2018)

17.3 Utility Survey

A utility survey will be performed to identify buried and overhead infrastructure that may be encountered during ground-intrusive activities, and to prevent damage to infrastructure assets and physical harm to field personnel and on-site workers. The utility survey will include:

- Notification of New York State Dig Safe 811;
- Completion of the Utility Location checklist provided in the field forms (**Appendix F**); and
- Identification of a minimum of three reliable lines of evidence, including:
    - Private utility location, such as ground penetrating radar, electromagnetic geophysical techniques, and/or other appropriate methods of detection;
    - Soft dig methods (i.e., air-knife) for clearing locations during drilling activities;
    - Review of public records, utility maps, and existing as-built-drawings; and
    - Site inspection of overhead utility infrastructure or ground infrastructure that indicates a buried utility.

17.4 Existing Groundwater Monitoring Well Abandonment

Groundwater monitoring wells installed during previous investigations are approximately 20 to 30 years old. During the site visit, three wells were identified (GZA-MW2, GZA-MW-4, ME-MW3), and seven wells could not be located. Due to the age, condition, and uncertainty of the screening depth interval, the wells will be abandoned per the NYSDEC CP-43: Groundwater Monitoring Well Decommissioning Policy (2009) during the site drilling activities. The locations of the wells are shown on **Appendix A: Figure 5**. The well abandonment SOP is included in **Appendix E**.

Table 17.4: Construction Details of Wells to Be Abandoned

Well ID	Top of Overburden (ft bgs)	Top of Weathered Shale (ft bgs)	Top of Competent Rock (ft bgs)	Depth of Boring (ft bgs)	Length of Well Screen (ft bgs)	Top of Sandpack (ft bgs)	Bottom of Sandpack (ft bgs)	Diameter of Well Screen and Riser (inch))
GZA-MW-2	0.5 ft	9	16.0 ft	25ft	10 ft	11	25	0.16
GZA MW-4	0.5	-	18 ft	31 ft	9 ft	19	31	0.16
MW-ME3	1	20	22.3	22	10	10	22.3	0.16

17.5 Groundwater Monitoring Well Installation

The monitoring well installations will be conducted in general accordance with the following standards:

- USACE EM 1110-1-4000 Monitoring Well Design, Installation, & Documentation (1998);
- American Society of Testing and Materials (ASTM) D5092 – Standard Practice for Design and Installation of Groundwater Monitoring Wells (ASTM D5092);
- ASTM Method D 2113-99 - Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation; and
- New York Department of Transportation (NYSDOT) Rock Core Evaluation Manual (2015).

Twenty-one new monitoring wells are proposed (**Appendix A: Figure 6**), and delineation of groundwater flow will be determined using 16 overburden and five bedrock wells. The 16 overburden monitoring wells will be drilled using 6-inch inner-diameter hollow-stem augers. Overburden materials at each location will be logged by the field geologist in accordance with USCS. If present, weathered bedrock identification will be based on the presence of structure/bedding indicative of the source rock. Each well will be screened from the bottom of the boring using 10 feet of 2-inch inner diameter, machine-slotted polyvinyl chloride (PVC) 0.010-inch screen and constructed in accordance with the procedures presented in the QAPP. The five bedrock wells will be drilled adjacent to five overburden wells and installed using HQ™ core barrel for the bedrock portion of the well down to the first water-bearing zone. Rock quality will be characterized for geology and rock quality (i.e., rock quality designation [RQD]) and evaluated to quantity the magnitude of fractures. It is assumed that bedrock monitoring wells will be completed with screens since the competency of the shallow bedrock is unknown based on available information. Stick-up monitoring wells will have bollards installed around them for protection.

1778 **Table 17.5: Rationale for Groundwater Sampling Locations**

Groundwater Sampling Location	Rationale for Sampling Location
MW1OB/ MW1BR, MW9OB/ MW9BR, MW10	These locations are representative of groundwater flowing from the landfill area towards the west side of the investigation area.
MW2, MW3	These locations are representative of historical activities associated with the missile assembly and testing building.
MW5OB/MW5BR	This location is representative of historical activities associated with the former men's barrack.
MW4OB/ MW4BR, MW14	The locations are representative of historical activities associated with drum and material storage.
MW6, MW7OB/ MW7BR, MW8, MW13, MW14, MW15	These locations are representative of groundwater flowing away from the investigation area at the south and east perimeter swales.
MW11	This location is representative of the area of the former transformer pad where PCBs may have been used in the transformer located on the pad adjacent to the Generator Building.
MW12	This location is representative of active salt shed activities.
MW13	This location is representative of activities in the vicinity of the historical generator building.
MW16	This location is upgradient of the investigation area and representative of reference concentrations.

1779 ## 17.6 Groundwater Well Development and Water-Level Monitoring

1780 Wells will be developed before sampling using the methodology outlined in USACE guidance document
1781 EM 1110-1-4000 Monitoring Well Design, Installation, and Documentation at Hazardous Toxic, and
1782 Radioactive Waste Sites (1998). The water will be containerized per the waste management protocol
1783 identified in Section 17.3.

1784 Monitoring well measuring point elevations will be measured by a New York State (NYS) Professional
1785 Surveyor to the nearest 0.010-inch NAVD 88. Water levels in all wells will be measured from the
1786 measurement point prior to each groundwater sampling event using a combination electronic water level
1787 and oil-water interface probe. Water levels will then be converted to water table elevations referenced to
1788 the measuring point elevations surveyed by a NYS-licensed surveyor. A new groundwater contour map
1789 will be developed using survey and water-level data.

1790 The Monitoring Well Development and Water-Level Monitoring SOP is included in **Appendix E**.

17.7 Groundwater Sampling

One groundwater sample will be collected from the 21 wells during each of two groundwater monitoring events. Monitoring well NHFLA-MW16, which is upgradient of the landfill, will serve as the reference well location. The first event is anticipated to occur during September during low water table conditions. The second event is anticipated to occur in December, during higher water table conditions.

A low flow purging/sampling method (using a bladder or peristaltic pump) will be used to collect the samples. Water quality parameters, including temperature, conductivity, turbidity, pH, ORP, and DO, will be measured during monitoring well development and low-flow purge activities. Samples for dissolved metals analyses will be filtered in the field prior to being placed in laboratory-supplied bottles. Detection levels will be confirmed with the laboratory. The Groundwater Sampling SOP is included in **Appendix E**.

Slug tests will be used to evaluate hydraulic conductivity. Five overburden wells will undergo slug testing, with two tests per well, consisting of one rising head test and one falling head test. The locations of slug test will be distributed across the investigation area to be representative of the site as a whole. The slug testing will be performed after the first groundwater monitoring event. The data will be corrected as necessary based on screen and filter pack saturation at the time of the testing.

17.8 Soil Investigation

Available background, surface, and subsurface soil data are limited, and additional data are required to vertically and horizontally delineate environmental impacts from potential source areas. The existing soil data set is inadequate for assessing where impacts to the environment have occurred due to DoD-activities; therefore, the soil sampling proposed in this QAPP is generally discrete sampling for the purposes of site investigation. The discrete soil sampling analytical results will be used to develop DUs for ISM sampling and risk evaluation, if necessary. The field tasks associated with the soil investigation are outlined in the table below.

Table 17.8: Soil Investigation Tasks

Soil Investigation Task	Reference Guidelines
Reference Surface and Subsurface Soil Sampling	Arcadis Hazmat Shipping Handbook 2018 Appendix E SOPs: – Soil Description – Soil Drilling and Sample Collection – Bedrock Core and Description – Sample Chain of Custody – Groundwater and Soil Sampling Equipment Decontamination – Investigation-Derived Waste Handling and Storage
Surface Soil Sampling	
Subsurface Soil Sampling	

If concentrations of DoD-specific chemicals in soil samples exceed the regulatory benchmarks or risk-based screening levels, then additional delineation samples may be collected following the completion of an initial risk evaluation and potential consultation with USACE and stakeholders.

17.9 Reference Soil Sampling

Currently, limited background soil data are available. Therefore, 32 co-located discrete surface and subsurface soil samples (**Appendix A: Figure 7A and Figure 7B**) will be collected from 16 locations and analyzed for metals and PAHs for the purpose of gathering site-specific reference soil data for comparison against sample concentrations in the former Launch Area. Select samples will also be evaluated for grain size using ASTM Method D422 (without hydrometer). The locations were selected from an adjacent area outside of the investigation area that is used by the Town for recreational purposes.

17.10 Surface Soil Sampling

Surface soil samples will be collected from up to 53 different locations. Previous investigations did not collect samples from the vicinity of the transformer pad for PCB analysis; therefore, three surface soil samples will be collected for PCB analysis. To evaluate the potential risks to site users associated with surface soil exposure, 29 surface soil samples (**Appendix A: Figure 7A and Figure 7B**) will be collected and analyzed for TAL metals and hexavalent chromium, TCL VOCs, TCL SVOCs, and hydrazine. In addition, up to five discretionary surface soil samples will be collected to support the SLERA. The SLERA samples will also be evaluated for grain size using ASTM Method D422 (without hydrometer). To evaluate potential background conditions, reference surface soil samples will be collected from 16 locations and analyzed for TAL metals, hexavalent chromium, and PAHs. Surface soil samples will be collected from the top of native material to 1-foot below using either a split spoon sampler (where co-located with a drilling location) or stainless-steel hand auger. Surface soil sample locations were selected based on historic activities onsite, and the rationale for the sampling location is shown in the table below.

Table 17.10: Rationale for Surface Soil Sampling Locations

Surface Soil Sampling Location	Rationale for Sampling Location
SS1 through SS17	These locations will be collected in conjunction with the overburden groundwater well installation. Refer to **Section 17.5** for the rationale.
SS11A, SS11B	These samples are representative of the area of the former transformer pad where PCBs may have been used in the transformer located on the pad adjacent to the Generator Building.
SS18 through SS24, and SS30	These locations are representative of historical activities associated with the silos.
SS25 through SS28	These locations are representative of historical activities associated with the Generator Building and/or the Missile Assembly Testing Building.
SS29	This location is associated with the drainage swale from the landfill area.
SS31 through SS35	These locations will be chosen in the field in areas of viable habitat not covered by historic or proposed sample locations.
SS36 through SS51	These locations are outside the areas representative of historical activities.

17.11 Subsurface Soil Sampling

Subsurface samples collected during installation of overburden wells will be analyzed for TAL metals and hexavalent chromium, TCL VOCs, TCL SVOCs, hydrazine, grain size, TOC, and moisture content. Up to two samples will be collected from each of the 16 overburden wells. A log will be generated for each

location, and for the bedrock portion of wells, rock core will be utilized to determine bedrock lithology, and evaluated for the quantity and magnitude of fractures. Upon retrieval, split spoon samples will be analyzed for the presence of volatile vapors using a PID and visually inspected for indications of impacts. Soil sample selection will be biased to areas of suspected impacts based on field screening. One sample will be collected from 1 to 3 feet below surface soil sampling interval. The second soil sample will be collected from the zone above the water table that exhibits the greatest impacts based on field screening. If no indications of impacts are evident, then an additional sample will be collected from the 2-foot interval just above the water table. After sample collection, the lithology will be logged using the Unified Soil Classification System.

Reference subsurface soil samples will be analyzed for TAL metals, hexavalent chromium, and PAHs.

17.12 Equipment Decontamination

Decontamination is performed on sampling equipment before sample collection to ensure that the sampling equipment that contacts a sample, or monitoring equipment that is brought into contact with environmental media to be sampled, is free from analytes of interest and/or constituents that could interfere with laboratory analysis for analytes of interest. Sampling equipment must be appropriately cleaned before using for sampling or contacting environmental media to be sampled and following completion of the sampling event before shipment or storage. The effectiveness of the decontamination procedure will be verified by collecting and analysing equipment blank samples.

The drilling contractor will provide a decontamination pad. Sampling equipment that may require decontamination include soil and groundwater sampling tools, drilling equipment, water-quality meters, oil-water meters, and other activity-specific sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between each sample collected, and before placing sampling equipment in protective cases, or containers for transport. The decontamination procedure is detailed in the SOP included in **Appendix E**.

17.13 Waste Management

Waste will be managed in accordance with state and federal regulations to ensure completeness, accuracy, and consistency of waste documentation and to protect employees and contractors from environmental and regulatory risk. All waste generated will be inventoried in the field at the point of generation. All waste containers must be U.S. Department of Transportation (DOT) approved, compatible with the waste stream, and appropriately labeled. Drums must be staged on a pallet and covered with poly-sheeting to protect the drums from precipitation. Rock core will be placed in labeled (including orientation) wooden core boxes and stored on-site at the Town facility until the conclusion of the RI.

It is anticipated that generated waste will be non-hazardous, and therefore disposed offsite at permitted non-hazardous disposal facility. Waste characterization analytical parameters will be collected per the disposal facility requirements. A profile will be submitted to the disposal facility for approval. A manifest will be generated for transportation of the drums. All loading and transportation will be performed in accordance with DOT regulations.

17.14 Survey of Assets

The location of all wells and relevant site features will be surveyed by a NYS-licensed surveyor to the nearest vertical 0.01 foot and horizontal 0.1 foot. The survey will be referenced to New York State Plane Coordinate West Zone NAD 83 for the horizontal datum, and NAVD88 for the vertical datum. Assets to be surveyed include wells, sampling locations, buildings, storage areas, and remaining Nike Battery features, such as silos, drainage and surface water features, etc. The elevations of the ground surface, measuring point (i.e., top of PVC riser), and top of protective casing at each monitoring well will be surveyed. A metes-and-bounds survey for the parcel will also be conducted.

17.15 Risk Assessment

The SERES-Arcadis JV will complete a baseline HHRA and SLERA in accordance with the Performance Work Statement and relevant USEPA and USACE guidance. **Appendix C** contains the Human Health and Ecological Risk Assessment Work Plan, which provides detail on the methodology including data evaluation, exposure assessment, toxicity assessment and risk characterization. Risk assessments will be conducted using soil and groundwater data collected during the RI. Historical soil data will be evaluated to determine its usability and may be included, as appropriate, in the quantitative assessment of health risks.

Preliminary human health and ecological CSMs developed for the former Launch Area will be used as the foundation for the baseline HHRA and SLERA. Information regarding site characteristics, site accessibility, land use, site activities, and potential human and ecological receptors will be gathered, and the data gaps outlined in **Section 10.7** will be addressed during the RI to ensure complete risk assessments.

17.15.1 Baseline Human Health Risk Assessment

The purpose of the HHRA will be to characterize health risks associated with soil and groundwater impacts that could prevail, currently and in the future, in the absence of remedial action. The HHRA will follow USEPA Risk Assessment Guidance for Superfund (RAGS) (1989, 2001, 2004, 2009) methodology, which is consistent with USACE guidance EM 200-1-4, Risk Assessment Handbook - Volume I: Human Health Evaluation (1999).

First, the preliminary human health CSM (see **Figure 10.3**) will be reviewed and updated, as needed. Screening levels presented in the UFP QAPP **Worksheet #15** will be verified and a data evaluation will be conducted to determine if further quantitative assessment of potential adverse human health effects is warranted. Background screening will be conducted according to the Tri-Service Position Paper on Background Levels in Risk Assessment (USACE 2011). If it is determined a full HHRA is not warranted, the HHRA will be truncated with results of the screening (*i.e.*, data evaluation and part of the exposure assessment). In the event a full HHRA is warranted, the HHRA will consist of the following five parts.

Data Evaluation: Summarizes the sources of analytical data for each sampled environmental medium; discusses the data quality and usability; and identifies the COPCs that are evaluated quantitatively in the HHRA. COPCs are the DoD-related constituents that may pose a human health risk. COPCs will be identified based on multiple factors including the comparison to screening levels and the comparison to reference concentrations. Assuming the overburden and bedrock groundwater are in hydrogeologic communication, all groundwater data will be initially evaluated as a single aquifer (i.e., data from 16 overburden and five bedrock monitoring wells will be combined) to identify COPCs for the potable use scenario. Groundwater data from only the overburden wells will be used to identify COPCs for the vapor intrusion pathway.

Exposure Assessment: Describes the pathways by which COPCs released to the environment may reach human receptors, and the exposure routes by which contact may result in uptake or absorption of the COPCs. **Figure 10.3** shows the preliminary human health CSM. Reasonable maximum exposure (RME) assumptions will be made based on USEPA guidance, where relevant. An EPC for each COPC in soil and groundwater will be estimated as the 95% upper confidence limit on the mean or the maximum detected concentration, as described in the HHRA Work Plan in **Appendix C**. The approach for estimating the EPC to evaluate the potable use scenario for each groundwater COPC will be consistent with USEPA OSWER Directive 9283.1-42 (USEPA 2014). Maximum detected concentrations (or the lesser of the maximum and 95% UCL where three or more data points are available for a monitoring well where the maximum concentration occurred) will be used as the EPC for each on-site groundwater COPC. Unless the hydrogeological investigation demonstrates the overburden and bedrock groundwater are in communication, separate EPCs will be used to represent overburden and bedrock groundwater. In the event residential wells downgradient of the former Launch Area are identified and groundwater COPCs in those wells are identified, maximum detected concentrations (or the lesser of the maximum and 95% UCL where three or more data points are available for a given well) will be used as the EPC for each COPC at the individual property. EPCs for other media (i.e., outdoor and indoor air) will be derived from numerical relationships between the chemical's concentration in soil or groundwater and the other medium based on chemical-specific properties and site-specific parameters (e.g., wind speed and soil type). The exposure assessment results in estimates of medium-, COPC-, receptor-, and exposure route-specific intake rates or exposure concentrations that are used to evaluate the potential for adverse health effects. If risks in excess of the USEPA acceptable levels are determined for an exposure pathway, the pathway will be re-evaluated using central tendency exposure parameter values, where available, in place of the upper-bound values used in the RME analysis. Central tendency estimates may be developed to represent more realistic or site-specific conditions (e.g., outdoor worker exposure frequency and exposure time could be adjusted to reflect climatic conditions or the current and most likely future land use as a Highway Department storage area).

Toxicity Assessment: Documents the oral, inhalation, and dermal toxicity values, gastrointestinal absorption factors used to estimate the dermal toxicity values, and the "target organs" or critical non-cancer effects of the COPCs. Toxicity values will be obtained according to the USEPA hierarchy of sources (2003).

Risk Characterization: Develops quantitative estimates of the potential for adverse human health effects, in terms of excess lifetime cancer risks and non-cancer hazards. If lead is identified as a COPC, the potential for adverse health effects from exposure to lead will be evaluated using USEPA recommended models (e.g., USEPA's Integrated Exposure Uptake Biokinetic Model or Adult Lead Model).

Uncertainty Evaluation: Identifies site-specific sources of uncertainty and discusses whether the potential for human health risk is more likely to be under- or over-estimated as a result. For example, in the risk assessment, the potential for adverse health effects associated with exposure to an undetected constituent with detection limits higher than risk-based screening levels, indicating total receptor risks may be underestimated, will be discussed qualitatively in the uncertainty section.

17.15.2 Ecological Risk Assessment

The RI will include a comprehensive SLERA, and if necessary, the initial step of a baseline ecological risk assessment (BERA) to evaluate potential risk to ecological receptors. The ecological risk assessment will follow appropriate guidelines from USEPA (e.g., Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments [1997]), USACE guidance EM 200-1-4 (Risk Assessment Handbook Volume II: Environmental Evaluation [2010]), and the Tri-Services guidance TSERAWG TG-090801 (A Guide to Screening Level Ecological Risk Assessment [2008]). The SLERA

will include Steps 1 and 2 of the USEPA's overall eight-step process as an initial evaluation of potential ecological risk at a site. If warranted, refinement of the SLERA will occur in Step 3a, the initial BERA step.

Step 1 will include a screening-level problem formulation, which provides information used to establish the overall goals, breadth, and focus of an ecological risk assessment, and a screening-level effects evaluation, which identifies current and appropriate media screening values below which there is negligible risk. Step 2 will involve screening-level exposure estimates and risk calculations for appropriate receptors and endpoints. The SLERA process will culminate in clearly defined scientific management decision points (SMDPs). The SMDPs represent critical steps in the process where risk management decision-making occurs. Generally, the following types of decisions are considered at the SMDPs: (1) whether the available information is adequate to conclude that ecological risks (if any) are negligible and, therefore, there is no need for further action on the basis of ecological risk; (2) whether the available information is not adequate to make a decision at this point, and the ecological risk assessment process should continue; and (3) whether the available information indicates a potential for adverse ecological effects, and a more thorough assessment or remediation is warranted.

If available information is not adequate to conclude that ecological risk is negligible, further refinement may be warranted in BERA Step 3a to incorporate exposure estimates using more realistic assumptions and additional methodologies to better estimate actual potential exposure. The refinement in Step 3a involves refining, refining risk calculations, food chain modeling of bioaccumulative MC, and refining the risk characterization. Step 3a may include exposure estimates based on both conservative exposure parameters (maximum food intake rates and minimum body weights) as well as refined exposure parameters (average food intact rates and body weights). The results of Step 3a will serve to identify if additional evaluation of ecological risk via a baseline ecological risk assessment (Steps 3b through 7 of the USEPA's eight-step process) is necessary. Major sources of uncertainty in the ecological risk assessment process will also be documented.

17.16 Final Report

After completing RI field activities, an RI Report will be developed compliant with CERCLA requirements. A draft version and final version of the RI Report will be submitted, which will:

- Evaluate the nature and extent of environmental impacts in soil and groundwater, including within fractured bedrock;
- Compare former Launch Area data against appropriate screening levels (i.e., RSLs) and reference concentrations;
- Discuss a conceptual site model, including evaluation of former Launch Area constituent(s) of concern source(s), pathways and receptors;
- Present data needed for a remedial design, if required; and
- Include a baseline HHRA and SLERA, and if necessary, the initial step of a BERA.

All environmental, geophysical, geological, hydrogeological, chemical, and spatial data collected during the course of the RI will be transferred to the FUDSChem database.

QAPP Worksheet #18: Sampling Locations and Methods/Standard Operating Procedure Requirements

(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

Sample ID	Matrix	Depth (feet bgs)	Sample Type[1]	Analytical Group	Number of Samples (plus field duplicates)	Rationale	SOP Ref. Number
LAND INVESTIGATION							
Refer to WS # 26/27 for the Sample Identification System	Surface Soil	12 inches below the top of native material	Discrete Biased	PCB	3 (1 duplicate)	There is currently no PCB data from the area around the former transformer pad.	WS #21
Refer to WS # 26/27 for the Sample Identification System	Surface Soil	12 inches below the top of native material	Discrete Biased	TAL metals, hexavalent chromium, TCL VOCs, TCL SVOCs, hydrazines, TOC, pH	29 (3 duplicates)	Available surface soil data is limited. Additional data is required to vertically and horizontally delineate environmental impacts from potential source areas. Some historical data has detection limits above regulatory limits or applicable screening levels.	WS #21

Sample ID	Matrix	Depth (feet bgs)	Sample Type[1]	Analytical Group	Number of Samples (plus field duplicates)	Rationale	SOP Ref. Number
Refer to WS # 26/27 for the Sample Identification System	Subsurface Soil	2-foot interval below the surface sample. If sufficient material is available above competent bedrock, another sample may be taken at the highest PID reading, or, if an elevated PID reading is not present, taken at the 2-foot interval above water table	Discrete Biased	TAL metals, hexavalent chromium, TCL VOCs, TCL SVOCs, hydrazines, grain size, TOC, moisture content	32 (4 duplicates)	Available subsurface soil data is limited. Additional data is required to vertically and horizontally delineate environmental impacts from potential source areas. Some historical data has detection limits above regulatory limits or applicable screening levels.	WS#21
GROUNDWATER INVESTIGATION							
Refer to WS # 26/27 for the Sample Identification System	Groundwater	NA	Discrete Biased	TAL metals (total and dissolved), hexavalent chromium, TCL VOCs, TCL SVOCs, hydrazines	21 (3 duplicates) for each of 2 events Total 42 (6 duplicates)	There is incomplete horizontal & vertical delineation of groundwater flow and impacts from potential source areas. Existing data is outdated. Some historical data has detection limits above regulatory limits or applicable screening levels.	WS #21
REFERENCE SAMPLES							
Refer to WS # 26/27 for the Sample Identification System	Surface Soil	12 inches below the top of native material	Discrete Sampling	TAL metals, hexavalent chromium, PAHs	16 (2 duplicate)	Available background surface soil data are limited, and additional data is required to vertically and horizontally delineate environmental impacts from potential source areas.	WS #21

Sample ID	Matrix	Depth (feet bgs)	Sample Type[1]	Analytical Group	Number of Samples (plus field duplicates)	Rationale	SOP Ref. Number
Refer to WS # 26/27 for the Sample Identification System	Subsurface Soil	2-foot interval below the surface sample	Discrete Sampling	TAL metals, hexavalent chromium, PAHs	16 (2 duplicate)	Available background subsurface soil data are limited, and additional data is required to vertically and horizontally delineate environmental impacts from potential source areas.	WS #21

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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 (Name, sample receipt address, Point of Contact, email, and phone numbers)

Katahdin Analytical Services
600 Technology Way
Scarborough, Maine 04074
Contact: Heather Manz
Phone: 207.536.1904
Email: hmanz@katahdinlab.com

- Level IV data packages to be delivered within 21 calendar days
- Sample Delivery Method: Federal Express
- DoD ELAP and New York State Laboratory Accreditation Program Certifications are included in **Appendix G**.

Parameter	Analytical and Preparation Method/ SOP Reference	Method	Bottle Type	Preservation	Holding Time [1]
Soil/Sediment					
VOCs	SW-846 5035, 8260C/ CA-202, CA-214	8260C	3 x EnCores; 1 x 2 oz jar for percent moisture	DI water; Methanol Cool to ≤ 6 °C	48 hours to freezing; 14 days to analysis
SVOCs	SW-846 3540C or 3550C, 8270D and 8270D-SIM/ CA-213, CA-226, CA-512, CA-526	8270D/8270 D-SIM	1 x 4 oz wide mouth glass jar	Cool to ≤ 6 °C	14 days to extraction; 40 days to analysis

Parameter	Analytical and Preparation Method/ SOP Reference	Method	Bottle Type	Preservation	Holding Time [1]
PCBs	SW846 3540C, 3545A or 3550C, 8082A/ CA-329, CA-500, CA-524, CA-537	8082A	1 x 4 oz wide mouth glass jar	Cool to ≤ 6 °C	1 year to extraction; 40 days to analysis
Hydrazines	SW846 8315A/ WI9448	8315A	1 x 4 oz amber glass jar	Cool to ≤ 6 °C	14 days to extraction; 3 days from extraction to analysis
Metals	SW846 3050B, 6010C, 7471A/ CA-605, CA-608, CA-611	6010C, 7471A	1 x 2-oz wide-mouth jar	None Mercury - Cool to ≤ 6 °C	6 months to analysis, Mercury: 28 days to analysis
Total Solids	SM 2540B/ CA-718	SM 2540B	1 x 2-oz wide-mouth jar	Cool to ≤ 6 °C	28 days to analysis
Total Organic Carbon	Lloyd Kahn/ CA-741	Lloyd Kahn	1 x 2-oz wide-mouth jar	Cool to ≤ 6 ° C	14 days to analysis
Grain Size Distribution	ASTM D422 / CA-551	ASTM D422	8-oz wide-mouth jar	Cool to ≤ 6 ° C	None
Hexavalent Chromium	SW846 7196A / CA-625	7196A	1 x 2-oz wide-mouth jar	Cool to ≤ 6 ° C	28 days to analysis
Groundwater					
VOCs	SW846 5030B, 8260C/ CA-202	8260C	3 x 40-ml VOA vials	HCl to pH <2 Cool to ≤ 6 °C	14 days to analysis
SVOCs	SW846 3510C, 3520C, 8270D and 8270D-SIM/ CA-213, CA-226, CA-502	8270D/8270 D-SIM	2 x 1 L amber glass jars	Cool to ≤ 6 °C	7 days to extraction; 40 days to analysis
Metals	SW846 3010A, 6020A, 7470A/ CA-604, CA-627, CA-615	6020A, 7470A	One 250-ml polyethylene bottle	None - Cool to ≤ 6 °C	6 months to analysis Mercury: 28 days to analysis
Hardness	SM 2340 B (calc)/ CA-627	SM2340B	One 250-ml polyethylene bottle	Nitric Acid to pH<2.	6 months to analysis
Hexavalent Chromium	SW846 7196A / CA-772	7196A	One 250-ml polyethylene bottle	Cool to ≤ 6 ° C	24 hours to analysis

2022 **Notes:** °C = degree Celsius L = liter ml = milliliter oz = ounce

QAPP Worksheet #20: Field QC Sample Quantities and Control Frequencies

(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

Matrix/ Analysis	Estimated Environ. Sample Quantity[1]	Field QC Sample						Laboratory QC Sample				Total
		Field Duplicate		Trip Blank		Equipment Blank		Matrix Spike		Matrix Spike Duplicate		
		Freq	Number	Freq	Number	Freq	Number	Freq [2]	Number	Freq [2]	Number	
Soil												
VOCs	68	1/10	7	1/cooler	20	1/day	10	1/20	4	1/20	4	113
SVOCs	100	1/10	10	NA	NA	1/day	10	1/20	5	1/20	5	130
PCBs	3	1/10	1	NA	NA	1/day	1	1/20	1	1/20	1	7
Hydrazines	68	1/10	7	NA	NA	1/day	10	1/20	4	1/20	4	93
Metals	100	1/10	10	NA	NA	1/day	10	1/20	5	1/20	5	130
Total Organic Carbon	68	1/10	7	NA	NA	NA	NA	1/20	4	1/20	4	83
Grain Size Distribution	68	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	68
pH	34	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	34
Hexavalent Chromium	100	1/10	10	NA	NA	1/day	10	1/20	5	1/20	5	130
Groundwater												
VOCs	44	1/10	5	1/cooler	10	1/day	10	1/20	3	1/20	3	75
SVOCs	44	1/10	5	NA	NA	1/day	10	1/20	3	1/20	3	65
Metals	44	1/10	5	NA	NA	1/day	10	1/20	3	1/20	3	65
Hardness	44	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	44
Hexavalent Chromium	44	1/10	5	NA	NA	1/day	10	1/20	3	1/20 (matrix duplicate)	3	65

Notes:

1. Sample quantities are approximate.
2. Frequency for MS/MSD samples is 1 per 20 field samples, not including field blanks and field duplicates.

QC = quality control
NA = not applicable

QAPP Worksheet #21: Field Standard Operating Procedure References

(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

SOP# or Reference	Title, Revision, Date and URL (if available)	Originating Organization	SOP Option of Equipment Type (if SOP provides different options)	Modified for Field Work? (Yes/No)	Comments
RI-SOP-01	Monitoring Well Installation	SERES-Arcadis JV	NA	No	Field SOPs are included in **Appendix E** of this QAPP
RI-SOP-02	Monitoring Well Development	SERES-Arcadis JV	NA	No	
RI-SOP-03	Manual Water-Level Monitoring	SERES-Arcadis JV	NA	No	
RI-SOP-04	Low-Flow Groundwater Purging and Sampling Procedures	SERES-Arcadis JV	NA	No	
RI-SOP-05	In-Situ and Ex-Situ Water Quality Parameters	SERES-Arcadis JV	NA	No	
RI-SOP-06	Slug Testing	SERES-Arcadis JV	NA	No	
RI-SOP-07	Soil Description	SERES-Arcadis JV	NA	No	
RI-SOP-08	Bedrock Core and Description	SERES-Arcadis JV	NA	No	
RI-SOP-09	Soil Drilling and Sample Collection	SERES-Arcadis JV	NA	No	
RI-SOP-10	Sample Chain of Custody	SERES-Arcadis JV	NA	No	
RI-SOP-11	Groundwater and Soil Sampling Equipment Decontamination	SERES-Arcadis JV	NA	No	
RI-SOP-12	Investigation-Derived Waste Handling and Storage	SERES-Arcadis JV	NA	No	
RI-SOP-13	Field Activities Documentation	SERES-Arcadis JV	NA	No	
RI-SOP-14	Utility Clearance	SERES-Arcadis JV	NA	No	

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 will be maintained, inspected, and tested as presented in the table below.

Requirement	Limited Applicability (Specific to Collection Method/Use)	Performance Standard	Frequency	Consequence of Failure
Global positioning system (GPS)	Soil and Sediment Samples	Calibration within the manufacturers specified range.	Daily when instrument is being used.	Redo until calibration results are with range.
Electric water level indicator	Groundwater Samples	Calibration within the manufacturers specified range.	Daily when instrument is being used.	Redo until calibration results are with range.
Turbidity meter	Groundwater Samples	• Calibration within the manufacturers specified range. • Acceptance Criteria (± 5% of standard)	Daily when instrument is being used.	Redo until calibration results are with range.

Requirement	Limited Applicability (Specific to Collection Method/Use)	Performance Standard	Frequency	Consequence of Failure
Multiparameter water quality meter	Groundwater Samples	• Calibration within the manufacturers specified range. • Acceptance Criteria o Dissolved Oxygen ▪ ± 0.5 milligrams per liter (mg/L) of sat. value ▪ < 0.5 mg/L for the 0 mg/L solution, but not a negative value o Specific Conductance (±5% of standard or ± 10 µS/cm [whichever is greater]) o pH (± 0.3 pH unit with pH 7 buffer) o Oxidation Reduction Potential (± 10 mV) • Drift Check	• Daily when instrument is being used. • End of the day.	Redo until calibration results are with range.
Electric submersible pump	Groundwater Samples	Working as designed.	Daily when instrument is being used.	Redo until calibration results are within range and if needed, replace pump

Requirement	Limited Applicability (Specific to Collection Method/Use)	Performance Standard	Frequency	Consequence of Failure
Bladder Pump	Groundwater Samples	Working as designed.	Daily when instrument is being used.	Redo until calibration results are within range and if needed, replace pump.
Peristaltic Pump	Groundwater Samples	Working as designed.	Daily when instrument is being used.	Redo until calibration results are within range and if needed, replace pump.
Photoionization Detector and/or other air monitoring equipment	Drilling, Well Installation, Groundwater Sampling	• Calibration using isobutylene within the manufacturers specified range using a 10.6 eV lamp. • Acceptance Criteria (zero fresh air recheck following calibration [100-2,000 parts per million]) • Drift Check	• Daily when instrument is being used. • End of the day.	Redo until calibration results are with range.

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QAPP Worksheet #23: Analytical Standard Operating Procedure References

(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

All analytical SOPs pertinent to environmental investigations are located for reference in **Appendix G**.

SOP #	Title, Revision Date and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Yes/No)
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, 10/18, Revision 19.	Definitive	Soil/Sediment/ Groundwater/VOCs	Gas Chromatography/ Mass Spectrometry (GC/MS)	Katahdin Analytical Services, LLC	N
CA-214	Closed-System Purge-And-Trap And Extraction For Volatile Organics In Soil And Waste Samples Using SW846 Method 5035, 03/18, Revision 7.	Definitive	Soil/Sediment/VOCs	VOC Extraction	Katahdin Analytical Services, LLC	N
CA-226	Analysis of SVOAs by Capillary Column GC/MS: SW-846 Method 8270D, 06/17, Revision 10. (Reviewed 01/19)	Definitive	Soil/Sediment/ Groundwater/SVOCs	GC/MS	Katahdin Analytical Services, LLC	N
CA-213	Analysis of SVOA by SW846 Method 8270 – Modified for Selected Ion Monitoring, 01/19, Revision 15	Definitive	Soil/Sediment/ Groundwater/SVOCs	GC/MS	Katahdin Analytical Services, LLC	N
CA-512	Preparation of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis, 04/19, Revision 14.	Definitive	Soil/Sediment/SVOC Sample Preparation	NA	Katahdin Analytical Services, LLC	N

SOP #	Title, Revision Date and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Yes/No)
CA-502	Preparation of Aqueous Samples For Extractable Semivolatile Analysis, 03/19, Revision 12.	Definitive	Groundwater/SVOC Sample Preparation	NA	Katahdin Analytical Services, LLC	N
CA-329	Analysis of PCBs As Total Aroclors By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082, 01/19, Revision 19.	Definitive	Soil/Sediment/ Groundwater/PCBs	GC/Electron Capture Detector (ECD)	Katahdin Analytical Services, LLC	N
CA-500	Preparation of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Pesticides/PCBs Analysis, 03/19, Revision 11.	Definitive	Soil/Sediment/PCB Sample Preparation	NA	Katahdin Analytical Services, LLC	N
CA-524	Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Pesticide/PCB Analysis, 03/19, Revision 11	Definitive	Soil/Sediment/PCB Sample Preparation	NA	Katahdin Analytical Services, LLC	N
CA-526	Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis, 04/19, Revision 12	Definitive	Soil/Sediment/SVOC Sample Preparation	NA	Katahdin Analytical Services, LLC	N
WI9448	Determination of Hydrazine, Monomethylhydrazine and 1,1-Dimethylhydrazine in Aqueous Samples by LC/MS/MS Using SW-846 8315A Modified, Rev 9, Effective 03/19/2019	Definitive	Soil/Sediment/ Hydrazines	Liquid Chromatography (LC)/MS/MS	Eurofins Lancaster Environmental (ELLE)	N
CA-551	Grain Size Analysis, 04/19, Revision 2.	Definitive	Solid/ Grain Size	Sieves/Hydrometer	Katahdin Analytical Services, LLC	N

SOP #	Title, Revision Date and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Yes/No)
CA-604	Acid Digestion of Aqueous Samples by USEPA Method 3010 for ICP Analysis of Total or Dissolved Metals, 01/19, Revision -.8	Definitive	Groundwater / Target Analyte List (TAL) Metals	Not applicable (digestion)	Katahdin Analytical Services, LLC	N
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA, 01/19, Revision 8.	Definitive	Soil/Sediment / TAL Metals	Not applicable (digestion)	Katahdin Analytical Services, LLC	N
CA-608	Trace Metals Analysis By ICP-AES Using EPA Method 6010, 01/19, Revision 19.	Definitive	Soil/Sediment / TAL Metals except mercury	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	Katahdin Analytical Services, LLC	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020, 01/19, Revision 13.	Definitive	Groundwater/ TAL Metals except mercury	ICP- MS	Katahdin Analytical Services, LLC	N
CA-611	Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471, 01/19, Revision 12.	Definitive	Soil/Sediment / Mercury	Cold Vapor Atomic Absorption (CVAA)	Katahdin Analytical Services, LLC	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470, 01/19, Revision 11.	Definitive	Water / Mercury	CVAA	Katahdin Analytical Services, LLC	N
CA-625	Alkaline Digestion and Subsequent Determination of Hexavalent Chromium in Solid Samples Using EPA SW846 Methods 3060 and 7196, 03/19, Revision 7.	Definitive	Soil/Sediment / Hexavalent Chromium	Manual Spectrophotometer	Katahdin Analytical Services, LLC	N
CA-717	Total Solids/Total Volatile Solids Determination in Solid Matrices, 02/19, Revision 11.	Definitive	Soil/Sediment/ Total Solids	Balance	Katahdin Analytical Services, LLC	N

SOP #	Title, Revision Date and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Yes/No)
CA-741	Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method, 01/19, Revision 8.	Definitive	Water / TOC	TOC Analyzer	Katahdin Analytical Services, LLC	N
CA-772	Colorimetric Analysis of Hexavalent Chromium Using the Automated Konelab Multiwavelength Photometric Analyzer, 01/20, Revision 5.	Definitive	Water / Hexavalent Chromium	Automated Spectrophotometer	Katahdin Analytical Services, LLC	N

Notes:

1. The ELLE SOP for hydrazine analysis is considered business confidential. A copy of the cover pages is included in Appendix G. Redacted copies will be provided upon request.

QAPP Worksheet #24: Analytical Instrument Calibration

(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

A list of analytical instrumentation that are anticipated for use is provided in the following table.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS for VOCs, SVOCs, and SVOCs by SIM	Initial Calibration (ICAL) A minimum 5-point initial calibration is required for all compounds	At instrument setup and after initial calibration verification (ICV) or continuing calibration verification (CCV) failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤15% (20% for SVOCs by SIM); Option 2: linear least squares regression for each analyte: r2 ≥0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥0.99.	Correct problem, then repeat ICAL.	Laboratory Analyst, Department Manager	CA-202 CA-213 CA-226
	Establish Retention Time (RT) Window Position	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.		
	Evaluation of Relative Retention Times (RRTs)	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 compounds and surrogates within ±20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS for VOCs and SVOCs (continued)	CCV	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 CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Laboratory Analyst, Department Manager	CA-202 CA-226
	BFB Tune (VOCSs) DFTPP Tune (SVOCs)	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.		
GC/ECD-for PCBs	ICAL - A minimum 5-point calibration is run except for mid-point calibration of Aroclors 1221, 1232 and 1248; if targets are detected, then 6-point calibration is performed.	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	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 >/= 0.995; Option 3: non-linear least squares regression (quadratic) for each (quadratic) for each analyte: r2 >/= 0.99.	Correct problem then repeat ICAL.	Laboratory Analyst, Department Manager	CA-329
	Retention Time 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.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/ECD-for PCBs	Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA.	Laboratory Analyst, Department Manager	CA-329
	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.		
	CCV	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for multi-component analytes (Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. 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 re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		
LC/MS/MS for Hydrazines	Tuning of LC/MS/MS	Tune before initial calibration	Refer to the instrument manufacturers' instructions	Retune and recalibrate	Laboratory Analyst, Department Manager	WI9448
	ICAL: Initial calibration with a minimum 6 points spanning at least a 50-fold concentration	Daily	Use a linear regression weighted 1/x (where x equal to concentration). $r^2 \geq 0.99$ and %RPD of calculated concentration against nominal concentration ±30%	Recalibrate, perform instrument maintenance. If calibration does not meet method criteria, recalibrate.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
LC/MS/MS for Hydrazines (continued)	ICV	After each ICAL	Target compounds ≤30% drift (%D)	Reanalyze the ICV. If ICV fails again do system maintenance and recalibrate.	Laboratory Analyst, Department Manager	WI9448
	CCV	Every 10 samples and at end of analytical run	Target compounds ≤30%D	Reanalyze the ending CCV. If ending CCV fails again do system maintenance, recalibrate, and reanalyze affected samples.		
ICP-AES for Metals except Mercury	ICAL: 1 calibration high standard and 1 calibration blank.	Daily ICAL prior to sample analysis.	One point calibration plus a blank per manufacturer's guidelines.	If ICV or low level ICV fails, correct problem, then repeat ICAL.	Laboratory Analyst, Department Manager	CA-608
	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.		
	CCV	After every 10 field samples, and at the end of the 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 either fails, take CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP-AES for Metals except Mercury (continued)	Low-Level Calibration Check Standard (Low-Level ICV)	Daily	All reported analytes within ±20% of true value.	Correct problem and repeat ICAL.	Laboratory Analyst, Department Manager	CA-608
	Initial and Continuing Calibration Blank	Before beginning a sample run, after every 10 field samples, and at the end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.		
	Interference Check Sample (ICS)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ±20% of true value.	Correct the problem, then re-prepare checks and reanalyze all affected samples.		
ICP-MS for Metals except Mercury	Tune	Daily prior to calibration.	Mass calibration must be within 0.1 atomic mass unit (amu) from the true value. Resolution must be <0.9 amu full width at 10% peak height. Four injections %RSD must be <5%.	Perform necessary equipment maintenance.	Laboratory Analyst, Department Manager	CA-627
	ICAL - 1 point calibration plus blank	Daily ICAL prior to sample analysis.	One point calibration plus a blank per manufacturer's guidelines.	If ICV or low level ICV fails, 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.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP-MS for Metals except Mercury (continued)	CCV	After every 10 field samples, and at the end of the 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 either fails, take CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Laboratory Analyst, Department Manager	CA-627
	Low-Level Calibration Check Standard (Low-Level ICV)	Daily	All reported analytes within ±20% of true value.	Correct problem and repeat ICAL.		
	Initial and Continuing Calibration Blank	Before beginning a sample run, after every 10 field samples, and at the end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.		
	Interference Check Sample (ICS)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ±20% of true value.	Correct the problem, then re-prepare checks and reanalyze all affected samples.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Cold Vapor Atomic Absorption (CVAA) for Mercury	ICAL – minimum 5 calibration standards and a calibration blank.	Daily ICAL prior to sample analysis.	Least squares regression, $r^2 \geq 0.995$.	Correct problem, then repeat ICAL.	Laboratory Analyst, Department Manager	CA-611, CA-615
	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Mercury within ±10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.		
	CCV	After every 10 field samples, and at the end of the analysis sequence.	Mercury 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 either fails, take CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		
	Initial and Continuing Calibration Blank	Before beginning a sample run, after every 10 field samples, and at the end of the analysis sequence.	No analytes detected > LOD	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
TOC by Lloyd Kahn/9060	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	Initially, when the daily CCV does not pass, but, no longer than every 3 months.	Correlation coefficient – $r \geq 0.995$	Inspect system, correct problem, rerun calibration, and affected samples.	Laboratory Analyst, Department Manager	CA-741
	ICV	Once after each ICAL, prior to beginning a sample run.	Lloyd Kahn: %R must within 80%-120%	(1) If the ICV fails high, report samples that are <PQL. (2) Re-digest, recalibrate and/or reanalyze other samples.		
	CCV	Every 10 samples and at the end of the run	Lloyd Kahn: %R 80%-120%	If the CCV fails high, report samples that are <PQL. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.		
Analytical Balance	Balance Verification	Every day before use.	Within criteria specified in KAS SOP CA-102.	Investigate problem. Do not use balance until verification has passed.	Laboratory Analyst, Department Manager	CA-717
Grain Size	Sieve – Visual inspection – ASTM D422	Every use	No clogging or tears in mesh	Remove from service	Laboratory Analyst, Department Manager	CA-551
Spectrophotometer for Hexavalent Chromium / 7196A	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	Prior to sample analysis	Correlation coefficient – $r \geq 0.995$	Investigate source of problem and recalibrate.	Laboratory Analyst, Department Manager	CA-625

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Spectrophotometer for Hexavalent Chromium / 7196A (Continued)	ICV	Once after each ICAL, prior to beginning a sample run.	%R 90-110%	(1) If the ICV fails high, report samples that are <PQL. (2) Re-digest, recalibrate and/or reanalyze other samples.	Laboratory Analyst, Department Manager	CA-625
	CCV	Every 10 samples	%R 90%-110%	If the CCV fails high, report samples that are <PQL. Re-digest, recalibrate and/or reanalyze samples back to last acceptable CCV recovery.		
Konelab for Hexavalent Chromium / 7196A	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	Prior to sample analysis	Correlation coefficient – $r \geq 0.995$	Investigate source of problem and recalibrate.	Laboratory Analyst, Department Manager	CA-772
	ICV	Once after each ICAL, prior to beginning a sample run.	%R 90-110%	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Konelab for Hexavalent Chromium / 7196A (Continued)	CCV	Every 10 samples and at the end of the run	%R 90%-110%	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 CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Laboratory Analyst, Department Manager	CA-772

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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)

The laboratory analytical instrumentation and equipment shall be maintained, tested, and inspected following the procedures and frequency specified by the DoD Environmental Laboratory Accreditation Program-evaluated laboratory Quality Manual, analytical methods used, and in accordance with Section 5.5, Module 2, "Calibration Requirements," of the *DoD QSM 5.3* (DoD, 2019).

Instrument and equipment maintenance logs must be kept documenting analytical instrumentation and equipment maintenance, testing, and inspection activities. Instrument maintenance procedures documented in laboratory QA Manual and SOPs will be followed.

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS	See Laboratory QA Manual and SOPs	VOCs SVOCs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-202 CA-213 CA-226
GC/ECD	See Laboratory QA Manual and SOPs	PCBs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-329
LC/MS/MS	See Laboratory QA Manual and SOPs	Hydrazines	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	WI9448
ICP-AES	See Laboratory QA Manual and SOPs	TAL Metals except Mercury	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-608
ICP-MS	See Laboratory QA Manual and SOPs	TAL Metals except Mercury	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-627

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
CVAA	See Laboratory QA Manual and SOPs	Mercury	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-611, CA-615
TOC Combustion Analyzer	See Laboratory QA Manual and SOPs	TOC	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-741
Balance	See Laboratory QA Manual and SOPs	Total Solids	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-717
Spectrophotometer	See Laboratory QA Manual and SOPs	Hexavalent Chromium	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-625
Konelab	See Laboratory QA Manual and SOPs	Hexavalent Chromium	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	See Laboratory QA Manual and SOPs	Laboratory Analyst, Department Manager	CA-772

Notes:

[1]SOP reference numbers correspond to analytical SOPs in QAPP **Worksheet #23**.
CVAA = cold vapor atomic absorption
GC = gas chromatography
ECD = electron capture detector
LC = liquid chromatography
ICP-AES = inductively coupled plasma atomic emission spectrometry
ICP-MS = inductively coupled plasma mass spectrometry
MS = mass spectrometry
QA = Quality Assurance
SOP = Standard Operating Procedure
TOC = total organic carbon

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: SERES-ARCADIS JV
Laboratory: Katahdin
Method of sample delivery (shipper/carrier): Federal Express
Number of days from reporting until sample disposal: At least 60 days

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling	Jeff Brayer *SERES-Arcadis JV*	See "Sample Identification System" below.
Chain-of-custody form completion	Jeff Brayer *SERES-Arcadis JV*	See RI-SOP-10. Also see "Sample Handling and Custody" below.
Packaging	Jeff Brayer *SERES-Arcadis JV*	See "Field Procedures" below.
Shipping coordination	Jeff Brayer *SERES-Arcadis JV*	See "Transfer of Custody and Shipment" below.
Sample receipt, inspection, and log-in	*Katahdin*	See "Laboratory Custody Procedures" below.
Sample custody and storage	*Katahdin*	See "Laboratory Custody Procedures" and "Final Evidence Files" below.
Sample disposal	*Katahdin*	See "Sample Holding Times" below and QAPP **Worksheet #19/30**.

Sample Identification System

A sample identification system will be established in accordance with the FUDSChem Naming Conventions. A sample numbering system will be used to identify each sample; the sample numbers will be sequentially assigned to ensure there is no duplicate of sample numbers. This system will provide a tracking procedure to allow retrieval of information about a particular sample and will assure each sample is uniquely numbered. The sample identification will consist of the components described below:

Location ID - The Location ID consists of the following elements:

		Location Type						
Property	-	OR	Location Sequence	Well Type (if needed)	-	Depth	-	Timeframe
		QC Type						

Property (five uppercase alpha and numeric characters used to represent the specific FUDS property):
NHFLA (refers Nike Hamburg Former Launch Arca; use for all samples)

Location Code for non-QC samples (two uppercase alpha characters designating the type of location) Location codes are not used for QC samples.

- BH = For locations where a reference sample was collected, the abbreviation BH is used to indicate that all samples were collected from a boring hole.
- MW= For locations where a monitoring well was installed, the abbreviation MW is used for both soil and groundwater to indicate that the soil was collected from the core drilled for the monitoring well installation, and the groundwater was obtained from the monitoring well.
- SL= For locations where neither a monitoring well was installed nor a boring advanced, the abbreviation SL is used to indicate surface soil locations.

QC Codes for QC samples only:

- DUP = field duplicate
- MS = matrix spike
- MSD = matrix spike duplicate

Location Sequence: A sequential number corresponding with proposed location (e.g., 1-100)

Well Type (as needed to distinguish overburden from bedrock)
For monitoring well locations where both an overburden and bedrock well have been installed approximately ten feet from each other, the following two alpha characters will be used to distinguish the screen placement relative to the geology of the location. Monitoring wells that do not have an OB or a BR designation are automatically overburden wells.

- OB= Overburden
- BR= bedrock

Depth Interval The depth interval from which the sample was collected will be represented by an alpha character. The field notes will indicate the actual depth interval to which each alpha character corresponds.

- A = top depth interval
- B= second depth interval
- C= third depth interval

Time Frame Indicator (Date Sampled): a timeframe indicator of the first three alpha characters followed by the year will be used (MMMYYYY)

Sample Handling and Custody

Sample custody procedures ensure the timely, correct, and complete analysis of each sample for all parameters requested. A sample is considered to be in someone's custody if it:

- Is in his/her possession
- Is in his/her view, after being in his/her possession
- Is in his/her possession and has been placed in a secure location
- Is in a designated secure area

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures provide for specific identification of samples associated with an exact location, the recording of pertinent information associated with the sample, including time of sample collection and any preservation techniques, and a chain-of-custody record that serves as physical evidence of sample custody.

The chain-of-custody documentation system provides the means to individually identify, track, and monitor each sample from the time of collection through final data reporting. Chain-of-custody procedures document pertinent sampling data and all transfers of custody until the samples reach the analytical laboratory. All chain-of-custody forms must be filled out and signed in ink. The following information is typically recorded on manual chain-of-custody forms:

- Project name and/or project number
- Signature of SERES-Arcadis JV field sampler collecting the samples
- Date and time of sample collection
- Discrete or incremental sampling sample designation
- Sample matrix
- Analyses required
- Preservation technique
- Signatures and dates for transfer of custody
- Air express/shipper's bill of lading identification number

The chain-of-custody form serves as an official communication to the laboratory detailing the particular analyses required for each sample. The chain-of-custody record will accompany the samples from the time of sampling through all transfers of custody. It will be kept on file at the laboratory where samples are analyzed and archived. Three copies of the chain-of-custody form are created: one copy is retained by the SERES-Arcadis JV field sampler on site, one copy is forwarded electronically to the SERES-Arcadis JV PM, and one copy is sent to the laboratory with the samples. The on-site SERES-Arcadis JV staff completes a chain-of-custody record to accompany each shipment from the field to the laboratory. The completed chain-of-custody is put in a zip-lock bag and taped to the inside cover of the sample shipping container. If there is more than one container in a shipment, copies of the chain-of-custody form will be placed in each container. The container is then sealed with custody seals and custody is transferred to the laboratory. Commercial carriers are not required to sign off on the chain-of-custody form as long as the forms are sealed inside the sample cooler and the custody seals remain intact.

Samples will be packaged for shipment as outlined below:

- Securely affix the sample label to the container with clear packing tape.
- Check the cap on the sample container to confirm that it is properly sealed.
- Wrap the sample container cap with clear packing tape or place the sample container in a plastic bag to prevent the label from becoming loose or separated from the sample container.

- Complete the chain-of-custody form with the required sampling information and confirm that the recorded information matches the sample labels. Note: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the chain-of-custody prior to this transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal. Place loosely in the cooler beneath, around, and/or on top of the sample containers.
- Fill the remaining space in the cooler with cushioning material.
- Place chain-of-custody forms in a plastic bag and seal. Tape the plastic bag with the forms to the inside of the cooler lid.
- Close the lid of the cooler, lock if applicable, and secure with duct tape.
- Wrap packing or strapping tape around both ends of the cooler at least twice.
- Mark the cooler on the outside with the shipping address and return address, affix "Fragile" labels and draw (or affix) arrows indicating "This Side Up." Cover the labels with clear plastic tape to prevent them from becoming loose or separated from the cooler.
- Place a signed custody seal (or two) over the sample cooler lid so tampering would be evident if the cooler was opened prematurely.

Field Procedures

The SERES-Arcadis JV field sampler is personally responsible for the care and custody of samples until they are properly dispatched. As few people as possible should handle the samples.

The SERES-Arcadis JV field sampler is responsible for entering the proper information in the field logbook, including all pertinent information such as sample identification number, date and time of sample collection, type of analysis, description of sample location, and relevant observations. The information entered into the field logbook will be used to generate a chain-of-custody. Field logbooks will provide the means of recording the data collecting activities that are performed. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a situation without reliance on memory. Entries will be made in ink, with no erasures. If an incorrect entry is made, the information will be crossed out with one strike mark and initialed by the person making the entry.

All sample containers will be labeled with the project identification, sample number, matrix, analysis required, preservation used, and the sampler's initials. Sample labels will be completed using waterproof ink. The completed sample labels will be affixed to each sample bottle and covered with clear tape.

The SERES-Arcadis JV field sampler will review all sampling activities to determine whether proper custody procedures were followed during the field work and if additional samples are required.

Transfer of Custody and Shipment

The custody of samples must be maintained from the time of sampling through shipment and relinquishment to the laboratory. Instructions for transferring custody are given below.

A chain-of-custody must accompany all samples. When transferring custody of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. This form documents sample custody transfer from the SERES-Arcadis JV field sampler, through the shipper, to the analytical laboratory. Because a common carrier will usually not accept responsibility for handling chain-of-custody forms, the name of the carrier is entered under "Received by," the bill-of-lading number is recorded in the comments section, and the chain-of-custody form is placed in a zip-lock plastic bag and taped to the inside lid of the shipping cooler. Copies of the chain-of-custody form will be placed in each cooler included in the shipment. Copies of the chain-of-custody and bill of lading will be retained by the SERES-Arcadis JV field sampler and placed into the project files, as well as sent to the SERES-Arcadis JV PM electronically.

Samples will be packaged for shipment and dispatched to the appropriate laboratory via overnight delivery service. Samples will be shipped within 24 hours of sampling if possible, or as soon thereafter. Shipping containers will be sealed for shipment to the laboratory, as described above. Custody seals will be applied to each cooler to document that the container was properly sealed, as well as to ensure the container was not tampered with during shipment. The custody seals will be placed on the coolers in such a manner that the custody seal would be broken if the cooler were opened.

Laboratory Custody Procedures

A designated sample custodian will accept custody of the samples and verify that all information on the sample labels matches that on the chain-of-custody. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt by the laboratory.

Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements.

To assure traceability of samples while in the possession of the laboratory, a unique laboratory identification number will be assigned to each sample.

The laboratory must document the following stages of analysis:

- Sample extraction/preparation
- Sample analysis
- Data reduction
- Data reporting

Laboratory personnel are responsible for the custody of the samples until they are returned to the sample custodian.

Sample Holding Times

Information on sample holding times and required preservation for each test method are provided in QAPP **Worksheet #19/30**.

Final Evidence Files

As the final phase of sample custody, the chain-of-custody records are archived in the project file. Laboratory custody forms, the sample preparation and analysis logbook, and data packages will become part of the laboratory final evidence file. Other relevant documentation including records, reports, correspondence, logs, photographs, and data review reports will be archived by SERES-Arcadis JV personnel.

QAPP Worksheet #28: Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4)
(EPA 2106-G-05 Section 2.3.5)

This worksheet includes information for each sampling technique, analytical method/SOP, matrix, and analytical group.

QAPP Worksheet #28a: Analytical Quality Control and Corrective Action (VOCs)

(VOCs in Soil/sediment by SW846 8260C)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 8260C
Laboratory SOP: CA-202

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	Relative percent difference (RPD) < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Trip blanks and Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Surrogates	Minimum 3 per field sample and QC samples	Percent recovery (%R) per DoD QSM ver. 5.3 Table C-23	Correct problem, then re-prep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-23

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Internal standards	Added to every field sample, standard, 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Laboratory Analyst, Department Manager	EICP area within 50% to +100% of ICAL midpoint standard.
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-23	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-23
MS/MSD[1]	One per analytical batch	%R, per DoD QSM ver. 5.3 Table C-23. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-23
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28b: Analytical Quality Control and Corrective Action (VOCs)

(VOCs in Groundwater by SW846 8260C)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 8260C
Laboratory SOP: CA-202

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	RPD < 35%	Qualify data as needed	Data Validator	RPD < 35%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Trip blanks and Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Surrogates	Minimum 3 per field sample and QC samples	%R per DoD QSM ver. 5.3 Table C-24	Correct problem, then re-prep and re-analyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-24
Internal standards	Added to every field sample, standard, 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Laboratory Analyst, Department Manager	EICP area within 50% to +100% of ICAL midpoint standard.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-24	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-24
MS/MSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-24. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-24
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

Notes: 1 LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28c: Analytical Quality Control and Corrective Action (SVOCs)

(SVOCs in Soil/sediment by SW846 8270D)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 8270D
Laboratory SOP: CA-226

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Surrogates	Full scan 6 per field sample and QC samples	%R per DoD QSM ver. 5.3 Table C-25	Correct problem, then re-prep and re-analyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-25
Internal standards	Added to every field sample, standard, 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Laboratory Analyst, Department Manager	EICP area within 50% to +100% of ICAL midpoint standard.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-25	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-25
MS/MSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-25. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-25
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28d: Analytical Quality Control and Corrective Action (SVOCs)

(SVOCs in Groundwater by SW846 8270D)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 8270D
Laboratory SOP: CA-226

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	RPD < 35%	Qualify data as needed	Data Validator	RPD < 35%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Surrogates	Full scan 6 per field sample and QC samples	%R per DoD QSM ver. 5.3 Table C-26	Correct problem, then re-prep and re-analyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-26
Internal standards	Added to every field sample, standard, 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Laboratory Analyst, Department Manager	EICP area within 50% to +100% of ICAL midpoint standard.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-26	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-26
MS/MSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-26. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-26
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28e: Analytical Quality Control and Corrective Action (SVOCs by SIM)

(SVOCs in Soil/sediment by SW846 8270D-SIM)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 8270D-SIM
Laboratory SOP: CA-213

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Deuterated monitoring compounds	Minimum of 2 deuterated monitoring compounds per field sample and QC samples	%R per DoD QSM ver. 5.3 Table C-27	Correct problem, then re-prep and re-analyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-27
Internal standards	Added to every field sample, standard, 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Laboratory Analyst, Department Manager	EICP area within 50% to +100% of ICAL midpoint standard.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-27	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-27
MS/MSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-27. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-27
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 40%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 40%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28f: Analytical Quality Control and Corrective Action (SVOCs by SIM)

(SVOCs in Groundwater by SW846 8270D-SIM)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 8270D-SIM
Laboratory SOP: CA-213

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	RPD < 35%	Qualify data as needed	Data Validator	RPD < 35%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Deuterated monitoring compounds	Minimum of 2 deuterated monitoring compounds per field sample and QC samples	%R per DoD QSM ver. 5.3 Table C-28	Correct problem, then re-prep and re-analyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-28
Internal standards	Added to every field sample, standard, 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Laboratory Analyst, Department Manager	EICP area within 50% to +100% of ICAL midpoint standard.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-28	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-28
MS/MSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-28. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-28
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 40%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 40%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28g: Analytical Quality Control and Corrective Action (PCBs)

(PCBs in Soil/sediment by SW846 8082A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 8082A
Laboratory SOP: CA-329

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
Surrogates	2 per field sample and QC samples	%R per DoD QSM ver. 5.3 Table C-17	Correct problem, then re-prep and re-analyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Laboratory Analyst, Department Manager	%R, per DoD QSM ver. 5.3 Table C-17
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-17	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-17
MS/MSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-17. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-17

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 30%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 30%
Second Column Confirmation	All positive results must be confirmed.	Results between primary and secondary column must be RPD ≤ 40%.	Apply qualifier if RPD >40% and discuss in the case narrative. The higher of the two results will be reported unless matrix interference is apparent.	Laboratory Analyst, Department Manager	RPD ≤ 40%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28h: Analytical Quality Control and Corrective Action (Hydrazines)

(Hydrazines in Soil/sediment by SW846 8315A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 8315A
Laboratory SOP: WI9431

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
LCS/LCSD[1]	One per analytical batch	%R lab generated control limits: 1,1-Dimethylhydrazine 69-128%; Hydrazine 61-122% Methylhydrazine 57-120%. Criteria doesn't apply if sample concentration is >4X the spike concentration	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R lab generated control limits: 1,1-Dimethylhydrazine 69-128% Hydrazine 61-122% Methylhydrazine 57-120%
MS/MSD[1]	One per analytical batch	%R lab generated control limits: 1,1-Dimethylhydrazine 10-108% Hydrazine 10-55% Methylhydrazine 10-46%. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R lab generated control limits: 1,1-Dimethylhydrazine 10-108% Hydrazine 10-55% Methylhydrazine 10-46%.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 30%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 30%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28i: Analytical Quality Control and Corrective Action (Metals)

(Metals in Soil/sediment by SW846 6010C/7471A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 6010C/7471A
Laboratory SOP: CA-608/ CA-611

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-3 and C-11	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-3 and C-11
MS/MSD[1]	One per 20 field samples	%R per DoD QSM ver. 5.3 Table C-3 and C-11. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-3 and C-11
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Post-digestion spike (ICP only)	Perform if MS/MSD fails. One per preparatory batch using same samples as MS/MSD if possible.	%R, 80-120%. Criteria applies for samples with concentrations < 50 X LOQ prior to dilution.	Qualify data as needed	Data Validator	%R, 80-120%
Serial dilution	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement. Criteria applies to sample concentrations > 50X LOQ prior to dilution.	Qualify data as needed	Data Validator	± 10% of the original measurement.

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28j: Analytical Quality Control and Corrective Action (Metals)

(Metals in Groundwater by SW846 6020A/7470A)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 6020A/7470A
Laboratory SOP: CA-627, CA-615

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	RPD < 35%	Qualify data as needed	Data Validator	RPD < 35%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-6 and C-12	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-6 and C-12
MS/MSD[1]	One per 20 field samples	%R per DoD QSM ver. 5.3 Table C-6 and C-12. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-6 and C-12
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Post-digestion spike (ICP only)	Perform if MS/MSD fails. One per preparatory batch using same samples as MS/MSD if possible.	%R, 80-120%. Criteria applies for samples with concentrations < 50 X LOQ prior to dilution.	Qualify data as needed	Data Validator	%R, 80-120%
Serial dilution	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement. Criteria applies to sample concentrations > 50X LOQ prior to dilution.	Qualify data as needed	Data Validator	± 10% of the original measurement.

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28k: Analytical Quality Control and Corrective Action (TOC)

(TOC in Soil/sediment by EPA Lloyd Kahn)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: EPA Lloyd Kahn
Laboratory SOP: CA-741

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > LOQ	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > LOQ
Equipment blanks	See Worksheet #20	No analytes detected > LOQ	Qualify data as needed	Data Validator	No analytes detected > LOQ
LCS/LCSD[1]	One per analytical batch	%R 80 to 120%	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R 80 to 120%
MS/MSD[1]	One per 20 field samples	%R 75 to 125%. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R 75 to 125%.
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 30%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 30%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28l: Analytical Quality Control and Corrective Action (Hardness)

(Hardness by calculation in Groundwater by SM 2340B)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SM 2340B
Laboratory SOP: CA-627

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	RPD < 35%	Qualify data as needed	Data Validator	RPD < 35%
Method blanks	One per analytical batch	No analytes detected > LOQ	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > LOQ
Equipment blanks	See Worksheet #20	No analytes detected > LOQ	Qualify data as needed	Data Validator	No analytes detected > LOQ
LCS/LCSD[1]	One per analytical batch	%R 80 to 120%	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R 80 to 120%
MS/MSD[1]	One per 20 field samples	%R 75 to 125%. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R 75 to 125%.
MS/MSD and LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.
2. SM = Standard Methods for the Examination of Water and Wastewater

QAPP Worksheet #28m: Analytical Quality Control and Corrective Action (Hexavalent Chromium)

(Hexavalent Chromium in Soil/sediment by SW846 7196A)

Matrix: Soil/Sediment
Concentration Level: Low
Analytical Method: SW846 7196A
Laboratory SOP: CA-625

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	RPD < 50%	Qualify data as needed	Data Validator	RPD < 50%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-9	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-9
LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%
MS/MSD[1]	One per 20 field samples	%R per DoD QSM ver. 5.3 Table C-9. Criteria doesn't apply if sample concentration is >4X the spike concentration	Dilute and reanalyze sample; persistent interference indicates the need to use the method of standard addition, alternative analytical conditions, or an alternative method.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-9

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
MS/MSD or Matrix Duplicate	One per analytical batch	RPD < 20%	Dilute and reanalyze sample; persistent interference indicates the need to use the method of standard addition, alternative analytical conditions, or an alternative method.	Laboratory Analyst, Department Manager	RPD < 20%
Soluble and Insoluble Pre-Digestion MS	One per analytical batch	%R 75-125%	Correct problem and re-homogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.	Laboratory Analyst, Department Manager	%R 75-125%
Post-Digestion MS	One per analytical batch	%R 85-115%	No specific corrective action.	Laboratory Analyst, Department Manager	%R 85-115%

Notes:

1. LCS/LCSD used when MS/MSD are not client-supplied.

QAPP Worksheet #28n: Analytical Quality Control and Corrective Action (Hexavalent Chromium)

(Hexavalent Chromium in Groundwater by SW846 7196A)

Matrix: Groundwater
Concentration Level: Low
Analytical Method: SW846 7196A
Laboratory SOP: CA-772

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	RPD < 35%	Qualify data as needed	Data Validator	RPD < 35%
Method blanks	One per analytical batch	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. Qualify data as needed	Laboratory Analyst, Department Manager	No analytes detected > 1/2 LOQ
Equipment blanks	See Worksheet #20	No analytes detected > 1/2 LOQ	Qualify data as needed	Data Validator	No analytes detected > 1/2 LOQ
LCS/LCSD[1]	One per analytical batch	%R per DoD QSM ver. 5.3 Table C-10	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-10
MS[1]	One per 20 field samples	%R per DoD QSM ver. 5.3 Table C-10. Criteria doesn't apply if sample concentration is >4X the spike concentration	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	%R per DoD QSM ver. 5.3 Table C-10
Matrix Duplicate or LCS/LCSD[1]	One per analytical batch	RPD < 20%	Qualify data as needed and note in case narrative.	Laboratory Analyst, Department Manager	RPD < 20%

Notes:

1. LCS/LCSD used when MS and matrix duplicate are not client-supplied.

QAPP Worksheet #29: Project Documents and Records

(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

This worksheet provides the minimum specifications for all data management tasks and deliverables, as well as procedures for controlling project documents, records, and databases. Its purpose is to ensure data completeness, data integrity, and ease of retrieval.

29.1 Data Management Specifications

29.1.1 Data Recording/Data Entry

Analytical results will be provided by the analytical laboratory in both a digital and a pdf format. The electronic data deliverable will be directly uploaded into the project database and will be used to directly produce tables, reducing the redundant manual entry of analytical results during report preparation and precludes transcription errors that may occur otherwise. Qualified technical personnel will review these electronic data against copies of final laboratory reports and DoD-related chemicals prior to releasing the data for validation. Any data that are manually transcribed from sampling logs or field books will be reviewed for accuracy by qualified staff other than staff who transcribed the data. The QA activities will be documented on a QA checklist.

29.1.2 Data Transformations and Data Reduction

Data transformation and reduction (data T&R) will be accomplished using EQuIS, ESRI ArcGIS, and Microsoft Excel. Data T&R will be reviewed for accuracy by qualified staff other than staff who performed the data T&R. The QA activities will be documented on a QA checklist.

29.2 Control of Documents, Records, and Databases.

All project documents and records will be generated, verified, and retained as shown in the Worksheet below.

Prior to field sampling, an eQAPP within FUDSChem will be created; the eQAPP will be fully-compliant with the project UFP-QAPP, incorporating methods, sample types, and quality specifications (Limit of Detection [LOD] Limit of Quantitation [LOQ], Holding Time, QC Sample Acceptance Limits, etc.). The pre-population of sampling event information (location information, sample type, matrix, methods, etc.) will also be performed to ensure accurate database field matching and status tracking within FUDSChem.

The analytical laboratory will generate portable document format (PDF) reports and will upload the Staged Electronic Data Deliverable (SEDD) 2a into FUDSChem for review against the approved eQAPP. Analytical data will be validated using the Automated Data Review (ADR) functions within FUDSChem. Following the ADR run, the data validator will review the FUDSChem ADR output and first reviewer's checklist against the laboratory report and supplement the FUDSChem ADR with a manual review, as necessary. A second data validation reviewer will perform the second review of the files to verify the project data quality requirements defined by the eQAPP and this UFP-QAPP have been satisfied. A data review report summarizing the findings from the data validation will be generated and upload it to the FUDSChem library.

Analytical data will be managed in the FUDSChem per the requirements specified in the New England District Data Management Plan to ensure data deliverables are in compliance with Performance Works Statement data requirements. Other types of data, which may be uploaded to FUDSChem include: spatial information pertaining to sample locations (northing, easting, and elevation), hydrogeological information and physical parameters (pH, temperature, conductivity, and turbidity for groundwater samples), and monitoring well information (such as screen intervals, depth to water, and well construction data).

Record	Generation	Frequency	Verification	Storage location/archival
Sample Collection and Field Records				
• Field logbook or data collection sheets • Chain-of-custody forms • Air bills • Contractor DQCRs • Deviations • CA reports • Correspondence • Field audit checklists	TBD, Field Sampler *SERES-Arcadis JV*	Daily or As Needed	Andy Vitolins, PM *SERES-Arcadis JV*	Project file and/or FUDSChem
• Data verification checklists	Jennifer Singer, Project Chemist, *SERES-Arcadis JV*	As Needed	Andy Vitolins, PM *SERES-Arcadis JV*	Project file and/or FUDSChem
• eQAPP in FUDSChem • Event planning in FUDSChem • Non-chemistry data pertaining to sample locations, hydrogeological information and physical parameters, and monitoring well information.	Jennifer Singer, Project Chemist or designee *SERES-Arcadis JV*	As Needed	Jennifer Singer, Project Chemist or designee *SERES-Arcadis JV*	Project File and/or FUDSChem
• Data validation report • Data usability assessment report	Jim Tomalia, Data Validator *Cadena*	As Needed	Jennifer Singer, Project Chemist *SERES-Arcadis JV*	Project file and/or FUDSChem

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Record	Generation	Frequency	Verification	Storage location/archival
Laboratory Records				
• Chain-of-custody records • Sample receipt records • Electronic data deliverables in Staged Electronic Data Deliverable (SEDD) Stage 2A deliverable format • Electronic data deliverables in Equis • Analytical results and supporting data • Sample data packages	Heather Manz, PM *Katahdin*	As Needed	Leslie Dimond, QA Manager *Katahdin*	Project file and/or FUDSChem
• Records of sample preparation • Records of sample analysis • Instrument calibration records • Raw data files	Laboratory analysts *Katahdin*	As Needed	Leslie Dimond, QA Manager *Katahdin*	Project file and/or FUDSChem

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QAPP Worksheet #31, 32, & 33 – Assessments and Corrective Actions

(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

This worksheet is used to document responsibilities and procedures for conducting project assessments, documenting assessments, responding to assessment findings, and implementing corrective actions (CAs). Appropriately scheduled assessments allow management to implement CA in a timely manner; thereby correcting non-conformances and minimizing their impact on DQOs/Project Quality Objectives.

ASSESSMENT

Different types of assessments are used for evaluating project activities, including, but are not limited to, laboratory assessments, field audits, and field documentation review. The tables below identify the type, frequency, and responsible parties of planned assessment activities that will be performed for the project.

CORRECTIVE ACTION

Once a process displays a characteristic out of specification with those required for the project or quality objectives, CA must be conducted to identify the cause of the deficiency or non-conformance. When the cause of the problem is identified, appropriate CA can be instituted and then monitored for effectiveness. Assessment response and corrective actions are specified in the tables below.

ROOT CAUSE ANALYSIS

Determining the root cause of a non-conformance is an integral part of the QC process. The depth and extent of the root cause analysis (RCA) depends on the situation; the root cause may be as simple (minor) as an overlooked step or procedure or it may be complicated. RCA is the responsibility of the functional manager or a designee. Input can be obtained as necessary from field personnel and technical advisors to identify the factors that led to the problem. The root cause is almost always “upstream” from where the problem is detected.

A two-step strategy will be employed for determining the root cause of a deficiency or nonconformance for this project. First, the problem will be traced back to the source. Second, the cause will be evaluated using basic questions such as who, what, when, where, why, and how. This process will be repeated until the cause is identified.

IMPLEMENTATION OF CORRECTION ACTION

Following the RCA, the project personnel will undertake the most effective remedy to correct the problem. Potential remedies to be considered may include the following:

- Supplemental personnel training
- Changes of equipment or modification of equipment currently in use
- Acquisition of supplemental equipment
- Implementation of new procedures or modification of existing procedures
- Changes in QC procedures

Successful implementation of CA will be documented on the Deficiency Notice or Non-conformance Report. Through follow-up phase surveillance, the SERES-Arcadis JV field sampler will verify that the CA implemented has rectified the non-conforming condition and is sufficient to prevent recurrence. The results of the CA will be presented in the RI Report.

Field procedure/documentation corrective actions and follow up will be verbally discussed then confirmed with an email between the SERES-Arcadis JV field sampler and SERES-Arcadis JV project manager.

Laboratory related issues identified by the validator and associated corrective action and follow up will be verbally discussed between the lab, validator and SERES-Arcadis JV project manager, and confirmed with an email.

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Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Review of QAPP, SOPs, and daily QC report with field staff	TBD *SERES-Arcadis JV*	Prior to sampling start up	Prior to sampling	Contained within daily QC report.	Prior to sampling
Daily logbook and field forms	TBD *SERES-Arcadis JV*	Daily	During field activities	Contained within written report.	As part of Draft Report
Laboratory assessment for appropriate certifications and capacity; QAPP review with laboratory staff	Jennifer Singer *Project Chemist* *SERES-Arcadis JV*	Prior to sampling start up	Prior to sampling	Receipt of copies of certifications. Email traffic concerning laboratory capacity prior to sampling start up. QAPP sign-off sheet received from the laboratory.	Prior to sampling
Daily tailgate safety meeting	TBD *SERES-Arcadis JV*	Daily	During field activities	Verbal debriefing and daily sign off log. If a safety violation occurs, an incident report is completed.	Last deliverable received no later than one week after field activities
Field sampling and chain-of-custody review against QAPP requirements	TBD *SERES-Arcadis JV*	Daily	During field activities	Communication in the form of an email.	Last email received no later than 24 hours after last sampling event
Laboratory report deliverables and analytical results review against QAPP requirements	Jennifer Singer *Project Chemist* *SERES-Arcadis JV*	Per sample delivery group (i.e., analytical samples received in each shipment)	Immediately following field sampling	Communication if the form of an email.	Three weeks after receipt of data
Data verification	Jennifer Singer *Project Chemist* *SERES-Arcadis JV* and/or Jim Tomalia *Data Validator* *Cadena*	Per sample delivery group (i.e., analytical samples received in each shipment)	Following analytical report	Communication in the form of an email requesting additional laboratory forms, backup data that may be missing and/ or clarification of the analytical report.	Three weeks after receipt of data
Data validation	Jim Tomalia *Data Validator* *Cadena*	Per sample delivery group (i.e., analytical samples received in each shipment)	Following analytical report	Communication in the form of an email requesting additional laboratory forms, backup data that may be missing and/ or clarification of the analytical report.	Three weeks after receipt of data

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Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsibility for Monitoring Corrective Action Implementation
Review of QAPP, SOPs, and daily QC report with field staff	Andy Vitolins *PM* *SERES-Arcadis JV*	Daily QC report will be amended with CA	Within 24 hours	TBD *SERES-Arcadis JV*	Andy Vitolins *PM* *SERES-Arcadis JV*
Daily logbook and field forms	TBD *SERES-Arcadis JV*	Daily QC report will be amended with CA	Within 24 hours	TBD *SERES-Arcadis JV*	Andy Vitolins *PM* *SERES-Arcadis JV*
Laboratory assessment for appropriate certifications and capacity; QAPP review with laboratory staff	Leslie Dimond *QA Manager* *Katahdin*	Response to email	Within 48 hours after notification	Leslie Dimond *QA Manager* *Katahdin*	Jennifer Singer *Project Chemist* *SERES-Arcadis JV*
Daily tailgate safety meeting	Andy Vitolins *PM* *SERES-Arcadis JV*	Included as part of the Incident Report	Within 48 hours after notification	TBD *SERES-Arcadis JV*	Andy Vitolins *PM* *SERES-Arcadis JV*
Field sampling and chain-of-custody review against QAPP requirements	Andy Vitolins *PM* *SERES-Arcadis JV*	Response to email	Within 24 hours after sampling	TBD *SERES-Arcadis JV*	Andy Vitolins *PM* *SERES-Arcadis JV*
Laboratory report deliverables and analytical results review against QAPP requirements	Leslie Dimond *QA Manager* *Katahdin*	If required, laboratory reports will be amended, and corrections noted in the case narrative	Within 72 hours of notification	Leslie Dimond *QA Manager* *Katahdin*	Jennifer Singer *Project Chemist* *SERES-Arcadis JV*
Data verification	Leslie Dimond *QA Manager* *Katahdin*	If required, laboratory reports will be amended, and corrections noted in the case narrative	Up to seven days	Leslie Dimond *QA Manager* *Katahdin*	Jennifer Singer *Project Chemist* *SERES-Arcadis JV*
Data validation	Heather Manz *PM* *Katahdin*	If required, laboratory reports will be amended, and corrections noted in the case narrative and documented in the validation report	Up to seven days	Heather Manz *PM* *Katahdin*	Jennifer Singer *Project Chemist* *SERES-Arcadis JV*

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QAPP Worksheet #34 –Data Verification and Validation Inputs

(UFP-QAPP Manual Section 5.2.1)
(EPA 2106-G-05 Section 2.5.1)

This worksheet is used to list the inputs that will be used during data verification and validation. Inputs include all requirements documents (*e.g.*, contracts, SOPs, planning documents), field records (both hard-copy and electronic), and interim and final reports. Data verification is a completeness check that all specified activities involved in data collection and processing have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data validation is the evaluation of conformance to stated requirements, including those in the contract, methods, SOPs, and the QAPP. Required documents as well as records subject to verification and validation are listed below. See **Appendix E** for the field SOPs.

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	**X**	
2	Contract	**X**	
3	Field SOPs	**X**	
4	Laboratory SOPs	**X**	
Field Records			
5	Field logbooks	**X**	**X**
6	Equipment calibration records	**X**	**X**
7	Chain-of-custody forms	**X**	**X**
8	Sampling diagrams/surveys	**X**	**X**
9	Relevant correspondence	**X**	**X**
10	Change orders/deviations	**X**	**X**
11	Field audit reports	**X**	**X**
12	Field CA reports	**X**	**X**
Analytical Data Package			
13	Cover sheet (laboratory identifying information)	**X**	**X**
14	Case narrative	**X**	**X**
15	Internal laboratory chain-of-custody	**X**	**X**
16	Sample receipt records	**X**	**X**
17	Sample chronology (i.e., dates and times of receipt, preparation, and analysis)	**X**	**X**
18	Communication records	**X**	**X**
19	Project-specific proficiency testing sample results		
20	LOD/LOQ establishment and verification	**X**	**X**
21	Standards traceability	**X**	**X**
22	Instrument calibration records	**X**	**X**
23	Definition of laboratory qualifiers	**X**	**X**

Item	Description	Verification (completeness)	Validation (conformance to specifications)
24	Results reporting forms	**X**	**X**
25	QC sample results	**X**	**X**
26	CA reports	**X**	**X**
27	Raw data	**X**	**X**
28	Electronic data deliverable	**X**	**X**

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QAPP Worksheet #35 –Data Verification Procedures

(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Follow-up inspections are conducted to ensure that procedures are being correctly performed, no changed conditions exist which may impact the quality of work, and lessons learned are being applied as identified. The responsible individual will inspect the relevant follow-up items from the checklist in the appropriate SOP at least as often as specified in this worksheet. **Worksheets #31, 32,** and **33** describe actions to be taken if nonconforming conditions are observed during the QC inspections.

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Field logbook (sampling methods and procedures)	• QAPP • Field SOPs	Establish required sampling methods were used and documented. Establish that any required field monitoring was performed, and results are documented. Verify that the sampling procedures and field measurements met performance criteria and that any deviations were documented in the field logbook.	**Daily:** TBD *SERES-Arcadis JV* **At conclusion of field activities:** Jennifer Singer *Project Chemist* *SERES-Arcadis JV*
Field logbook (documentation)	• QAPP • Field SOPs	Verify the records are present and complete for each day of field activities. Verify that all planned samples, including field QC samples, were collected and the 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.	**Daily:** TBD *SERES-Arcadis JV* **At conclusion of field activities:** Jennifer Singer *Project Chemist* *SERES-Arcadis JV*

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Chain-of-custody forms	• QAPP • Field SOPs • Laboratory SOPs/QA Manual	All samples to be analyzed by the laboratory will be shipped via overnight delivery service under chain-of-custody. Prior to shipment of the samples to the laboratory, the chain-of-custody will be checked by the SERES-Arcadis JV field sampler, for completeness and correctness. Upon receipt at the laboratory, the sample custodian will check the chain-of-custody forms and shipping documentation for verification against the sample coolers they represent and will sign and data the chain-of-custody to acknowledge sample receipt. The laboratory is responsible for verifying the integrity of the custody seals and that the sample containers are received in good condition. The Laboratory Information Management System will provide evidence of sample custody from receipt by the laboratory until appropriate disposal.	**Daily:** TBD *SERES-Arcadis JV* **Upon receipt:** *Sample Custodian* *Katahdin*
Laboratory correct action and report procedure	• QAPP • Laboratory SOPs/QA Manual	Routine CAs apply to all analytical QC parameters and analytical system specification as defined in the laboratory SOPs. Bench analysts have full responsibility and authority for performing routine CA, which are documented as part of the analytical record. Defective processes, holding time violations, systematic errors and quality defects that occur are to be reported by the analyst to the laboratory supervisor and a non-conformance record initiated. The Laboratory PM will then notify the SERES-Arcadis JV Project Chemist and PM. All notifications must be made in a timely manner. The non-conformance record should become part of the analytical record.	**Before release:** Leslie Dimond *QA Manager* *Katahdin* **Upon receipt:** Jennifer Singer *Project Chemist* *SERES-Arcadis JV*
Analytical data package	• QAPP • Laboratory SOPs/QA Manual	All data produced by the laboratory will be required to undergo several levels of review, which will include two levels of management review at the laboratory. The laboratory will review the data packages internals for completeness and verification that all the required forms and raw data are included for each data package type. The laboratory QA Manager may also select to review randomly chosen data packages for additional internal audits. Any deviations should be documented in the report narrative.	Leslie Dimond *QA Manager* *Katahdin*

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Analytical data package	• QAPP • Laboratory SOPs • USEPA National Functional Guidelines	The Project Chemist or Data Validator will verify that data have been received for all samples sent to the laboratory. An evaluation of the data will be performed to determine whether the laboratory met the QC requirements for the analysis as stated in the analytical method, laboratory SOPs, QAPP, and USEPA National Functional Guidelines for Data Validation. This verification should include (at a minimum): (1) review of dates of sample preparations and analyses to verify they have been performed within applicable holding times, (2) review of associated blanks for potential contamination, (3) determination that project quantitation limits were achieved, and (4) review of QC sample performance criteria. Any deviations should be documented in the report narrative.	Jennifer Singer *Project Chemist* *SERES-Arcadis JV* Jim Tomalia *Data Validator* *Cadena*
Laboratory electronic data deliverable	• QAPP • FUDSChem specifications	The electronic data deliverable will be reviewed using FUDSChem software for correctness and completeness.	Jennifer Singer *Project Chemist* *SERES-Arcadis JV* Jim Tomalia *Data Validator* *Cadena*

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QAPP Worksheet #36 –Data Validation Procedures

(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

RI actions will be documented and submitted for an overall data package certification. A certification package is prepared by the SERES-Arcadis JV for review by USACE. This package will document the steps taken to ensure the quality of the information relied upon to determine the nature and extent of constituents of concern at the Launch Area of the Nike Battery BU 51/52.

Data Validator:	Jim Tomalia, Cadena
Analytical group/method:	VOCs, SVOCs, PCBs, Hydrazines, Metals, TOC, Hardness, Hexavalent Chromium
Data deliverable requirements:	SEDD Stage 2A EQuIS 7
Analytical specifications:	VOCs by SW846 8260C SVOCs by SW846 8270D and 8270D-SIM PCBs by SW846 8082A Hydrazines by SW846 8315A Metals by SW846 6010C/6020A/7470A/7471A TOC by EPA Lloyd Kahn Hardness by calculation by Standard Methods 2340B Hexavalent Chromium by SW846 7196A
MPC	DoD QSM version 5.3 (see **Worksheets #12 and #28**)
Percent of data packages to be validated:	100% Stage 2A
Percent of raw data reviewed:	0%
Percent of results to be recalculated:	0%
Validation procedures:	UFP-QAPP, DoD QSM version 5.3, and USEPA Contract Laboratory Program (CLP) National Functional Guidelines as appropriate.

Notes:

100% of the data will be reviewed and verified. Data validation will be performed in FUDSChem.

QAPP Worksheet #37 – Data Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

(EPA 2106-G-05 Section 2.4)

The Data Usability Assessment will be performed by SERES-Arcadis JV for data associated with the Nike Battery BU 51/52. Cadena will perform third party data validation, to keep the data validation independent of the SERES-Arcadis JV project team. Documentation generated during the Data Usability Assessment will consist of data validation checklists with a summary of overall data usability and a summary table of qualified results. Data validation will be performed on the critical samples as defined in the UFP-QAPP, DoD QSM version 5.3, and USEPA CLP National Functional Guidelines as appropriate. 100% of the data will be reviewed and verified. Data validation will be performed in FUDSChem.

The Data Usability Assessment process involves data verification and validation. Data verification is the process by which laboratory results are checked to provide that the proper QC steps were performed, and key items have met QC objectives (both analytical and contractual). Key steps of the data verification include:

- identifying sample collection, handling, and analysis procedures
- documenting handling and analysis activities (e.g., QC checklist)
- verifying (internally, at the data generator level) all sampling, handling, on-site analytical laboratory data
- verifying laboratory data (e.g., laboratory-qualified data)
- verifying sampling, on-site analytical laboratory data
- verifying data package deliverable completeness
- reviewing the case narrative
- presenting all analytical results
- summarizing QC sample data

All required data deliverables must be present in the data package to proceed to the next step of data validation.

Data validation entails a review of the sample collection, handling, and QC data, to verify that the laboratory was operating within required limits, and which (if any) environmental samples were related to out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

The DQIs used to evaluate conformance with the project DQOs are presented below.

DQIs are generally defined in terms of six parameters:

1. representativeness
2. comparability
3. completeness
4. precision
5. accuracy
6. sensitivity

Each parameter is defined below. Specific objectives for the site actions are presented in other sections of this QAPP, as referenced below.

37.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions and is dependent on sampling and analytical variability and the variability of environmental media at the site. Actions have been designed to assess the presence of chemical constituents at the time of sampling. The QAPP presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. Use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to ensure each individual sample accurately and precisely represent site conditions.

37.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between phases of the actions (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in this QAPP, established QC procedures and use of appropriately trained personnel.

37.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90%. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

37.4 Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the site actions will adhere to established protocols presented in the QAPP. Checks for analytical precision will include the analysis of MS/MSD, laboratory duplicates and field duplicates. Checks for field measurement precision will include duplicate field measurements.

The precision of data will be measured by calculating the RPD by the following equation:

$$\text{RPD} = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one (1) of two (2) duplicate measurements.

B = Analytical result from the second measurement.

37.5 Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, MSs, blank spikes and surrogate standards will be used to assess the accuracy of the analytical data.

Accuracy will be calculated in terms of percent recovery as follows:

$$\%\,R = \frac{A-X}{B} \times 100$$

Where:

A = Value measured in spiked sample or standard.

X = Value measured in original sample.

B = True value of amount added to sample or true value of standard.

37.6 Sensitivity

Sensitivity is a quantitative measurement to determine if the analytical laboratory's procedures/methodologies and their associated detection limits can satisfy the project requirements as they relate to the project action limits. The detection limits as described in DoD QSM 5.3 are DL, LOD, and LOQ. Analytes that are not detected are reported as less than the LOD. Detects less than the LOQ but greater than the DL are reported as estimated and J-flagged by the laboratory. The LOD and LOQ are verified by the laboratory on a quarterly basis. The current DoD detection limits for the analytical laboratories are presented in **Worksheet #15**.

37.7 Data Validation and Usability

Cadena will validate data generated using the UFP-QAPP, DoD QSM version 5.3, and USEPA CLP National Functional Guidelines as appropriate. These procedures and criteria may be modified, as necessary, to address project-specific and method-specific criteria, control limits and procedures. Data validation will consist of data screening, checking, reviewing, editing and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Deviations from the analytical methods/QAPP requirements will be detailed by the laboratory in the report case narrative. Changes to the reporting requirements specified in the QAPP will be communicated to the laboratory PM and also noted on the chain of custody form as appropriate.

Upon receipt of laboratory data, the data validator will execute the following procedures:

- Evaluate completeness of data package.
- Verify that field chain-of-custody forms were completed, and samples were handled properly.
- Verify that holding times were met for each parameter. Holding time exceedances, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters were analyzed according to the methods specified.

- Review QC data (i.e., confirm that duplicates, blanks, and spikes were analyzed on the required number of samples, as specified in the method, and verify that duplicate and MS recoveries are acceptable).
- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the PM and/or Laboratory Manager, as appropriate.
- If data appear suspect, investigate the specific data of concern.

Deficiencies discovered because of the data review, as well as the CAs implemented in response, will be documented, and submitted in the form of a written report addressing the following topics, as applicable to each method:

- assessment of the data package
- description of any protocol deviations
- failures to reconcile reported data
- assessment of any compromised data
- overall appraisal of the analytical data
- table of site name, sample quantities, matrix and fractions analyzed

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean that data must be produced without QC qualifiers. Qualified data can provide useful information.

During the review process, laboratory qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results will be qualified with the following codes in accordance with DoD General Data Validation Guidelines, November 2019:

U The analyte was not detected and was reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result was an estimated value with an unknown bias. J+ The result was an estimated quantity, but the result may be biased high.

J The result was an estimated quantity, but the result may be biased low.

N The analysis indicates the presence of an analyte for which there was presumptive evidence to make a "tentative identification."

NJ The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value was the estimated concentration in the sample.

UJ The analyte was not detected and was reported as less than the LOD or as defined by the customer. However, the associated numerical value is approximate.

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.

It should be noted that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the SERES-Arcadis JV QC Manager at this point.

37.8 Validation Reports

The data validation reports will identify all deficiencies and the potential impact on the results. The SERES-Arcadis JV QC Manager (or his designee) will amend qualifiers generated during the validation process to the FUDSChem database. The validation checklists and the database will be the primary location of all applicable data qualifiers. Qualifiers will not be applied to the hard copy analytical reports.

37.9 Field Data Review

Field data are generated from in-field measurement, which may include a geophysical survey, well development and groundwater sampling. The quality objective for the in-field measurement activities is to obtain accurate measurements of sample characteristics, including aqueous pH, conductivity, temperature, turbidity, and dissolved oxygen, using appropriate equipment. Data are recorded in field logbooks or on field sampling sheets and calibration logs. Calibration logs will be reviewed by the SERES-Arcadis JV field sampler with other field documentation to identify any potential impacts to data quality and usability. Field logbooks are reviewed as part of the QC inspections.

37.10 Reconciliation with Data Usability Requirements

Data results will be examined to determine the performance that was achieved for each data usability criterion. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Data that has been rejected will not be used. Data that has been qualified but not rejected will be considered useable (i.e., qualified as estimated) and definitive data. If there is an instance where further limitations must be placed on qualified data, the associated data is non-definitive data and should be used for screening purposes only.

Additional action may be warranted when performance does not meet performance objectives for critical data. Options for CA relating to incomplete information, questionable results or inconsistent data may include any or all the following:

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty, and eliminate the need to qualify or reject data. If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- extrapolation of missing data from existing data points
- use of historical data
- evaluation of the critical/noncritical nature of the sample

If the data gap cannot be resolved by these actions, the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, the following action must be taken: additional sample collection and analysis.

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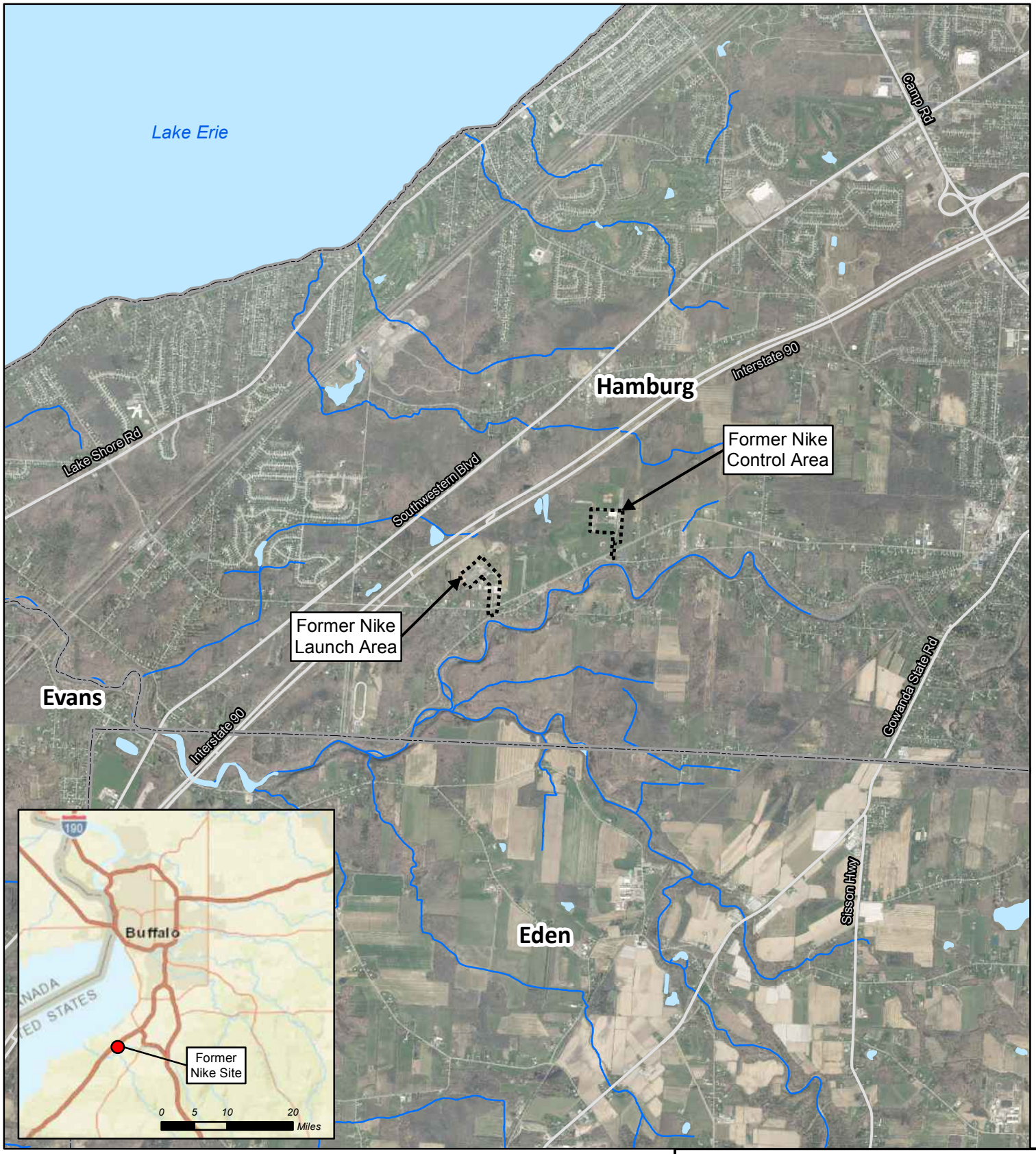
APPENDIX A
Site Maps



Former Nike Launch and Control Areas (Approx.)
Waterbody
Streams and Rivers
Major Roads
Town Boundaries

N
W E
S

0 0.5 1 2
Miles

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Source: GZA, 1999

FORMER NIKE BU -51/52 SITE

REGIONAL MAP

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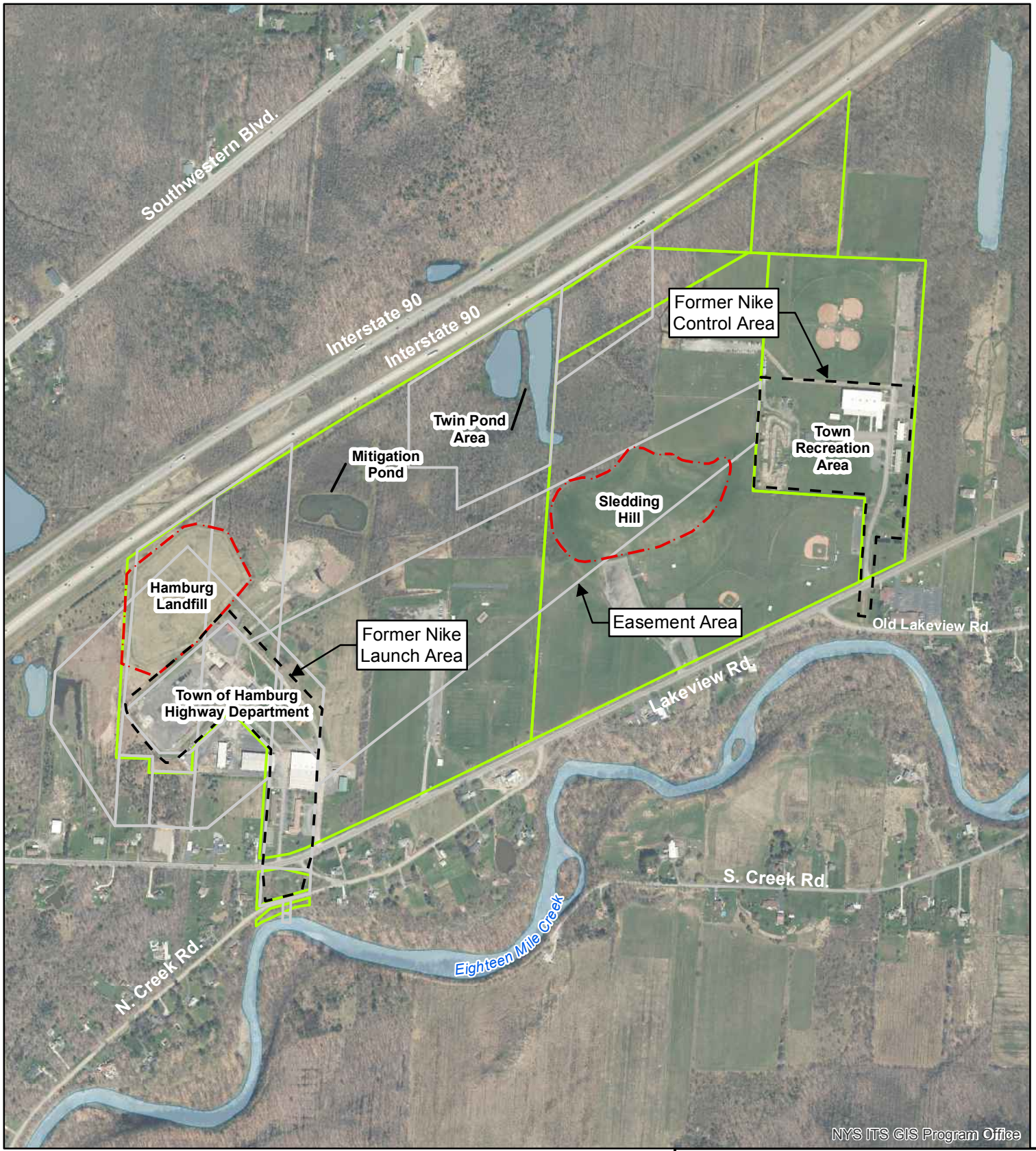
FIGURE
1

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 1_Regional Map.mxd



Former Nike Launch and Control Areas (Approx.)
Other Site Features
Perpetual Easements (Approx.)
Tax Parcel Boundary
Waterbody

0 200 400 800 Feet

N W E S

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Source: GZA, 1999

FORMER NIKE BU -51/52 SITE

SITE PLAN

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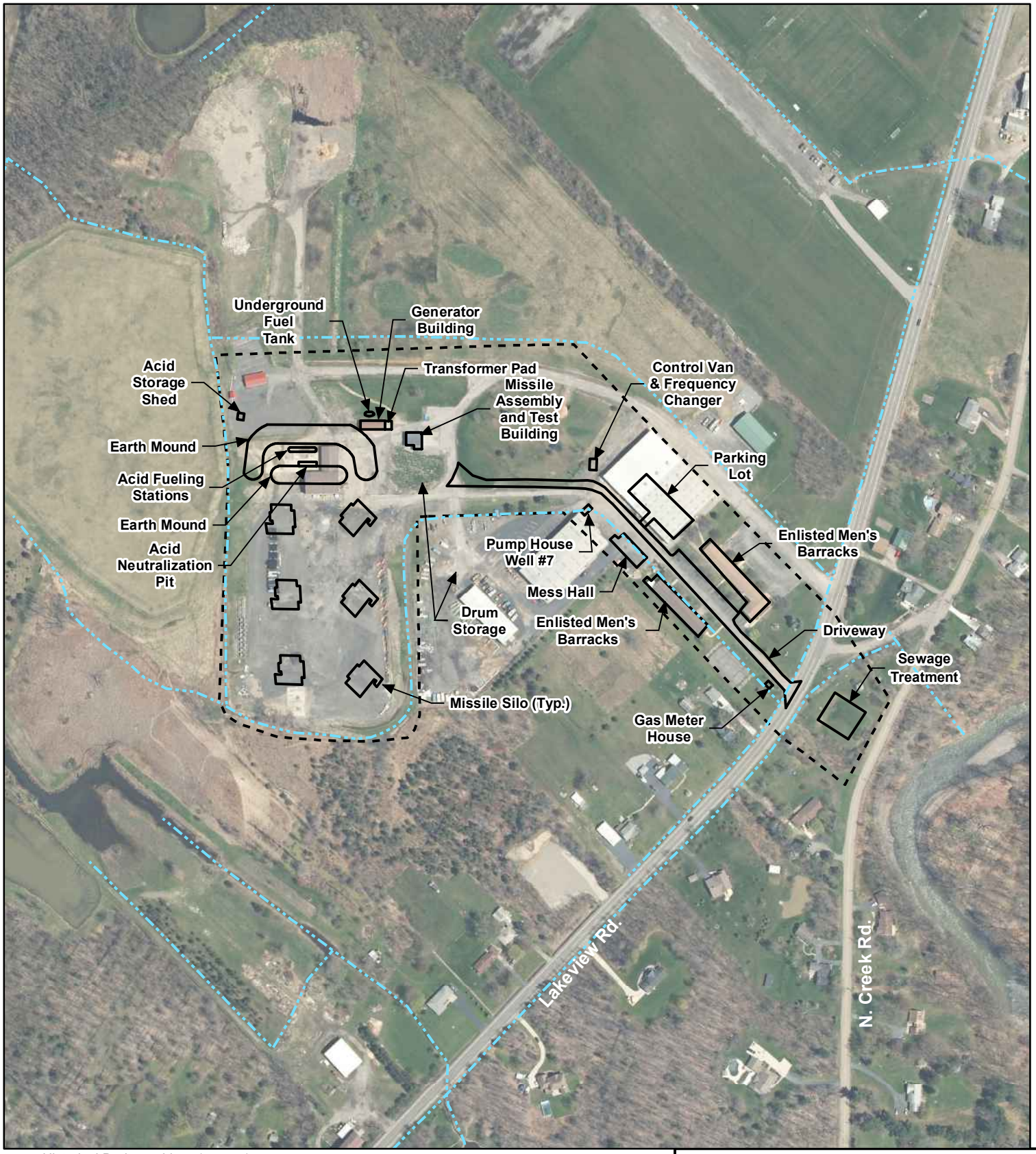
MARCH 2020

FIGURE 2

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 2_Site Plan.mxd



Historical Drainage Lines (approx.)
Historical Site Features
Former Nike Launch Area (approx.)

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Source: GZA, 1999

0 200 400 800 Feet

FORMER NIKE BU -51/52 LAUNCH AREA

HISTORICAL SITE FEATURES

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MARCH 2020

FIGURE 3

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 3_Historical Site Features.mxd



Historical Drainage Lines (approx.)
Current Site Features
Former Nike Launch Area (approx.)
Landfill

0 200 400 800 Feet

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Sources: Battelle, 2000; GZA, 1999

FORMER NIKE BU -51/52 LAUNCH AREA

CURRENT SITE FEATURES

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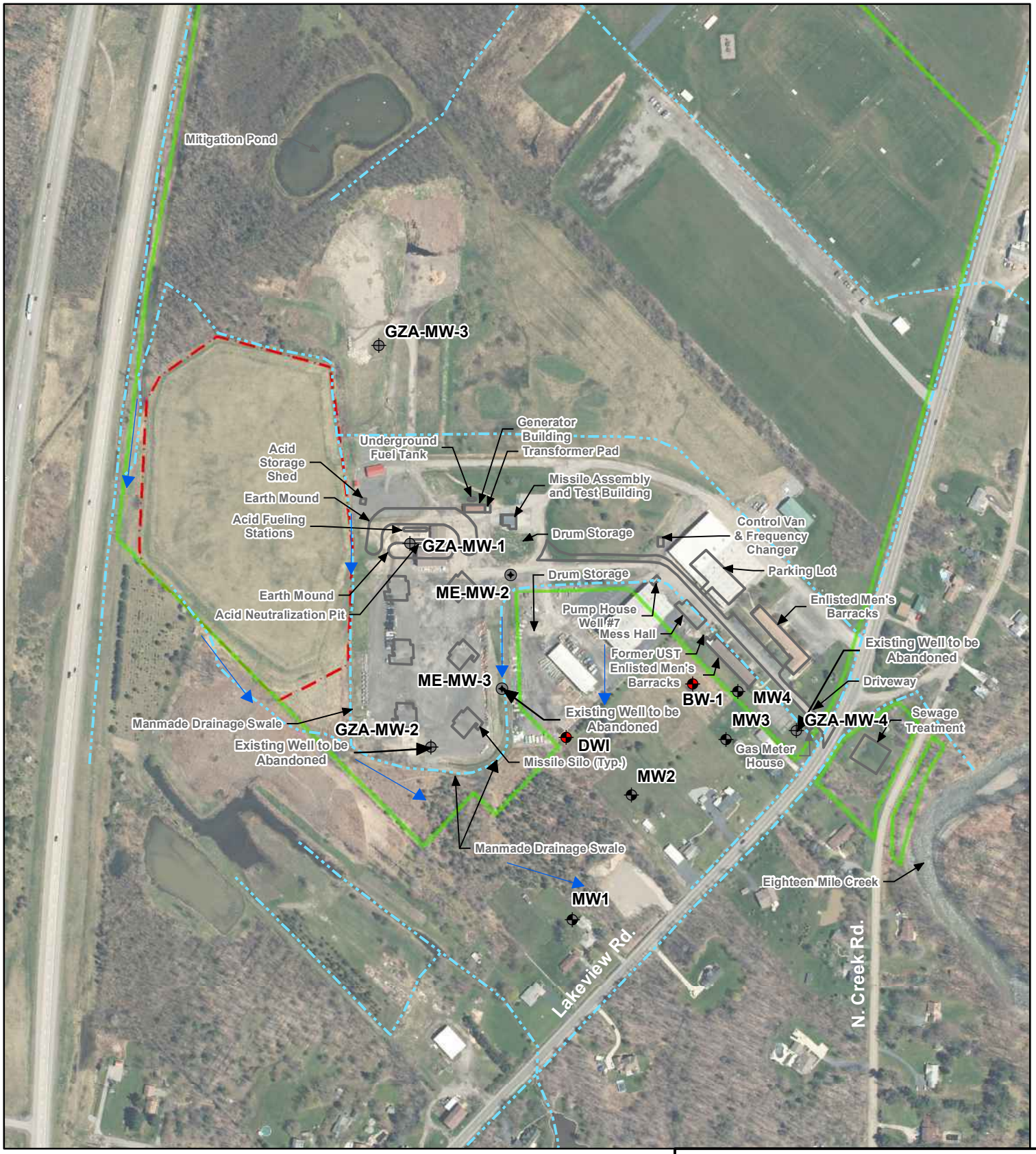
MARCH 2020

FIGURE 4

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 4_Current Site Features.mxd



Historical Site Features
Tax Parcel Boundary
Landfill
Historical Drainage Lines (approx.)
Drainage Direction
Monitoring Well (Battelle, 2000)
Monitoring Well (Hellert, 1999)
Monitoring Well (M&E, 1989)
Geoprobe Boring with Monitoring Well (GZA, 1999)

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator

0 100 200 400 600 800 Feet

FORMER NIKE BU -51/52 LAUNCH AREA

HISTORIC GROUNDWATER MONITORING WELL LOCATIONS

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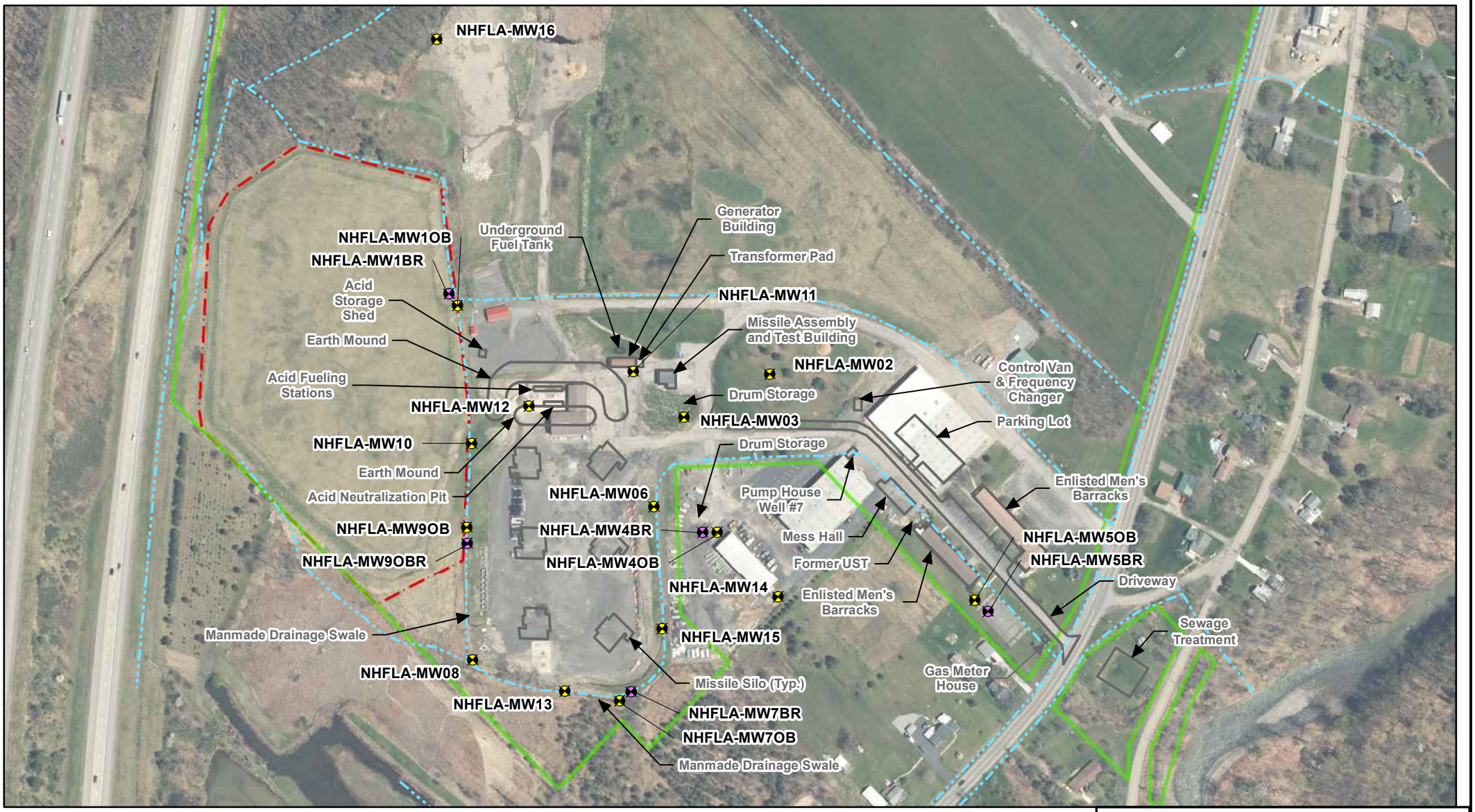
March 2020

FIGURE 5

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 5_Historic Groundwater Monitoring Well Locations.mxd



Proposed Bedrock Monitoring Well
Proposed Overburden Monitoring Well
Historical Drainage Lines (approx.)
Historical Site Features
Tax Parcel Boundary
Landfill

0 80 160 320 480 640 Feet

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator

FORMER NIKE BU -51/52 LAUNCH AREA

PROPOSED MONITORING WELL LOCATIONS

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FIGURE
6

Document Path: C:\Users\mai01043\OneDrive - ARCADIS\Zdrive\Nikee\New folder\Figure 6_Proposed Monitoring Well Locations.mxd



Proposed Overburden Well with Surface and Subsurface Soil Sample

Proposed Reference Surface and Subsurface Soil Sample

Historical Drainage Lines (approx.)

Tax Parcel Boundary

Landfill

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator

0 60 120 240 360 480
Feet

NOTE:
The reference soil sample locations are approximate and will be checked in the prior to collection to verify that the locations are representative of the site (e.g., similar soil types).

FORMER NIKE BU -51/52 LAUNCH AREA

PROPOSED SURFACE & SUBSURFACE SOIL SAMPLE LOCATIONS

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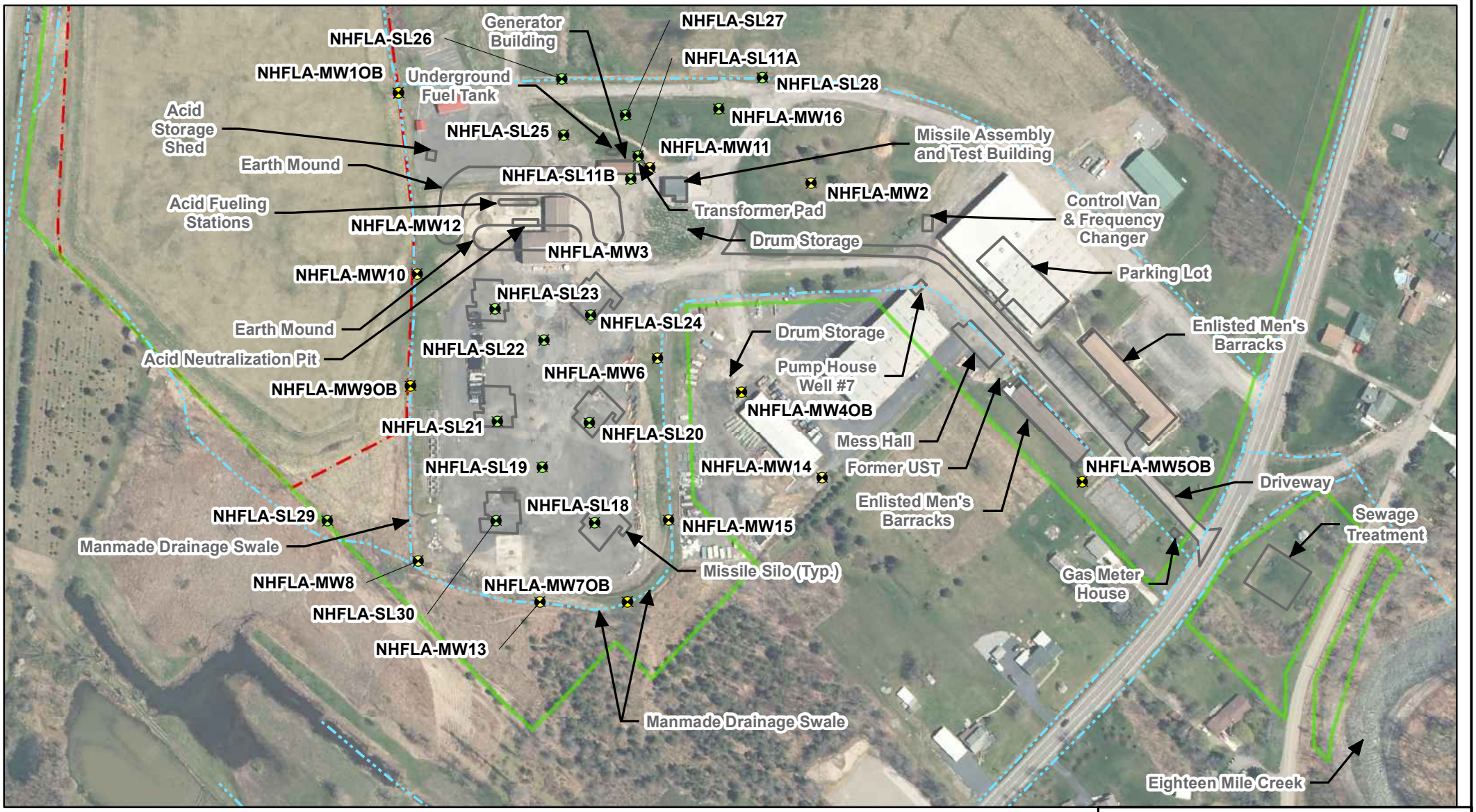
SEPTEMBER 2020

FIGURE 7A

Document Path: C:\Users\mai01043\OneDrive - ARCADIS\Zdrive\Nikee\New folder\Figure 7A_Proposed Surface Soil Sample Locations.mxd



Proposed Surface Soil Sample

Proposed Overburden Monitoring Well with Surface and Subsurface Sample

Historical Drainage Lines (approx.)

Historical Site Features

Tax Parcel Boundary

Landfill

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator

0 60 120 240 360 480 Feet

NOTE:
The reference soil sample locations are approximate and will be checked in the prior to collection to verify that the locations are representative of the site (e.g., similar soil types).

FORMER NIKE BU -51/52 LAUNCH AREA

PROPOSED SURFACE & SUBSURFACE SOIL SAMPLE LOCATIONS

SERES ARCADIS a joint venture

SEPTEMBER 2020

FIGURE 7B

Document Path: C:\Users\mai01043\OneDrive - ARCADIS\Zdrive\Nikee\New folder\Figure 7B_Proposed Surface Soil Sample Locations.mxd

APPENDIX B
Accident Prevention Plan

(provided separately)

APPENDIX C
RISK ASSESSMENT WORK PLAN

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

HUMAN HEALTH AND ECOLOGICAL RISK ASSESSMENT WORK PLAN

Nike Antiaircraft Missile Battery BU-51/52
Erie County, New York
Formerly Used Defense Site (FUDS) Program
Project Number C02NY0079

September 2020

HUMAN HEALTH AND ECOLOGICAL RISK ASSESSMENT WORK PLAN

Nike Antiaircraft Missile Battery BU-51/52

Erie County, New York

FUDS Project Number C02NY0079

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VERSION CONTROL

Issue	Revision No	Date Issued	Description
1	NA	3 December 2019	Draft Document
2	1	3 February 2020	Draft Final Document
3	2	3 March 2020	Draft Final Document
4	3	13 May 2020	Final Document
5	4	11 September 2020	Final Document

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B Record of Phone Conversation (Arcadis and Town of Hamburg), 21 November 2019
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ACRONYMS AND ABBREVIATIONS

ADAF	age dependent adjustment factor
ALM	adult lead model
ATSDR	Agency for Toxic Substances and Disease Registry
BERA	baseline ecological risk assessment
bgs	below ground surface
BTV	background threshold value
CalEPA	California Environmental Protection Agency
COPC	constituent of potential concern
CSF	cancer slope factor
CSM	conceptual site model
CT	central tendency
DAD	dermally absorbed dose
DoD	Department of Defense
DQO	data quality objective
EC	exposure concentration
EcoSSL	Ecological Soil Screening Level
ELCR	excess lifetime cancer risk
EPC	exposure point concentration
ERA	ecological risk assessment
ESV	Ecological Screening Value
FUDS	Formerly Used Defense Site
HEAST	Health Effects Assessment Summary Table
HHRA	human health risk assessment
HI	hazard index
HMW	high molecular weight
HQ	hazard quotient
HTRW	hazardous, toxic, and radioactive waste
IEUBK	Integrated Exposure Uptake Biokinetic
IPaC	Information for Planning and Consultation
IRIS	Integrated Risk Information System

IUR	inhalation unit risk
LANL	Los Alamos National Laboratory
MCL	Maximum Contaminant Level
µg/dL	micrograms per deciliter
NCP	National Contingency Plan
NHP	Natural Heritage Program
Nike Battery	Nike Antiaircraft Missile Battery BU-51/52
NYCRR	New York Codes, Rules, and Regulations
NYSDEC	New York State Department of Environmental Conservation
ORNL	Oak Ridge National Laboratory
PAH	polycyclic aromatic hydrocarbon
PbB	blood lead level
PEF	particulate emission factor
PPRTV	Provisional Peer-Reviewed Toxicity Values
ProUCL	ProUCL® 5.1.002
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RAGS	Risk Assessment Guidance for Superfund
RAWP	risk assessment work plan
RfCs	reference concentrations
RfD	reference dose
RI	remedial investigation
RME	reasonable maximum exposure
RSL	Regional Screening Level
SCO	soil cleanup objective
SF	slope factor
SLERA	screening level ecological risk assessment
SMDP	scientific/management decision point
SVOC	semi-volatile organic compound
TAL	target analyte list

TCL	target compound list
TOGS	Technical Operational Guidance Series
TPH	total petroleum hydrocarbons
UCL	upper confidence limit
UF	uncertainty factor
URF	unit risk factor
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
VF	volatilization factor
VISL	Vapor Intrusion Screening Levels
VOC	volatile organic compound
%	percent

1 INTRODUCTION

This risk assessment work plan (RAWP) presents the intended approach and methodology for evaluating the potential for adverse human health and ecological effects from exposure to constituents in soil and groundwater at the former Nike Antiaircraft Missile Battery BU-51/52 (Nike Battery), a Formerly Used Defense Site (FUDS), located in Hamburg, Erie County, New York. This RAWP is presented as an appendix to the project-specific Uniform Federal Program - Quality Assurance Project Plan (QAPP), which was developed to support the remedial investigation (RI) for the former Launch Area at the Nike Battery. The RI report will include a human health risk assessment (HHRA) and screening level ecological risk assessment (SLERA). The risk assessments will follow appropriate guidelines from the United States Environmental Protection Agency (USEPA) and United States Army Corps of Engineers (USACE) (e.g., EM 200-1-4 Volumes I and II).

This RAWP is presented in the following four sections:

- Section 1.0, Introduction – provides a brief explanation of the objectives and scope of the HHRA and SLERA, and contains information regarding the site location, site history, and constituents detected in soil and groundwater samples collected during previous investigations of the former Launch Area.
- Section 2.0, Human Health Risk Assessment Methodology – describes the process for evaluating potential for human exposure and associated health risks.
- Section 3.0, Ecological Risk Assessment Methodology – describes the process for evaluating ecological exposure and potential for ecological impairment.
- Section 4.0, References.

Information regarding the site location, site history, and constituents detected during previous investigations is briefly summarized below. Additional details are presented in the QAPP, and therefore not repeated in detail in this RAWP.

1.1 Site Location

The Nike Battery is located on Lakeview Road in Hamburg, New York (**Figures 1 and 2**). The former Launch Area is bound on the north by Interstate I-90; on the west by the former Easement Area of the Nike Battery; on the east by residential areas; and on the south by residential areas, Lakeview Road, and Eighteen Mile Creek. The closest residential housing is located approximately 500 feet to the south, on Lakeview Road.

1.2 Site History

Historically, the Nike Battery consisted of a Control Area, Easement Area, and Launch Area. The FUDS-eligible hazardous, toxic, and radioactive waste (HTRW) project is limited to the former Launch Area, and the former Control Area and Easement Area are not the subject of current investigations. The focus of this RAWP is therefore the former Launch Area. The former Launch Area is currently used by the Town of

Hamburg's Highway Department for office space, truck garage space, and storage of gravel piles, road salt, and other highway-related materials. **Figure 2** provides current features of the Nike Battery under ownership of the Town of Hamburg. A closed and capped landfill, formerly operated by the Town of Hamburg, is located north of, and adjacent to, the former Launch Area. **Figure 3** provides historical site features of the former Launch Area from the period of Department of Defense (DoD) ownership. A complete history of the site is presented in the QAPP.

1.3 Previously Detected Constituents

The QAPP includes summary tables showing constituents that were previously detected in soil and groundwater samples from the former Launch Area at concentrations exceeding regulatory benchmarks. Existing soil data were compared to the USEPA Regional Screening Levels (RSLs) for Residential Soil (USEPA 2019a). The RSLs are based on a target cancer risk of 1×10^{-6} or a target non-cancer hazard quotient (HQ) of 0.1. Existing soil data were also compared to the New York State Department of Environmental Conservation (NYSDEC) 6 New York Codes, Rules, and Regulations (NYCRR) Part 375 Residential soil cleanup objectives (SCOs). The surface soil data were also compared to the USEPA Region IV Ecological Screening Values (ESVs) for soil.

Existing groundwater data were compared to the USEPA tapwater RSLs (which are also based on a target cancer risk of 1×10^{-6} or a target non-cancer HQ of 0.1). The existing groundwater data were also compared to the Safe Drinking Water Act Maximum Contaminant Levels (MCLs) and Class GA Standards provided in NYSDEC Technical Operational Guidance Series (TOGS) Memorandum 1.1.1 for informational purposes. The constituents detected in soil and groundwater at concentrations above regulatory benchmarks are identified in **Section 10.2.2** of the **QAPP**, and include the following:

- Soil
    - Semi-volatile organic compounds (SVOCs): (high molecular weight [HMW] polycyclic aromatic hydrocarbons [PAHs], total petroleum hydrocarbons [TPH], hydrazine)
    - Inorganics: (aluminum, antimony arsenic, cadmium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, selenium, thallium, vanadium, zinc)
- Groundwater
    - Volatile organic compounds (VOCs): (acetone, chloroform, benzene, ethylbeneze, toluene, m-xylene, o-xylene, p-xylene)
    - SVOCs: (bis[2-ethylhexyl]phthalate, benzo(a)pyrene, benzo(b)fluoranthene, chrysene, dibenzofuran, fluoranthene, indeno[1,2,3-cd]pyrene)
    - Inorganics (aluminum, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, silver, sodium, thallium, cyanide)

These constituents are potentially associated with historical activities at the former Launch Area and will be used to help guide additional sampling and analysis, as proposed in the QAPP.

1.4 Proposed Supplemental Sampling

The QAPP outlines the process for collecting supplemental data for the site, including groundwater and surface and subsurface soil data. These data will be evaluated (along with historic data, as appropriate) in the HHRA and SLERA. The intermittent drainage ditches that exist in the former Launch Area are not expected to provide aquatic habitat, and sediment and surface water sampling is not proposed at this time (although soil sampling is proposed in these areas). However, if on-site aquatic habitat (e.g., vernal pools, permanent or semi-permanent water in the drainage ditches or swales) that may be affected by site conditions is observed, then surface water and/or sediment sampling may be considered.

2 HUMAN HEALTH RISK ASSESSMENT

The objectives of the HHRA will be to:

- Evaluate potential human health risks, currently and in the future, in the absence of any major action to control or mitigate potential contamination (i.e., baseline risks).
- Assist in determining the need for and extent of soil and groundwater remediation.
- Provide a basis for deciding if remedial action is necessary to protect human health or ecological receptors.

The HHRA will follow guidance outlined in the USEPA's *Risk Assessment Guidance for Superfund (RAGS), Volume I, Human Health Evaluation Manual, Part A* (USEPA 1989), Part D (USEPA 2001), Part E (USEPA 2004), and Part F (USEPA 2009), which is consistent with USACE guidance EM 200-1-4, *Risk Assessment Handbook - Volume I: Human Health Evaluation* (USACE 1999). The HHRA will be presented in a series of tables that follow the RAGS Part D (USEPA 2001) format (see **Attachment A**).

The HHRA will consist of the four-step process typically used to assess potential human health risks. These steps are summarized below and further described in the following sections.

- Data Evaluation – Relevant soil and groundwater data will be compiled and evaluated to determine the usability of the data and to select constituents of potential concern (COPCs).
- Exposure Assessment – Actual and/or potential constituent release and transport mechanisms will be identified, potentially-exposed human populations and possible exposure pathways will be described, concentrations of COPCs at potential points of human exposure will be determined, and human exposures to the COPCs will be estimated.
- Toxicity Assessment – Qualitative and quantitative toxicity information for each COPC will be summarized and toxicity values used to characterize risk will be compiled.
- Risk Characterization – The likelihood and magnitude of adverse health effects, in the form of incremental lifetime cancer risks and non-cancer hazard indices, will be estimated. Sources of uncertainty in the HHRA will be noted and discussed.

2.1 Data Evaluation

The data evaluation focuses on the compilation of usable analytical data and the selection of COPCs in soil and groundwater. The HHRA will be conducted using soil and groundwater data collected during the RI and historical data (as appropriate). Sampling plans for collecting surface and subsurface soil and groundwater data from the former Launch Area and for collecting site-specific reference sample data are described in the QAPP. Following sample collection and analysis, the laboratory analytical data will be validated in accordance with procedures outlined in the QAPP. Historical soil data may be combined with the RI soil sample data, if it is determined they meet data quality objectives (DQOs) established in the

QAPP and are representative of current conditions. The historical data will be evaluated for inclusion in the HHRA following the procedures outlined in the USEPA (1991) *Guidance for Data Useability in Risk Assessment*. Generally, data characteristics used to satisfy the quality assurance/quality control (QA/QC) requirements include precision, accuracy, representativeness, comparability, detection limit verification, and blank contamination elimination or qualification. All environmental data included in the risk assessments will have a definitive level of data quality and, as such, will be determined "usable." Also, the historical data will be compared to the proposed RI sample data set, and if results appear anomalous, they will be further evaluated for overall data usability. Any uncertainty regarding the usability or validity of the historical data will be discussed in the uncertainty section of the risk assessments.

Surface soil data for the HHRA will be defined as all samples from 0 to 1-foot below ground surface (bgs). Subsurface soil samples will consist of samples from unsaturated soils at 1-foot bgs to the water table. Prior subsurface investigations of the former Launch Area indicate the depth to shallow groundwater ranges from approximately 4 to 7 feet bgs and suggests hydrogeologic communication between the overburden and bedrock groundwater units. Assuming the overburden and bedrock groundwater are in hydrogeologic communication, all groundwater data will be initially evaluated as a single aquifer (i.e., data from 17 overburden and 5 bedrock monitoring wells will be combined) to identify COPCs for the potable use scenario. Groundwater data from only the overburden wells will be used to identify COPCs for the vapor intrusion pathway.

Once the HHRA data sets have been established, data summary tables will be prepared. The following parameters will be presented for each analyte detected in at least one sample within a given data set: minimum and maximum detected concentrations and associated data qualifiers, location of the maximum detected concentration, detection frequency, and range of reporting limits. In summarizing the analytical data and calculating exposure point concentrations (EPCs) for the HHRA, results of duplicate samples will be combined as follows:

- If a constituent is detected in both the parent and corresponding duplicate sample, the analytical results will be averaged.
- If a constituent is detected in only one of the two samples, the detected concentration will be used.
- If a constituent is not detected in either sample, the reporting limits will be averaged.

To focus the HHRA on those constituents that, if contacted, have the greatest potential to pose human health risks, the list of detected constituents will be narrowed to a final list of COPCs, according to the following screening process:

- Essential nutrients (i.e., calcium, magnesium, potassium, and sodium) will be categorically eliminated as COPCs.
- Detected constituent concentrations in soil will be compared to the most recent version of the USEPA residential soil RSLs available at the time of report preparation (USEPA 2019a). The RSLs are protective of exposures via incidental ingestion, dermal contact and inhalation. RSLs based on a target cancer risk of 1×10^{-6} or a target non-cancer HQ of 0.1 will be used. Constituents in soil with maximum concentrations greater than the residential soil RSLs will be identified as COPCs.

- Detected metals and PAH concentrations in soil will also be compared to background threshold values (BTVs) calculated using the site-specific reference soil sample data collected during the RI. Metals and PAHs with maximum concentrations greater than the residential soil RSLs but less than BTVs will be eliminated from further quantitative evaluation (USACE 2011). If metals and PAH concentrations are greater than the residential soil RSLs and BTVs, the site and reference soil sample data will be compared using other statistical methods, such as boxplots or two-sample hypothesis tests (e.g., t-test or Wilcoxon Mann Whitney test) to potentially support that the former Launch Area soil concentrations are consistent with natural or anthropogenic background conditions and as the basis for eliminating additional constituents from quantitative risk evaluation. Results of the data comparisons will be documented in the discussion of reference soil sample data and in the uncertainty section of the risk assessment.
- Detected constituent concentrations in groundwater will be compared to the most recent version of the USEPA tapwater RSLs available at the time of report preparation (USEPA 2019a). The tapwater RSLs are protective of chronic residential exposures to chemicals in groundwater via ingestion, dermal contact, and inhalation (of volatile chemicals only) exposure routes. RSLs based on a target cancer risk of 1×10^{-6} or a target non-cancer HQ of 0.1 will be used. Constituents in groundwater with maximum concentrations greater than the tapwater RSLs will be identified as COPCs.
- To evaluate the potential vapor intrusion pathway from shallow groundwater, volatile chemical analytical data from shallow groundwater samples will be compared to the USEPA Vapor Intrusion Screening Levels (VISLs; USEPA 2019b). VISLs based on a target cancer risk of 1×10^{-6} or a target non-cancer HQ of 0.1 will be used. A site-specific groundwater temperature based on the average of measurements collected during monitoring well sampling will be used to adjust the Henry's Law Constant used to calculate groundwater VISLs. Volatile chemicals in shallow groundwater samples with detected concentrations greater than the VISLs will be identified as COPCs for the vapor intrusion pathway.
- Constituents that do not have screening levels will be retained as COPCs and will be addressed qualitatively in the discussion of uncertainties.

2.2 Exposure Assessment

The objective of the exposure assessment is to estimate the type and magnitude of human exposure to constituents at or originating from a site. This is accomplished by establishing assumptions about the potential for human exposure (e.g., exposed populations, exposure pathways, exposure frequency), calculating representative EPCs for each COPC, and modeling human exposure in the form of daily intakes (termed dermally absorbed dose [DAD] for dermal contact exposure) or inhalation exposure concentrations (EC). These estimates of exposure are combined in the risk characterization with constituent-specific toxicity values to calculate incremental lifetime cancer risks and non-cancer hazards.

2.2.1 Current and Future Land Uses

The current and foreseeable future land use for the former Launch Area is industrial. The property is owned by the Town of Hamburg and utilized by the Highway Department for storage, office space, and

maintenance. The Town of Hamburg police department uses the area of the former enlisted men's barracks along the southwestern property boundary as a police and firearm training center. A small arms range with an earthen backstop berm is in the northwestern corner of the former Launch Area adjacent to the landfill.

The former Launch Area is zoned for residential-agricultural use (Town of Hamburg 2018). Residential properties are located approximately 500 feet south of the former Launch Area, along Lakeview Road. However, future residential use of the Launch Area is not considered a realistic or reasonable scenario based on the current use and land ownership, proximity to the Hamburg Landfill, and the presence of the underground silos. Per a conversation with the Town Engineer on 21 November 2019, redevelopment of the former Launch Area for future residential use is highly unlikely and is not part of the Town's plans (Personal communication with Mike Quinn, 2019). A record of the phone conversation is provided in **Attachment B**.

The former Control Area and Easement Area at the Nike Battery are northeast of the former Launch Area and are currently used as the Town of Hamburg's Lakeview Recreational Area, consisting of a playground, sledding hill, ball fields, and a BMX track (Bluestone 2019). The former Control Area and Easement Area are not the subject of current investigations.

2.2.2 Potential Receptors and Exposure Pathways

Based on the current and most likely future land use, potential human receptors include on-site commercial/industrial indoor workers, on-site outdoor maintenance workers, on-site construction/utility workers, and off-site residents.

A graphical representation of potentially complete exposure pathways for each receptor population is presented in the preliminary human health conceptual site model (CSM) (**Figure 4**). A complete exposure pathway includes: 1) a constituent source and release mechanism; 2) a transport or retention medium; 3) an exposure point where human contact with the contaminated medium may occur; and 4) an exposure route (i.e., ingestion, dermal absorption, or inhalation) at the contact point (USEPA 1989). If any one of these elements is missing, the pathway is considered incomplete. Exposure pathways on **Figure 4** were identified as "potentially complete" rather than "complete" because the presence of constituents at concentrations of concern in environmental exposure media will be determined following review of the RI sampling data.

RAGS Part D Table 1, included in **Attachment A**, presents the exposure scenarios and receptor populations that will be evaluated for the former Launch Area. The scenario time frame, source medium, exposure medium, exposure point, receptor population, receptor age, exposure route, type of analysis and rationale for selection or exclusion of an exposure pathway are provided.

Generally, human exposure to COPCs in soil may occur through incidental ingestion, dermal contact, and inhalation (particulates and volatile constituents). Under the current and most likely future land use, direct exposure to COPCs in groundwater may occur through incidental ingestion, dermal contact, and inhalation (mostly for VOCs), if shallow groundwater is encountered during site excavation. Indirect

exposure to volatile constituents in shallow groundwater could occur through inhalation of indoor air, if an occupied building overlies affected groundwater.

Groundwater at the former Launch Area is not used as a drinking water source. Potable water is supplied to the former Nike Battery areas and nearby residents by the Erie County Water Authority, which receives surface water from Lake Erie and the Niagara River. Therefore, use of groundwater as drinking water is currently an incomplete exposure pathway. However, there are no restrictions that would limit future use of the surficial aquifer as a drinking water source. There is the potential for groundwater to be used as drinking water by on-site commercial/industrial indoor workers and off-site nearby residents in the future. Therefore, these groundwater exposure pathways are potentially complete under hypothetical future exposure scenarios.

Based on a preliminary search of public databases containing well records and considering what is currently known about surrounding land uses (which are primarily residential), irrigation wells and industrial wells were not identified. Therefore, use of groundwater for irrigation or as industrial process water was not identified as a potential exposure scenario.

The HHRA will evaluate the following potentially complete exposure pathways:

On-site

- Current and future on-site indoor commercial/industrial workers may be exposed to COPCs in surface soil via incidental ingestion and inhalation of wind-blown fugitive dust and volatiles.
- Current and future on-site indoor commercial/industrial workers could be exposed to volatile COPCs, if present in underlying groundwater, that migrate from shallow groundwater to indoor air of on-site buildings.
- Under a hypothetical future scenario, on-site indoor commercial/industrial workers could be exposed to COPCs in groundwater via ingestion of groundwater used as potable water, dermal contact while washing hands, and inhalation of volatiles released to indoor air during potable use of groundwater. Although this exposure pathway is currently incomplete, there are no restrictions that would limit future use of the surficial aquifer as a drinking water source.
- Current and future on-site outdoor maintenance workers may be exposed to COPCs in surface soil via incidental ingestion, dermal contact, and inhalation of wind-blown fugitive dust and volatiles.
- Current and future on-site construction/utility workers may be exposed to COPCs in combined surface and subsurface soil via incidental ingestion, dermal contact, and inhalation of volatiles and dust during excavation activities.
- Given the shallow depth to groundwater, current and future on-site construction/utility workers may be exposed to COPCs in groundwater via incidental ingestion, dermal contact, and inhalation of volatiles in shallow groundwater that infiltrates the bottom of an excavation.

Off-site

- Current and future off-site residents could be exposed to volatile COPCs, if present in underlying groundwater, that migrate from shallow groundwater to indoor air of nearby residences.
- Under a hypothetical future scenario, off-site residents may be exposed to COPCs in groundwater via ingestion of groundwater used as potable water, dermal contact while washing hands and showering, and inhalation of volatiles released to indoor air during household use of groundwater (e.g., clothes washing). Although this exposure pathway is currently incomplete, there are no restrictions that would limit future use of the surficial aquifer as a drinking water source.

2.2.3 Exposure Point Concentrations

The QAPP, of which this RAWP is a part, states that 28 surface soil samples will be collected during the RI for target analyte list (TAL) metals, target compound list (TCL) VOCs, TCL SVOCs, and hydrazine to evaluate the potential risks associated with surface soil exposure to site users. Previous investigations did not include soil samples around the former transformer pad for polychlorinated biphenyl (PCB) analysis. Therefore, three surface soil samples will be collected for PCB analysis adjacent to the former transformer pad to assess if a release occurred. Additional samples may be collected to delineate PCBs in soil, if warranted. Up to 34 subsurface soil samples will be collected for analysis during installation of 17 overburden groundwater monitoring wells. One soil sample will be collected from 1 to 3 feet bgs at all locations. The second soil sample will be collected from the zone above the water table that exhibits the greatest impacts based on field screening conducted using visual, olfactory, and photoionization detector methods. If no indications of impacts are evident, the second soil sample will be collected from the 2-foot interval above the water table. Subsurface soil samples will be analyzed for TAL metals, TCL VOCs, TCL SVOCs, hydrazines, grain size, total organic carbon, and moisture content.

Twenty-two new monitoring wells (17 overburden and five bedrock wells) will be installed during the RI to evaluate the nature and extent of any groundwater impacts and for delineation of groundwater flow. One groundwater sample will be collected from each well during each of two groundwater monitoring events. Groundwater samples will be analyzed for TAL metals, TCL VOCs, TCL SVOCs, and hardness.

To determine the constituent concentrations to which an individual might be exposed over many years, representative EPCs will be calculated from the useable soil and groundwater data. The USEPA (1992, 1989) recommends that the arithmetic average concentration of the data be used for evaluating long-term exposure and that, because of the uncertainty associated with estimating the true average concentration at a site, the 95 percent (%) upper confidence limit (UCL) on the arithmetic average be used as the EPC. The 95% UCL concentration provides reasonable confidence that the true average will not be underestimated. The USEPA also indicates that where there is a question about the distribution of the data, a statistical test should be used to identify the best distributional assumption for the data set (USEPA 1992).

The ProUCL® 5.1.002 (ProUCL) program developed by the USEPA's Technology Support Center for Monitoring and Site Characterization will be used to plot the data, test the distributional assumptions, and

calculate 95% UCL concentrations. When entering the data into ProUCL, if a COPC was not detected in a sample, the sample reporting limit will be entered as a proxy concentration and the sample result will be coded as non-detect. ProUCL contains rigorous parametric and nonparametric statistical methods that can be used on full or uncensored data sets and on data sets with below detection limit observations (also called left-censored data sets). Depending on the distribution and 95% UCL estimation method, ProUCL will use only detected data or will incorporate detection limits.

The USEPA (2015) indicates that statistical estimates of EPCs may not be reliable for small data sets, or for data sets with only a few detected values (e.g., less than four to six). The soil samples planned for collection during the RI (i.e., 28 surface soil samples and up to 34 subsurface soil samples), potentially combined with historical soil data, and two rounds of groundwater samples from each of 22 monitoring wells should be sufficient quantity to calculate 95% UCL concentrations. Generally, for data sets with fewer than eight samples or fewer than five detected values, the maximum detected concentration will be used as the EPC. In instances where the 95% UCL concentration calculated by ProUCL is greater than the maximum detected concentration, the maximum detected concentration will be retained as the EPC.

The EPC used to evaluate the potable use scenario for each groundwater COPC will be estimated in accordance with USEPA OSWER Directive 9283.1-42 (USEPA 2014). Maximum detected concentrations (or the lesser of the maximum and 95% UCL where three or more data points are available for a monitoring well where the maximum concentration occurred) will be used as the EPC for each on-site groundwater COPC. Unless the hydrogeological investigation demonstrates the overburden and bedrock groundwater are in communication, separate EPCs will be used to represent overburden and bedrock groundwater. In the event residential wells downgradient of the former Launch Area are identified and groundwater COPCs in those wells are identified, maximum detected concentrations (or the lesser of the maximum and 95% UCL where three or more data points are available for a given well), will be used as the EPC for each COPC at the individual property. To evaluate the potential exposure of construction/utility workers to constituents in shallow groundwater, maximum detected concentrations of COPCs in monitoring wells identified as having depths to water less than 10 feet bgs will be used.

EPCs for other media (i.e., outdoor and indoor air) will be derived from numerical relationships between the chemical's concentration in soil or groundwater and the other medium based on chemical-specific properties and former Launch Area-specific parameters (e.g., wind speed and soil type).

EPCs for particulates in outdoor air will be estimated by multiplying the EPCs for the COPCs in soil by a particulate emission factor (PEF). The PEF is based on simplified soil-to-air transmission relationships described in *Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites* (USEPA 2002). The USEPA has derived a default PEF due to wind under passive conditions that is used to calculate the RSLs (USEPA 2019a). This default PEF (1.36E+09 m^3/kg) will be used to estimate COPC concentrations in outdoor air for the on-site indoor commercial/industrial worker and on-site outdoor maintenance worker. A site-specific PEF will be calculated for the on-site construction/utility worker using the equation for a construction scenario, available in USEPA 2002. USEPA default values will be used except for the Q/C variable presented in the site-specific PEF equation (Equation 5-5; USEPA 2002), which is based on the local climate and size of the site. The Q/C value will be based on climatic data from the closest regional airport.

EPCs for volatile COPCs in outdoor air will be estimated by multiplying the EPCs for the COPCs in soil by chemical-specific volatilization factors (VFs) based on equations in USEPA 2002. Site-specific VFs will be calculated for the on-site indoor commercial/industrial worker and on-site outdoor maintenance worker using soil parameter (e.g., bulk density and porosity) values specific to the predominant soil type encountered during the RI at the former Launch Area (Equation 4-8; USEPA 2002). Subchronic VFs will be calculated for on-site construction/utility workers, for which the assumed exposure duration is one year (Equation 5-14; USEPA 2002). The Q/C value in the VF equations will be based on climatic data from the closest regional airport.

EPCs for volatile COPCs in outdoor air of an excavation will be estimated by multiplying the EPCs for the COPCs in groundwater by a VF based on equations available in the Virginia Unified Risk Model (VDEQ 2019). To evaluate the vapor intrusion pathway, EPCs for the volatile COPCs in indoor air will be calculated assuming a shallow groundwater to indoor air attenuation factor of 0.001, which is the default value in the VISL calculator (USEPA 2019b).

Finally, to evaluate inhalation of volatile COPCs that could be released to indoor air during potable use of the groundwater, EPCs for volatile COPCs in groundwater will be multiplied by the VF (0.5 L/m^3) used to calculate the tapwater RSLs (USEPA 2019a).

2.2.4 Estimates of Constituent Intake/Exposure

Estimates of chemical intake and exposure will be developed to portray reasonable maximum exposure (RME) under current and future exposure scenarios. The RME scenario considers the highest exposure that might reasonably be expected to occur, one that is well above the average case of exposure but within the range of possibility. Use of RME parameter values to model baseline human health risks is a conservative approach, in that it yields upper bound cancer risk and non-cancer hazard estimates (USEPA 1989). If risks in excess of USEPA acceptable levels are determined for an exposure pathway, the pathway will be re-evaluated using central tendency (CT) exposure parameter values, where applicable, in place of upper-bound values specific to the RME analysis. Central tendency estimates may be developed to represent more realistic or site-specific conditions (e.g., outdoor worker exposure frequency and exposure time could be adjusted to reflect climatic conditions or the current and most likely future land use as a Highway Department storage area).

The RME parameters and the intake, exposure, and dose equations that will be used to estimate human exposure are presented in the RAGS Part D Table 4 series in **Attachment A**. For commercial/industrial workers and residents, chronic exposures will be estimated. For construction/utility workers, where the exposure duration is assumed to be one year, subchronic exposures will be estimated.

2.3 Toxicity Assessment

The toxicity assessment, also termed the dose-response assessment, will characterize the relationship between the magnitude of exposure and the potential that an adverse health effect will occur. Toxicity assessment involves determining whether exposure to a constituent can cause an increase in the

incidence of adverse health effects and characterizing the nature and strength of the evidence of causation. The toxicity information is then quantitatively evaluated, and the relationship between the dose of constituent received and the incidence of adverse health effects in the exposed population is evaluated.

2.3.1 Sources of Toxicity Data

The USEPA and other regulatory agencies have performed toxicity assessments for numerous chemicals, and the guidance they provide will be used in the HHRA. These include reference doses (RfDs) and reference concentrations (RfCs) for the evaluation of noncarcinogenic health effects from chronic and subchronic exposure to chemicals and cancer potency slope factors (CSFs) and unit risk factors (URFs) for evaluating incremental cancer risk from lifetime exposure to chemicals (i.e., excess lifetime cancer risks [ELCRs]).

Sources of toxicological information and toxicity values, in order of preference consistent with USEPA (2003) guidance, include:

- Tier 1 - Integrated Risk Information System (IRIS) (USEPA 2019c). IRIS is an internet database containing current information on human health effects that may result from exposure to chemicals in the environment and has received internal and external scientific review.
- Tier 2 - Provisional Peer-Reviewed Toxicity Values (PPRTV) (USEPA 2019d). PPRTVs were developed by the USEPA Office of Research and Development/National Center for Environmental Assessment/Superfund Health Risk Technical Support Center and are available as chemical-specific issue papers.
- Tier 3 - Additional sources of toxicity information, including but not limited to the California Environmental Protection Agency (CalEPA) Office of Environmental Health Hazard Assessment's chronic reference exposure levels and cancer potency values, the Agency for Toxic Substances and Disease Registry (ATSDR) minimal risk levels, and toxicity values published in the USEPA Health Effects Assessment Summary Tables (HEAST).

2.3.2 Adverse, Non-cancer Health Effects

The National Contingency Plan (NCP) (USEPA 1990) indicates that acceptable exposure levels for chemicals with non-cancer health effects should represent concentration levels to which the human population, including sensitive subpopulations (e.g., the elderly, young children), may be exposed without adverse health effects during a lifetime or part of a lifetime, incorporating an adequate margin of safety. The potential for non-cancer health effects associated with oral and dermal exposures is evaluated by comparing an estimated chemical intake or DAD over a specified time period with an RfD derived for a similar exposure period. The RfD is an estimate of a daily exposure level for the human population, including sensitive subpopulations, that is likely to be without an appreciable risk of deleterious effects during a lifetime. Therefore, the ratio of the intake or DAD to the RfD, termed the hazard quotient (HQ), assumes there is a level of exposure (i.e., the RfD) below which it is unlikely for even sensitive subpopulations to experience adverse health effects.

The potential for non-cancer health effects associated with inhalation exposures is evaluated by comparing COPC concentrations in air (i.e., ECs) to RfCs derived for a similar exposure period (USEPA 2009). HQs will be estimated by calculating the ratio of the EC to the RfC.

The toxicological information to be presented for COPCs with adverse, non-cancer health effects include: RfDs, RfCs, primary target organ(s), and uncertainty/modifying factors. Generally, order-of-magnitude uncertainty factor (UFs) reflect the various types of toxicological data (e.g., a laboratory animal study extrapolated to the human condition) used to estimate the RfDs and RfCs. Modifying factors, which can range from greater than zero to 10, reflect qualitative professional judgment regarding scientific uncertainties (e.g., the completeness of the overall database) not covered by the UF. Application of the uncertainty and modifying factors is intended to result in RfDs and RfCs that are protective of human health.

For current/future construction/utility workers, where exposure is assumed to occur over a one-year period, subchronic RfDs and RfCs will be used, where available. For some constituents, subchronic RfDs and RfCs may be estimated from chronic RfDs and RfCs available in IRIS by removing the UF applied where a chronic RfD or RfC was extrapolated from a subchronic study. Chronic RfDs and RfCs will be used as conservative approximations where subchronic values are not available or cannot be estimated.

RfDs are not available to evaluate dermal exposure. In their absence, oral RfDs will be used and adjusted following USEPA (2004) guidance to reflect absorbed dose. This allows for comparison between exposures estimated as absorbed doses and toxicity values expressed as absorbed doses.

2.3.3 Carcinogenic Effects

Regardless of the mechanism of effect, risk evaluation methods employed by the USEPA generally derive from the hypothesis that thresholds for cancer induction by carcinogens do not exist and that the dose-response relationship is linear at low doses. Based on this hypothesis, USEPA has derived estimates of incremental cancer risk from lifetime exposure to potential carcinogens. This is accomplished by establishing the carcinogenic potency of the chemical through critical evaluation of the various test data and fitting dose-response data to a low-dose extrapolation model. The cancer slope factor (CSF), which describes the dose-response relationship at low doses, is expressed as a function of intake [i.e., $(mg/kg\text{-}day)^{-1}$].

Incremental lifetime cancer risks will be estimated by multiplying an estimated daily intake or DAD prorated over 70 years by the CSF. The resulting risk estimate is expressed as a unitless probability (e.g., 2×10^{-5} or 2 in 100,000) of an individual developing cancer. The unitless probability represents the incremental (or increased) lifetime cancer risk associated with the estimated exposure above the background risk of developing cancer. This linear equation is valid only at low risk levels (i.e., below estimated risks of 0.01). According to the USEPA (1989), this approach does not necessarily give a realistic prediction of risk. The true value of the risk at trace ambient concentrations is unknown, and may be as low as zero.

To evaluate inhalation exposures, inhalation unit risks (IURs) that relate cancer potency to a chemical concentration in air are used (USEPA 2009). Incremental lifetime cancer risks will be estimated by multiplying the EC by the IUR.

The toxicological information to be presented for carcinogenic COPCs includes: CSFs, IURs, and weight-of-evidence classifications under USEPA's 1986 guidelines for carcinogen risk assessment (USEPA 1986) or cancer guideline descriptions under USEPA's revised carcinogen risk assessment guidelines (USEPA 2005a).

As with RfDs, USEPA has not derived slope factors (SFs) to evaluate dermal exposure. In their absence, SFs for oral exposure will be used and adjusted per USEPA guidance to reflect absorbed dose. This allows for risk estimation based on exposures estimated as absorbed doses and SFs expressed as absorbed doses.

The USEPA indicates that early-life exposure to carcinogenic chemicals with a mutagenic mode of action can result in a greater contribution to cancers appearing later in life (USEPA 2005b). To account for this, age-dependent adjustment factors (ADAF) are applied to the SFs and IURs for carcinogenic COPCs with a mutagenic mode of action. The USEPA (2005b) recommends a ten-fold adjustment for exposure during 0 and 2 years of age, a three-fold adjustment for exposures between 2 and 16 years of age, and no adjustment for exposures after turning 16 years of age. To facilitate the application of ADAFs, intakes and DADs will be calculated for each of the following age groups: 0-2 and 2-6 for the child (off-site residents); 0-2, 2-6, 6-16, and 17-26 for the adult (off-site residents). An ADAF of 10 will be applied to the cancer toxicity values to evaluate exposure from the ages 0 to 2, and an ADAF of 3 will be applied to evaluate exposure from the ages of 2 to 6 and 6-16. No adjustment will be made to evaluate exposure from the ages of 17 to 26.

2.3.4 Chemical Mixtures

USEPA guidance will also be used to account for the overall potential for human health risk from exposure to multiple constituents. For the evaluation of adverse, non-cancer health effects, USEPA guidance assumes that sub-threshold exposures to several chemicals at the same time could result in an adverse health effect. The sum of the non-cancer HQs (for individual chemicals, exposure routes, exposure pathways, or potentially exposed populations) is the hazard index (HI). Generally, HIs are only used in the evaluation of a mixture of chemicals that induce the same effect by the same mechanism of action. For this HHRA, the HIs of a mixture of constituents that can have different effects will be used as a screening-level approach, as recommended by the USEPA (1989). This approach may overestimate the likelihood of adverse, non-cancer health effects. Therefore, for HIs greater than 1, toxic endpoint-specific HIs will be calculated based on the toxicological endpoint(s) used to derive the RfD.

For the evaluation of carcinogenic risk, USEPA guidance indicates that the individual risks associated with exposure to each constituent can be summed. This approach will be used in this HHRA and assumes independence of action by the constituents involved (i.e., that there are no synergistic or antagonistic interactions and that all constituents produce the same effect: cancer).

2.3.5 Lead

The USEPA has not developed standard estimates representing a dose-response assessment for lead, because a clear threshold for some of the more sensitive effects in humans from exposure to lead has not been identified (ATSDR 2007). Rather, exposure to lead is typically evaluated in terms of the increase in blood lead levels (PbBs) following exposure. The United States Department of Health and Human Services' Centers for Disease Control and Prevention and the ATSDR have designated 5 micrograms per deciliter (µg/dL) as a PbB of concern to protect sensitive populations (e.g., neonates, infants, and children). However, the USEPA has not yet adopted this level of concern for use in lead risk assessments. The USEPA's stated goal for lead is that children have no more than a 5% probability of exceeding a PbB of 10 µg/dL. As such, this level is assumed to also provide protection for adults.

For adult workers exposed to lead in soil, the comparison of PbBs to the health-protective goal will be facilitated through use of the USEPA's Adult Lead Methodology and Adult Lead Model (ALM). With the ALM, concern is for a fetus that may be carried by an exposed pregnant female receptor, with the assumption that the results apply to both exposed females and males as well. The ALM evaluates exposure to soil only; as a result, adult construction/utility worker exposures to lead in shallow groundwater will not be evaluated.

For children (off-site residents) exposed to lead in shallow groundwater (e.g., drinking water), the evaluation will be facilitated through use of the USEPA's Integrated Exposure Uptake Biokinetic Model for Lead in Children (IEUBK). With the IEUBK, concern is for an exposed child during ages 0 to 7 years.

2.4 Risk Characterization

The risk characterization involves combining exposure estimates with toxicity information to assess the potential for adverse health effects for each human exposure scenario evaluated in the HHRA. In this section, the non-cancer hazards and cancer risks for each exposure scenario identified for the former Launch Area will be presented and discussed. The potential for adverse, non-cancer health effects from exposure to lead will also be discussed with respect to results of the ALM and IEUBK. Lastly, uncertainties associated with the HHRA process will be identified and described in terms of whether the potential for risk was likely to be over- or under-estimated.

2.4.1 Non-cancer Hazards

The potential for non-cancer health effects associated with constituent exposure is evaluated by calculating the ratio of an estimated intake or EC over a specified time period with a chemical-specific RfD or RfC derived for a similar exposure period. The RfD or RfC is an estimate of a daily exposure level for the human population, including sensitive subpopulations, that is likely to be without an appreciable risk of deleterious effects during a lifetime. The non-cancer HQ therefore assumes there is a level of exposure below which it is unlikely for even sensitive subpopulations to experience adverse health effects. The total individual HQs will be summed for each exposure pathway and scenario to yield HIs representative of the potential for adverse, non-cancer health effects from cumulative exposure. For the non-cancer assessment, exposure scenarios with an HI greater than 1 (i.e., 1E+00) will be of potential concern.

Where a pathway-specific HI exceeds 1, the HQs will be considered based on critical effect. That is, critical effect HIs will be developed by summing HQs of COPCs with RfDs or RfCs based on toxic effects on the same target organ.

2.4.2 Cancer Risks

Individual cancer risks are expressed as unitless probabilities of a person developing cancer. The total individual (i.e., COPC-specific) cancer risks will be summed for each exposure pathway and scenario to arrive at an estimate of the potential for cancer risk from cumulative exposure. For known or suspected carcinogens, the NCP established that acceptable exposure levels are generally concentration levels that represent an incremental upper-bound lifetime cancer risk in the range from 1×10^{-4} (i.e., 1E-04 or 1 in 10,000) to 1×10^{-6} (i.e., 1E-06 or 1 in 1,000,000) or less (USEPA 1990). The cancer risks estimated for each exposure scenario will therefore be compared to this risk range established by the NCP.

2.4.3 Lead Evaluation

The potential for adverse health effects from exposure to lead is evaluated through comparison of predicted PbBs to a health-protective target PbB. The USEPA's stated goal for lead is that children have no more than a 5% probability of exceeding a PbB of 10 µg/dL. As such, this concentration is assumed to also provide protection for adults.

The ALM and IEUBK will be used to predict PbBs and estimate the probability that target PbBs are exceeded following adult and child exposures to lead. The USEPA lead models will be accessed at: www.epa.gov/superfund/programs/lead/products.htm. Exposure to lead will be addressed in the RAGS Part D Adult Lead Worksheets and the IEUBK Lead Worksheets, which will be attached to the HHRA.

2.4.4 Uncertainty Discussion

Risk assessment involves the integration of complex analyses of constituent concentrations in the environment, the fate and transport of MC in the environment, the potential for human exposure, and constituent-specific potency and/or toxicity. Some uncertainties are associated with each component in this process.

Uncertainty in a risk assessment is typically accounted for by identifying the sources of uncertainty and characterizing whether the risks may be over- or under-estimated. Within this section, uncertainty will be qualitatively evaluated for each component of the HHRA, including identification of COPCs, exposure assessment, toxicity assessment, and risk characterization. The key site-related variables and major assumptions used in the HHRA (e.g., fate and transport models, exposure parameter values) will be identified and described. If warranted, uncertainty analyses may be conducted to demonstrate whether risks are likely to be over- or under-estimated.

3 ECOLOGICAL RISK ASSESSMENT

A SLERA will be performed to evaluate the likelihood of adverse ecological effects occurring as a result of exposure to COPCs associated with the former Launch Area. The SLERA will follow relevant guidelines, including guidance from USEPA (1997) *Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments* and USACE (2010) *Environmental Quality – Risk Assessment Handbook, Volume II: Environmental Evaluation*.

3.1 Screening-Level Ecological Risk Assessment Approach

The evaluation of ecological risks is typically a multi-step process that incorporates several scientific/management decision points (SMDPs). An SMDP requires a decision between the risk manager and risk assessment team to evaluate and approve or redirect the work up to that point. The SMDPs are intended to ensure that the ecological risk assessment (ERA) proceeds in an acceptable direction.

This RAWP assumes that the initial steps up through a SLERA will be completed for the former Launch Area. The SLERA represents the first two steps of the USEPA (1997) eight-step ERA process that includes:

- Step 1 – Preliminary Problem Formulation and Toxicity Evaluation
- Step 2 – Preliminary Exposure Estimate and Risk Calculation
- Step 3 – Problem Formulation
- Step 4 – Study Design and Data Quality Objective Process
- Step 5 – Verification of Field Sampling Plan
- Step 6 – Site Investigation and Data Analysis
- Step 7 – Risk Characterization
- Step 8 – Risk Management.

A flow chart of the SLERA process is presented in **Figures 5A, 5B,** and **5C**. The process for conducting the SLERA is presented in **Figure 5A**. Based on the results of the SLERA, a refined SLERA (**Figure 5B**) or a baseline ecological risk assessment (BERA) (**Figure 5C**) may also be conducted. However, for purposes of this RAWP it is assumed that only a SLERA will be required for the site.

Step 1 of the SLERA is the screening-level problem formulation process and ecological effects evaluation. For the screening-level problem formulation, a CSM will be developed to address the following five areas:

1. Environmental setting and known or suspected contaminants
2. Contaminant fate and transport mechanisms
3. Mechanisms of ecotoxicity associated with contaminants and likely categories of receptors that could be affected
4. Potentially complete exposure pathways
5. Selection of endpoints to screen for ecological risk.

A preliminary ecological CSM for the former Launch Area is presented in **Figure 6**. This ecological CSM conservatively assumes potentially complete exposure pathways for several media and receptors despite the lack of confirmed presence of suitable habitat. The ecological CSM will be refined in supplemental steps of the risk assessment process as additional information becomes available regarding environmental conditions and chemical characterization, affected media, available habitat, and potential ecological receptors present at the former Launch Area.

As part of Step 1, ecologists will conduct a site habitat assessment in the spring to document habitat conditions. The habitat assessment will include the documentation of former Launch Area cover types and a survey to document potential ecological resources. Information from the U.S. Fish and Wildlife Service (USFWS) and New York Natural Heritage Program (NHP) will also be reviewed to determine the potential occurrence of threatened/endangered species or critical habitats. An initial search of the USFWS Information for Planning and Consultation (IPaC) website (https://ecos.fws.gov/ipac/) in September 2019 found one federally-listed threatened mammal species (northern long eared bat [*Myotis septentrionalis*]) which may be present within Erie County, New York. A search of the NYSDEC Nature Explorer website conducted in September 2019 confirmed the potential presence of the federal- and state-threatened northern long-eared bat in the vicinity of the Site. Information from the habitat assessment will be used to update the ecological CSM in the SLERA.

Step 2 of the ERA process is the screening-level exposure estimate and risk calculation. In the SLERA, risk will be conservatively estimated by comparing measured chemical concentrations with ecotoxicological screening values. The data that will be used in the screening to characterize exposure concentrations will include only those data from areas of the former Launch Area that represent viable habitat for ecological receptors (if any). Much of the former Launch Area is developed by the Town of Hamburg Highway Department, and the surface of the former Launch Area consists of buildings, parking lots, storage areas, roads, and maintained lawns. These areas provide limited habitat to ecological receptors; therefore, data from these areas will not be considered in the SLERA. Areas of viable habitat, if present, will be identified based on the observations from the habitat assessment.

3.2 Screening-Level Problem Formulation

Problem formulation defines the goals and establishes the scope and focus of an ERA. The problem formulation includes a description of the environmental setting and a summary of available data. This information is used to identify the exposure pathways, target receptors, and potential effects, and serve as the focus for Step 2.

3.2.1 Preliminary Ecological Characterization

The former Launch Area is a developed area characterized by areas of asphalt and gravel pavement and building structures. The proximity of human activities and the degree to which the area has been reworked is expected to limit usage for much of the site by ecological receptors. The intermittent drainage ditches that exist in the former Launch Area are not expected to provide aquatic habitat. Other surface water bodies in the vicinity of the site include the mitigation pond (approximately 0.5 miles north of the site), the twin pond area (approximately 1 mile north of the site), and Eighteen Mile Creek (approximately 1 mile southeast of the site). These areas provide aquatic habitat for ecological receptors. However, preliminary data indicate that there are no complete transport mechanisms for site-related constituents to reach these areas, and therefore it is expected that they will not be evaluated in the SLERA. If the RI data indicate the potential for transport, then these areas may be considered in the risk assessment process.

As described in the SLERA approach below, the preliminary ecological characterization will be verified and further refined based on a habitat field reconnaissance and ecological receptor survey.

3.2.2 Potential Ecological Receptors

Based on the available information regarding potential contaminant transport and site use by ecological receptors, preliminary ecological exposure pathways considered potentially complete include exposure of vegetation communities, terrestrial invertebrates, and wildlife (primarily birds and mammals) to COPCs present in soil, and ingestion of bioaccumulative COPCs via the food chain by birds and mammals that may reside or forage in the habitats present on site.

It is necessary to select representative species to evaluate the different feeding guilds and trophic levels (e.g., herbivorous and omnivorous birds and mammals) in the food web. The ecological receptors determined to be appropriate representative receptors for the site include the following:

- Herbivorous birds and mammals: mourning dove (*Zenaida macroura*) and meadow vole (*Microtus pennsylvanicus*)
- Invertivorous/omnivorous birds and mammals: American robin (*Turdus migratorius*) and short-tailed shrew (*Blarina brevicauda).* American robin and short-tailed shrew will be used to represent both invertivorous and omnivorous birds and mammals by modifying the diet assumptions to represent each feeding guild.
- Carnivorous birds and mammals: red fox (*Vulpes Vulpes*) red-tailed hawk (*Buteo jamaicensis*)

As part of the SLERA site figures and aerial photographs will be reviewed to identify areas with potential ecological habitat in the vicinity of the site. Habitat characteristics will be further documented during a site visit to identify vegetative cover types and identify areas of potential impact (e.g., visible contamination, stressed vegetation). The site visit will also document potential ecological receptor species observed or expected to occur at the site based on available habitat.

According to the USFWS, the federally-listed threatened northern long eared bat may be present within Erie County, New York. A search of the NYSDEC Nature Explorer website (http://www.dec.ny.gov/natureexplorer/app/) confirmed the potential presence of the federal- and state-threatened northern long-eared bat in the vicinity of the Launch Area. Bats typically forage in open habitat, frequently over bodies of water (Fenton and Bell 1979; Barclay 1991; Saunders and Barclay 1992) and are expected to eat both aquatic emergent and terrestrial invertebrates. Due to the lack of water bodies at the site this species is not expected to utilize the site habitat. However, the SLERA will evaluate the potential site usage of the northern long-eared bat, and (if appropriate) the potential species-specific risk.

3.2.3 Potential Exposure Pathways

The preliminary CSM (**Figure 6**) identifies potential ecological receptors and exposure pathways at the former Launch Area. Based on the available information regarding potential contaminant transport and site use by ecological receptors, potentially complete ecological exposure pathways are exposure of plant communities, terrestrial invertebrates, and wildlife (primarily birds and mammals). For those areas of the Launch Area that represent viable ecological habitat, ecological receptors may be exposed to on-site surface soil (0 to 1-foot bgs) through ingestion, dermal contact (during feeding or preening activities), and (for bioaccumulative constituents) uptake through the food web. Potential exposure for ecological receptors to subsurface soils would be limited to instances of ecological receptors burrowing activities. Exposure to groundwater is not expected because the depth to groundwater is approximately 4 to 7 feet bgs, unless groundwater seeps are identified at the Launch Area. The intermittent drainage ditches that exist in the former Launch Area are not expected to provide aquatic habitat. Other surface water bodies in the vicinity of the site may provide aquatic habitat for ecological receptors; however, preliminary data indicate that there are no complete transport mechanisms for site-related constituents to reach these areas.

3.2.4 Preliminary Assessment Endpoints and Measurement Endpoints

Assessment and measurement endpoints will be used to help guide the evaluation of ecological risks. Assessment endpoints are formal expressions of the actual environmental value to be protected from risk (Suter et al. 1993) and are typically tied directly to specific ecological values needing protection. The assessment endpoints identified for evaluation in the SLERA are based on the complete and significant exposure pathways identified in the CSM (**Figure 6**). Measurement endpoints include measures of exposure and measures of effects, and are quantitative expressions of observable or measurable changes that are used to evaluate the assessment endpoints (USEPA 1997). For this SLERA, the preliminary assessment endpoints and measurement endpoints are defined as follows:

Exhibit 1: Preliminary Assessment Endpoints and Measurement Endpoints

Assessment Endpoints	Measurement Endpoints
Soil Invertebrates	Compare soil data to conservative benchmarks for soil invertebrates
Plant Community	Compare soil data to conservative benchmarks for plants
	Observations of stressed vegetation
Birds and Mammals	Compare soil data to conservative benchmarks for birds/mammals

3.3 Screening Level Exposure Estimate and Risk Calculation

As described in **Figure 5A**, a screening will be conducted to compare maximum detected concentrations in each media for each COPC to reference concentrations and conservative ecological screening values. Historical data on chemical concentrations for the former Launch Area are available from several reports (Metcalf and Eddy 1989; GZA 1999; Battelle 2000). As described in the QAPP, additional soil data are proposed. Historical soil data may be combined with the RI soil sample data, if it is determined they meet DQOs established in the QAPP and are representative of current conditions.

Based on the existing data the relevant medium for ecological receptors at the former Launch Area is expected to be surface soil for those areas that represent viable habitat (if any) including the vegetated portions of the swales and ditches. Although most of the former Launch Area is expected to represent limited habitat, the soil data from those areas of the former Launch Area that may provide habitat will be compared to reference concentrations and relevant screening benchmarks as a preliminary assessment of potential ecological risks. Surface soil data for the SLERA will be defined as all samples from 0 to 1-foot bgs. The analytical data will be summarized in a manner generally consistent with the HHRA approach for calculating EPCs (e.g., handling duplicate samples), as described in **Section 2.1**.

3.3.1 Ecological Screening Values

The data for the areas of the former Launch Area that represent viable habitat (if any) will be compared to reference concentrations as well as appropriate ecotoxicological screening values. The screening values will be selected using the following hierarchy:

- USEPA Ecological Soil Screening levels (EcoSSLs; USEPA 2007);
- USEPA Region 4 Ecological Soil Screening Values (USEPA 2018); and
- Department of Energy Laboratories i.e., Los Alamos National Laboratory (LANL 2017).

Soil screening tables using the selected ESVs and historical soil data are provided in **Attachment C**. These values may be supplemented (as necessary) based on the results of the soil sampling and availability of suitable benchmarks. For example, foodweb models and toxicological endpoints may be

used to back-calculate protective soils benchmarks for birds and mammals for bioaccumulative compounds if published values are not available.

3.3.2 Screening Level Risk Calculation

In this step, the maximum detected concentration in site soils (and other media if appropriate) will be compared with the corresponding ecological screening values to derive screening level risk estimates. COPCs will be evaluated using the HQ method. HQs are calculated by dividing the appropriate exposure point concentration (for the SLERA, maximum detected concentrations) by the corresponding medium-specific screening benchmark. COPCs with HQs greater than or equal to 1 will be carried forward for additional evaluation in a refined SLERA (**Figure 5B**) and/or a BERA (**Figure 5C**). Detected COPCs for which ESVs are not available may also be carried forward. If the maximum detected concentration of a COPC is below the ecological screening value, the COPC is not considered to pose risk to ecological receptors for that medium.

3.4 Recommendation for Scientific Management Decision Point 1

The results from the SLERA will presented in RI report. The SLERA will describe ecological conditions at the former Launch Area, present a revised ecological CSM, and present the results from the screening-level exposure estimate and risk calculation. If the exposure estimate for a constituent is below the ecological soil screening value, it is not considered to pose risk to ecological receptors. The SLERA will also discuss potential sources of uncertainty in the assessment.

The SLERA process will conclude with a SMDP where the risk manager and risk assessment team will decide one of the following conclusions:

1. Ecological threats are almost, or entirely, absent; therefore, no further characterization of ecological risk is needed.
2. Based on the conservative assumptions in the SLERA, there is the possibility of adverse ecological effects, and a more detailed assessment with more information about the former Launch Area is needed.

If the process continues with a more detailed assessment, then either a refined SLERA (BERA Step 3a) or a more complete BERA (Steps 3b through 7 of the USEPA's eight-step process) may be conducted. The results from the SLERA (Steps 1 and 2) will focus the more detailed assessment by identifying exposure pathways and preliminary COPECs and eliminating those contaminants and exposure pathways that pose negligible risks. Further SLERA refinement in BERA Step 3a may incorporate exposure estimates using more realistic assumptions and additional methodologies to better estimate potential exposure. For example, Step 3a may involve refining risk calculations through food chain modeling based on both conservative exposure parameters (maximum food intake rates and bioaccumulation factors) as well as refined exposure parameters (average food intact rates and bioaccumulation factors). The results of Step 3a will serve to identify if additional evaluation of ecological

risk via a more complete BERA is necessary. Major sources of uncertainty in the ecological risk assessment process will also be documented.

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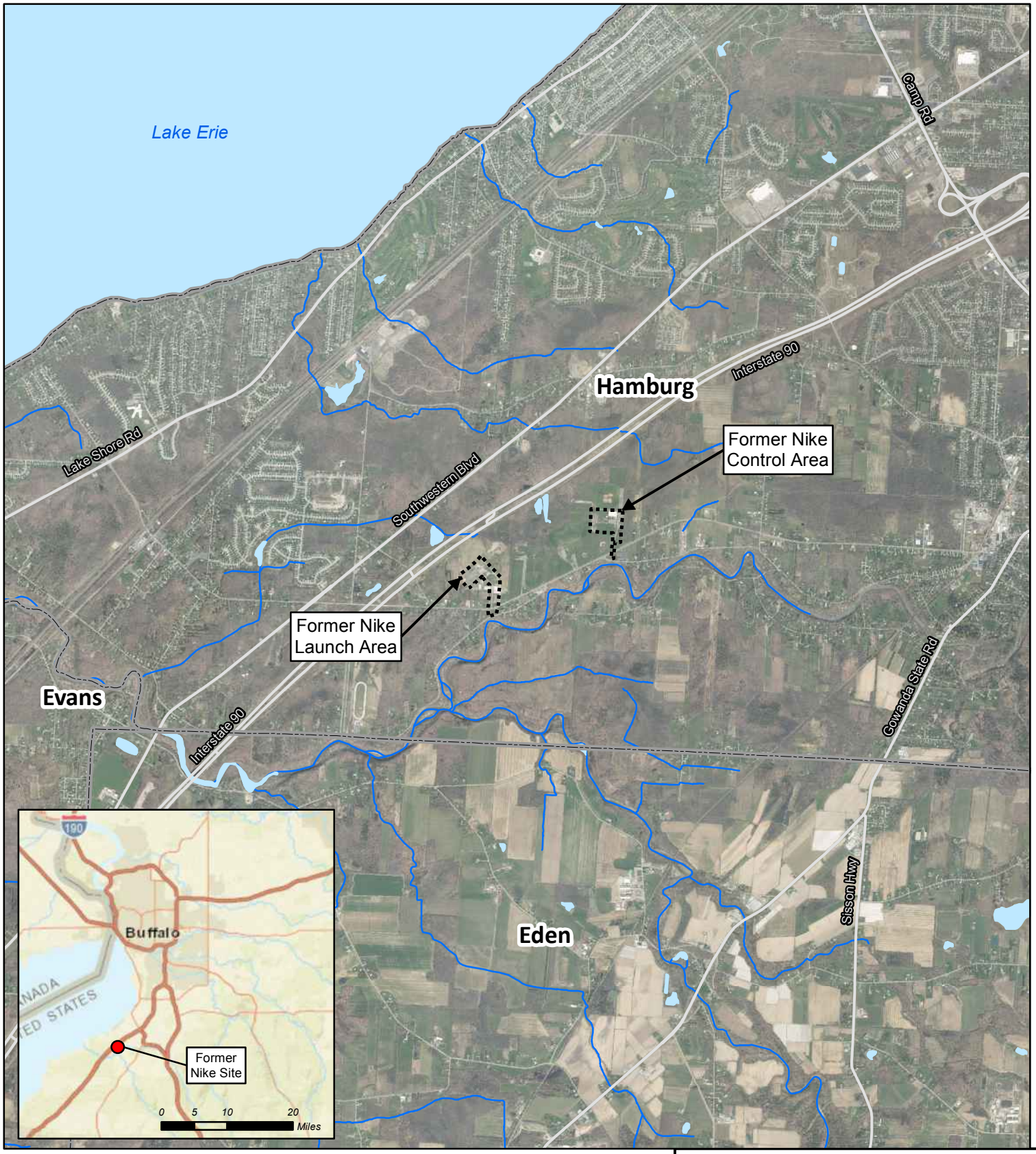
FIGURES



Former Nike Launch and Control Areas (Approx.)
Waterbody
Streams and Rivers
Major Roads
Town Boundaries

N
W E
S

0 0.5 1 2
Miles

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Source: GZA, 1999

FORMER NIKE BU -51/52 SITE

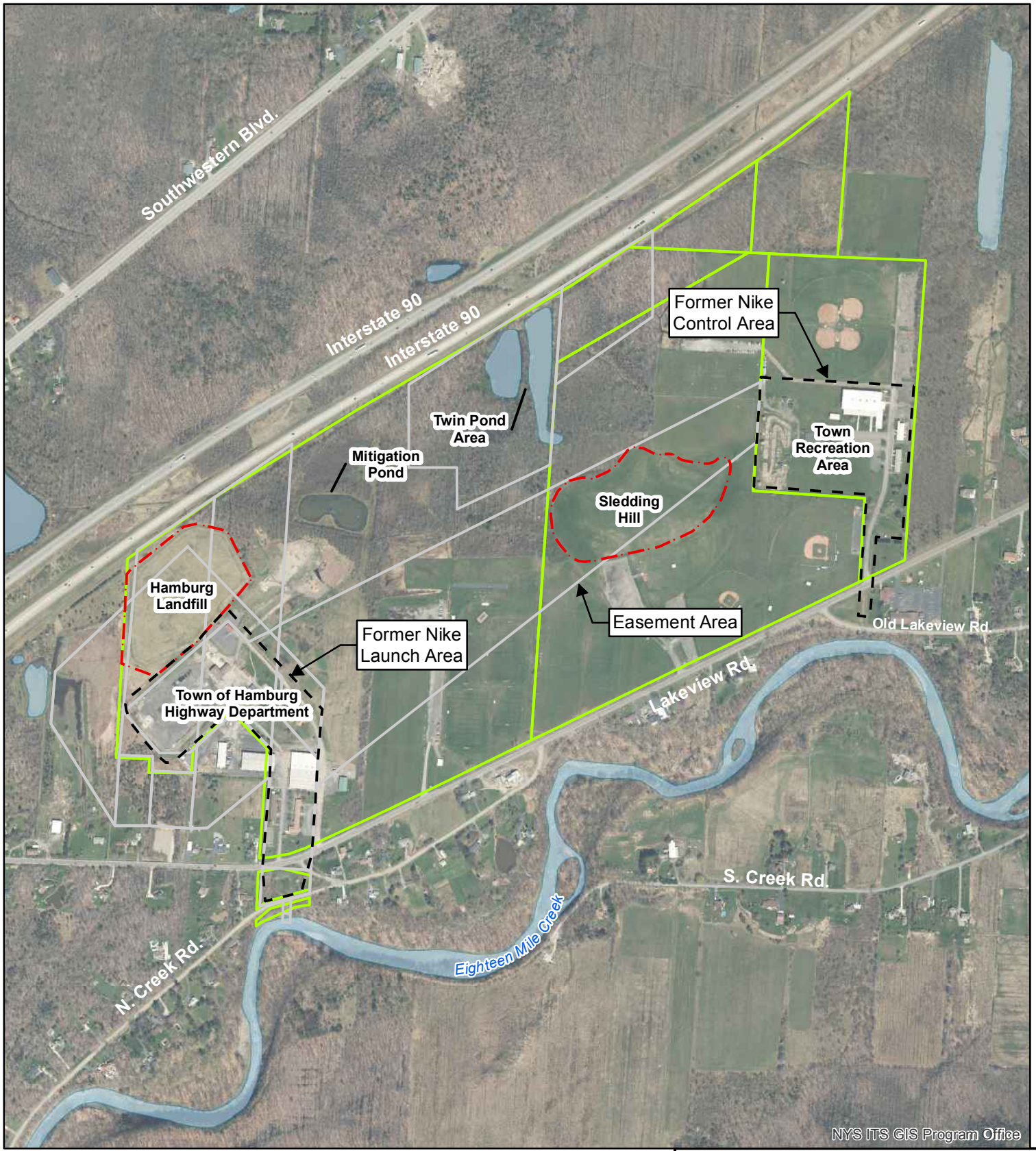
REGIONAL MAP

SERES ARCADIS a joint venture

September 2020

FIGURE
1

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 1_Regional Map.mxd



Former Nike Launch and Control Areas (Approx.)
Other Site Features
Perpetual Easements (Approx.)
Tax Parcel Boundary
Waterbody

0 200 400 800 Feet

N W E S

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Source: GZA, 1999

FORMER NIKE BU -51/52 SITE

SITE PLAN

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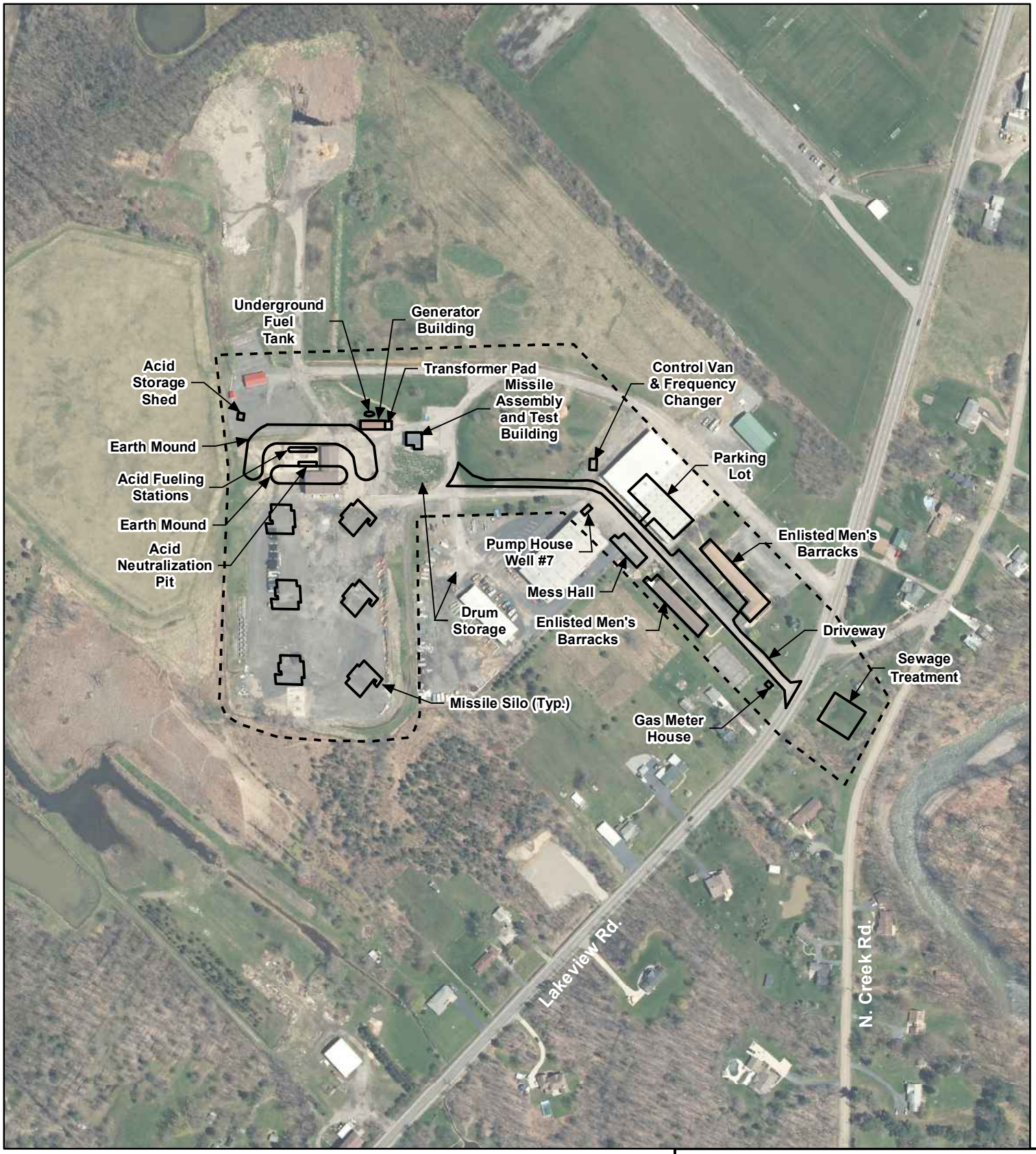
September 2020

FIGURE 2

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 2_Site Plan.mxd



Historical Site Features
Former Nike Launch Area (approx.)

Coordinate System: NAD 1983 UTM Zone 18N
Projection: Transverse Mercator
Source: GZA, 1999

0 200 400 800 Feet

FORMER NIKE BU -51/52 LAUNCH AREA

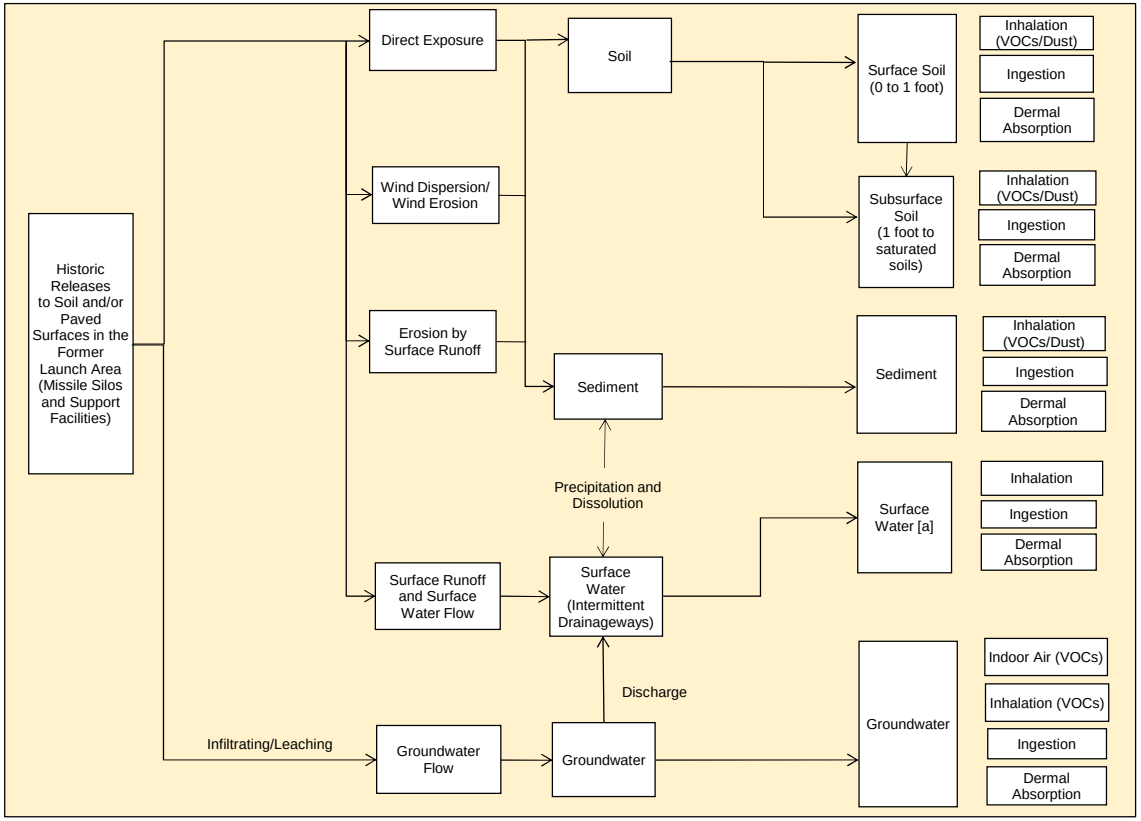
HISTORICAL SITE FEATURES

SERES ARCADIS a joint venture

September 2020

FIGURE 3

Document Path: Z:\GISProjects_ENV\Nike Project\Mxd\Figure 3_Historical Site Features.mxd



Current / Most Likely Future Scenario				Future Scenario	
On-site Commercial / Industrial Indoor Worker	On-site Outdoor Maintenance Worker	On-site Construction Worker	Off-site Nearby Resident	On-site Commercial / Industrial Indoor Worker	Off-site Nearby Resident
●	●	●	○	●	○
●	●	●	○	●	○
○	●	●	○	○	○
○	○	●	○	○	○
○	○	●	○	○	○
○	○	●	○	○	○
○	○	○	○	○	○
○	○	○	○	○	○
○	○	○	○	○	○
○	○	○	○	○	○
○	○	○	○	○	○
○	○	○	○	○	○
●	○	○	●	●	●
○	○	●	○	●	●
○	○	●	○	●	●
○	○	●	○	●	●

Notes:

○ Pathway is incomplete.
● Pathway is potentially complete.

The pathways depicted in this CSM are based on the current understanding of the Site and will be revised, as warranted, throughout the RI process. For example, the former sewage treatment plant may be an additional source area with treated water discharge conveyance to surface water/sediment as a potential exposure pathway.

[a] Represents potential exposure to Eighteen Mile Creek.

COPC = constituent of potential concern

CSM = conceptual site model

RI = remedial investigation

Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, New York

HUMAN HEALTH CONCEPTUAL SITE MODEL

ARCADIS Design & Consultancy for natural and built assets

FIGURE 4

Figure 5A: Nike BU 51/52 - Screening Level Ecological Risk Assessment (SLERA) Flow Chart

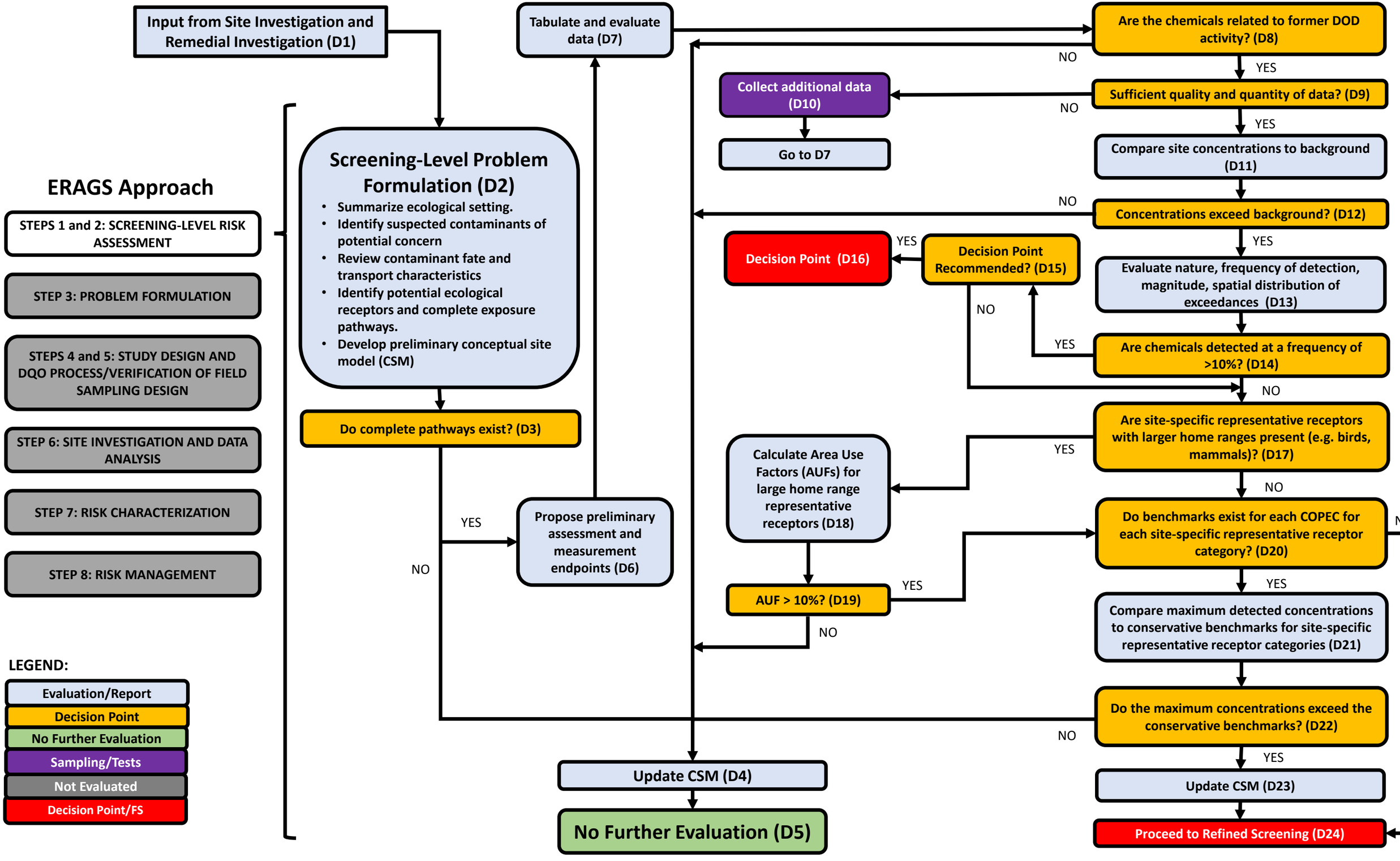


Figure 5B: Nike BU 51/52 Refined Screening Level Ecological Risk Assessment (RSLERA) Flow Chart

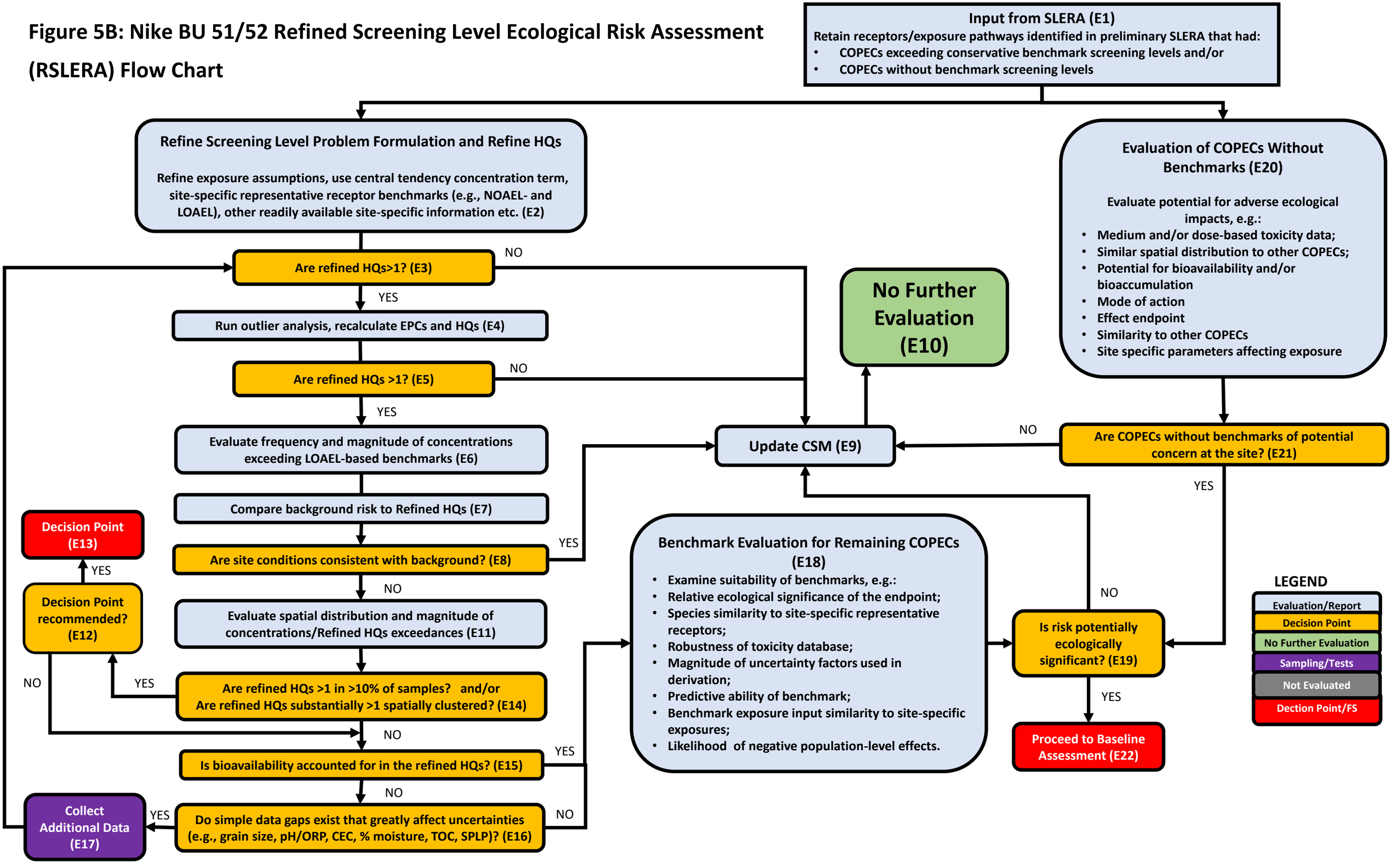


Figure 5C: Nike BU 51/52 Baseline Ecological Risk Assessment (BERA) Flow Chart

ERAGS Approach

STEPS 1 and 2: SCREENING-LEVEL RISK ASSESSMENT

STEP 3: PROBLEM FORMULATION

STEPS 4 and 5: STUDY DESIGN AND DQO PROCESS/VERIFICATION OF FIELD SAMPLING DESIGN

STEP 6: SITE INVESTIGATION AND DATA ANALYSIS

STEP 7: RISK CHARACTERIZATION

STEP 8: RISK MANAGEMENT

Input from RSLERA (F1)
Evaluate receptors and pathways for COPECs that indicate the potential for ecologically significant risk.

Perform a Baseline Problem Formulation, e.g.: evaluate toxicity and bioaccumulation; determine final assessment and measurement endpoints; complete exposure pathways; refine conceptual site model; and ask questions/form hypotheses to direct data collection, analyses, and risk evaluation (F2)

Develop sampling and analysis plan based upon completed exposure pathways (F3)

Surface Water (F4)

Data Collection Options
e.g.,
- Toxicity testing (survival, growth, reproduction)
- Bioaccumulation evaluation
- Tissue analysis (epibenthic, fish)
- Community assessment (epibenthic, fish)
- Fish health profile
- Fish growth and age metrics evaluation
- Biomarker assessment

Sediment (F5)

Data Collection Options
e.g.,
- Toxicity testing (survival, growth, reproduction)
- Bioaccumulation evaluation
- Tissue analysis (benthic, fish)
- Community assessment (benthic, fish)
- Toxicity Identification Evaluation (TIE)

Soil (F6)

Data Collection Options
e.g.,
- Toxicity testing (survival, growth, reproduction)
- Bioaccumulation evaluation
- Tissue analysis (plant, invertebrate, bird, mammal)
- Vegetative community analysis
- Assess populations (numbers, biomass)
- Dietary tissue analysis

Using the various lines of evidence, evaluate if a risk exists for each Assessment and/or Measurement Endpoint (F7)

Is risk potentially ecologically significant? (F8)

NO

YES

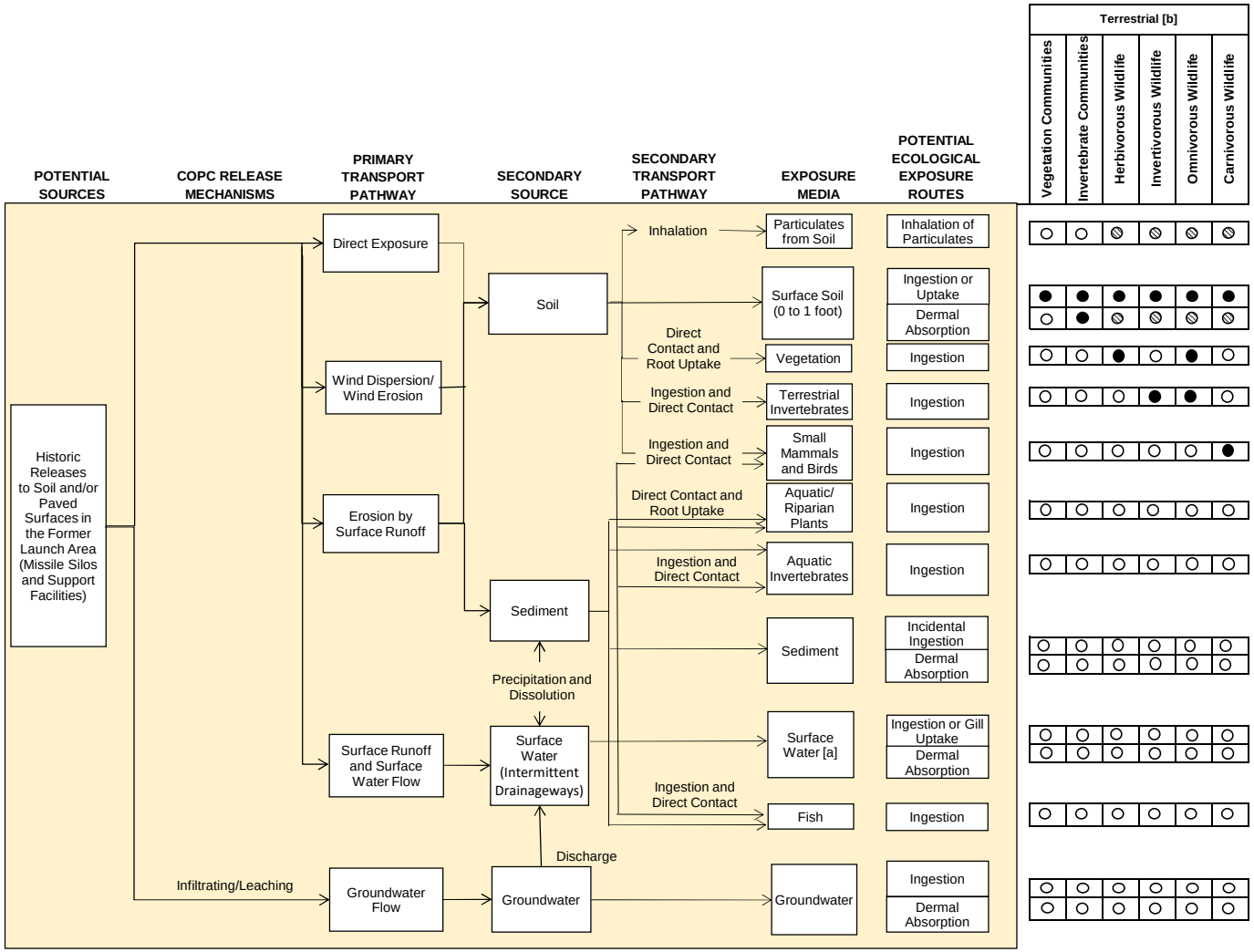
No Further Evaluation (F10)

Update CSM (F9)

Perform Feasibility Study for Retained Receptors (F11)

LEGEND:

- Evaluation/Report
- Decision Point
- No Further Evaluation
- Sampling/Tests
- Not Evaluated
- Decision Point/FS



Notes:

○	Pathway is considered incomplete
●	Pathway is considered potentially complete (quantitatively evaluated)
⦸	Pathway is considered potentially complete (qualitatively evaluated; methodology not available or considered insignificant exposure)

The pathways depicted in this CSM are based on the current understanding of the Site and will be revised, as warranted, throughout the RI process. For example, the former sewage treatment plant may be an additional source area with treated water discharge conveyance to surface water/sediment as a potential exposure pathway.

[a] Represents potential exposure to Eighteen Mile Creek and/or the Twin Ponds Area, which are offsite and unlikely to be affected by site conditions.

[b] Representative receptors are discussed further in Appendix C and include the following: meadow vole (herbivorous mammal), mourning dove (herbivorous bird), American robin (invertivorous/omnivorous bird), short-tailed shrew (invertivorous/omnivorous mammal), red-tailed hawk (carnivorous bird), and red fox (carnivorous mammal).

COPC = constituent of potential concern; CSM = conceptual site model; RI = remedial investigation

Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, New York

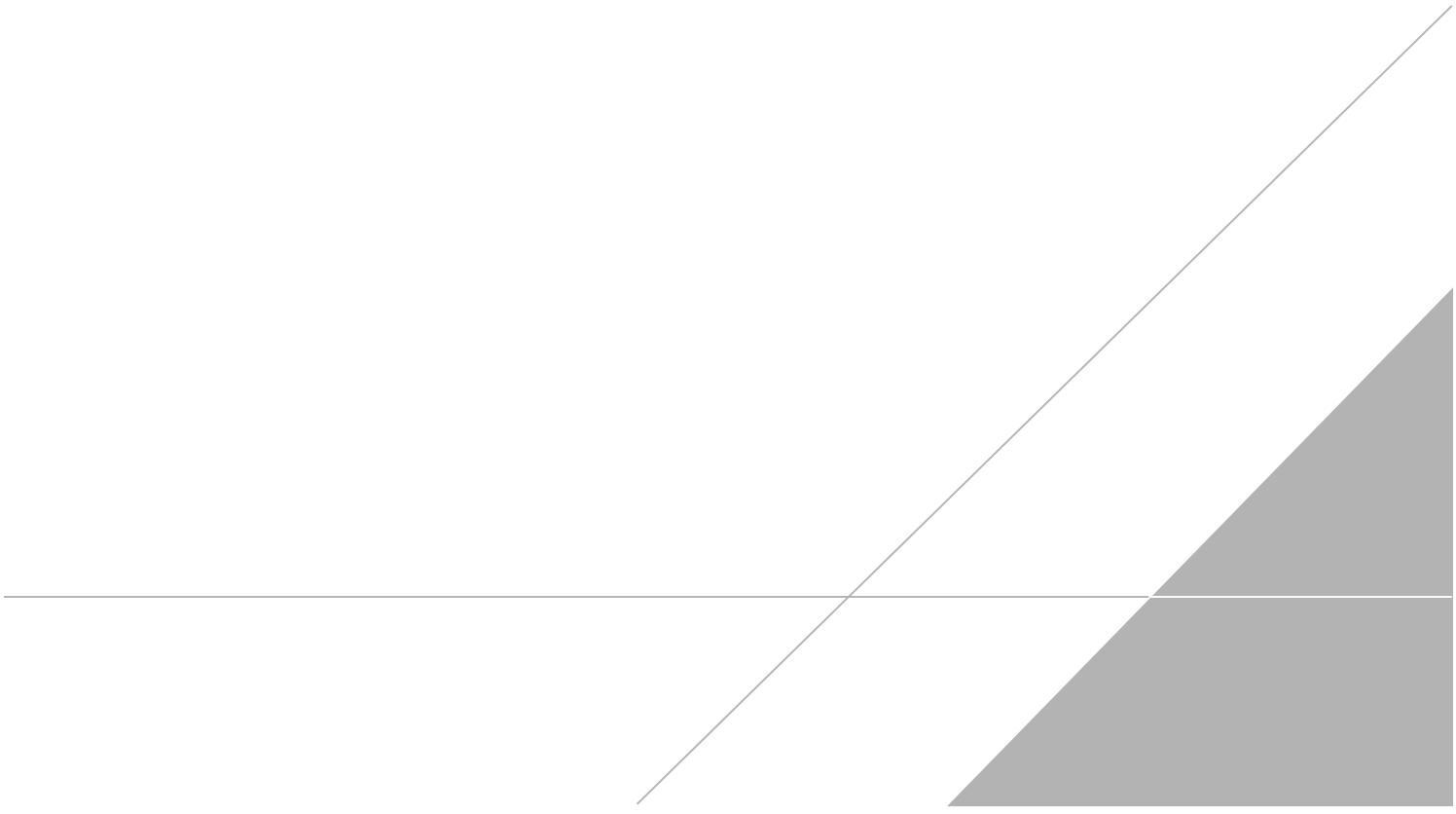
PRELIMINARY ECOLOGICAL CONCEPTUAL SITE MODEL

ARCADIS Design & Consultancy for natural and built assets

FIGURE 6

ATTACHMENT A

Risk Assessment Guidance for Superfund (RAGS) Part D Tables



RAGS Part D Planning Table 1
Selection of Human Exposure Pathways
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe	Medium	Exposure Medium	Exposure Point	Human Receptor Population	Receptor Age	Exposure Route	Type of Analysis	Rationale for Selection or Exclusion of Exposure Pathway
Current / Most Likely Future [a]	On-site Soil	Surface Soil (0-1 ft bgs)	Former Launch Area	Indoor Commercial / Industrial Worker	Adult	Incidental Ingestion	Quantitative	The former Launch Area at the Nike Antiaircraft Missile Battery BU-51/52 (Site) is currently used by the Town of Hamburg's Highway Department for storage, office space, and maintenance. Surface soil (0-1 ft bgs) could be contacted by commercial/industrial workers (indoor workers) at the Site.
						Dermal Contact	None [d]	
						Inhalation (VOCs and Dust)	Quantitative	
				Outdoor Maintenance Worker	Adult	Incidental Ingestion	Quantitative	Surface soil (0-1 ft bgs) could be contacted by maintenance workers who primarily work outdoors at the Site (e.g., Highway Department workers).
						Dermal Contact	Quantitative	
						Inhalation (VOCs and Dust)	Quantitative	
		All Soil	Former Launch Area	Construction / Utility Worker	Adult	Incidental Ingestion	Quantitative	In the event of intrusive work at the Site, surface and subsurface soils could be contacted by construction or utility workers.
						Dermal Contact	Quantitative	
						Inhalation (VOCs and Dust)	Quantitative	
	Groundwater	On-site Shallow Groundwater	Tapwater	Indoor Commercial / Industrial Worker	Adult	Ingestion	None	Potable water is supplied to the Site by the Erie County Water Authority; therefore, this exposure pathway is currently incomplete.
						Dermal Contact	None	
						Inhalation (VOCs)	None	
			Indoor Air of on-site Buildings	Indoor Commercial / Industrial Worker	Adult	Inhalation (VOCs)	Quantitative	Commercial/industrial workers (indoor workers) could be exposed to VOCs, if present in underlying groundwater, that migrate from shallow groundwater to indoor air of on-site buildings.
			Excavation / Trench for Utility Work	Construction / Utility Worker	Adult	Incidental Ingestion	Quantitative	Groundwater at the Site is approximately 4 to 7 ft bgs in the overburden and flows south toward Eighteen Mile Creek. Given the shallow depth to groundwater, construction or utility workers may contact shallow groundwater that infiltrates the bottom of an excavation.
						Dermal Contact	Quantitative	
						Inhalation (VOCs)	Quantitative	
		Off-site Shallow Groundwater	Tapwater in Nearby Residence	Resident	Adult	Ingestion	None	Potable water is supplied to nearby residences by the Erie County Water Authority; therefore, this exposure pathway is currently incomplete.
						Dermal Contact	None	
						Inhalation (VOCs)	None	
					Child [c]	Ingestion	None	
						Dermal Contact	None	
						Inhalation (VOCs)	None	
			Indoor Air of Nearby Residence	Resident	Adult	Inhalation (VOCs)	Quantitative	Nearby residents could be exposed to VOCs, if present in underlying groundwater, that migrate from shallow groundwater to indoor air of off-site residences.
					Child	Inhalation (VOCs)	Quantitative	

RAGS Part D Planning Table 1
Selection of Human Exposure Pathways
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe	Medium	Exposure Medium	Exposure Point	Human Receptor Population	Receptor Age	Exposure Route	Type of Analysis	Rationale for Selection or Exclusion of Exposure Pathway
Potential Future [b]	On-site Soil	All Soil	Former Launch Area	Hypothetical Future Resident	Adult	Incidental Ingestion	None	Residential use is not considered a reasonable future use of the Site based on the current land use and land ownership, and proximity to the Hamburg Landfill.
						Dermal contact	None	
						Inhalation (Dust)	None	
					Child	Incidental Ingestion	None	
						Dermal contact	None	
						Inhalation (Dust)	None	
	Groundwater	On-site Shallow Groundwater	Tapwater in on-site Buildings	Indoor Commercial / Industrial Worker	Adult	Ingestion	Quantitative	Although this exposure pathway is currently incomplete, there are no restrictions that would limit future use of the surficial aquifer as a drinking water source.
						Dermal Contact	Quantitative	
						Inhalation (VOCs)	Quantitative	
			Tapwater in Hypothetical Future on-site Residence	Hypothetical Future Resident	Adult	Ingestion	None	Residential use is not considered a reasonable future use of the Site based on the current land use and land ownership, and proximity to the Hamburg Landfill.
						Dermal Contact	None	
						Inhalation (VOCs)	None	
					Child	Ingestion	None	
						Dermal Contact	None	
						Inhalation (VOCs)	None	
			Indoor Air of Hypothetical Future on-site Residence	Hypothetical Future Resident	Adult	Inhalation (VOCs)	None	Residential use is not considered a reasonable future use of the Site based on the current land use and land ownership, and proximity to the Hamburg Landfill.
					Child	Inhalation (VOCs)	None	
		Off-site Shallow Groundwater	Tapwater in Nearby Residence	Resident	Adult	Ingestion	Quantitative	Although this exposure pathway is currently incomplete, there are no restrictions that would limit future use of the surficial aquifer as a drinking water source.
						Dermal Contact	Quantitative	
						Inhalation (VOCs)	Quantitative	
					Child	Ingestion	Quantitative	
						Dermal Contact	Quantitative	
						Inhalation (VOCs)	Quantitative	

Notes:
[a] The Current / Most Likely Future scenario considers the current and most likely future land use of the former Launch Area as an industrial site owned by the Town of Hamburg and used by the Highway Department for storage, office space, and maintenance.
[b] The Potential Future scenario considers an additional hypothetical exposure scenario in which the surficial aquifer is used as a drinking water source.
[c] A child is defined as 0 to 6 years of age.
[d] Per current USEPA guidance (USEPA 2019c), the dermal exposure route is not evaluated for indoor commercial/industrial workers.

ft bgs - feet below ground surface.
RAGS - Risk Assessment Guidance for Superfund.
VOCs - volatile organic constituents.
USEPA - United States Environmental Protection Agency.

References:
Town of Hamburg, 2018. Zoning Map. Town of Hamburg, Erie County. https://www.townofhamburgny.com/wp-content/uploads/2018/07/Hamburg-Zoning-with-LWRP-Boundary.pdf
USEPA. 2019c. User's Guide. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-users-guide.

RAGS Part D Table 4.1
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: On-site Soil
Exposure Medium: Surface Soil (0-1 ft bgs)

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	Indoor Commercial / Industrial Worker	Adult	Former Launch Area	EPCs	Exposure point concentration, soil	Chemical-specific	mg/kg	TBD	Chronic Daily Intake (mg/kg-day) =
				IRS	Soil ingestion rate	50	mg/day	USEPA 2014	EPCs × IRS × RBA × EF × ED × CF × 1/BW × 1/AT
				RBA	Relative bioavailability	Chemical-specific	unitless	USEPA 2019c	
				EF	Exposure frequency	250	days/year	USEPA 2014	
				ED	Exposure duration	25	years	USEPA 2014	
				CF	Conversion factor	1E-06	kg/mg	--	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	9,125	days	USEPA 1989	
Inhalation	Indoor Commercial / Industrial Worker	Adult	Former Launch Area	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	8	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	250	days/year	USEPA 2014	where:
				ED	Exposure duration	25	years	USEPA 2014	EPCa = EPCs × 1/PEF, for particulates
				PEF	Particulate emission factor due to wind under passive conditions	1.36E+09	m^3/kg	USEPA 2002	or
				VFs	Volatilization factor, soil	Chemical-specific	m^3/kg	USEPA 2002	EPCa = EPCs × 1/VFs, for volatile constituents
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	219,000	hours	USEPA 2009	

Notes:
ft bgs - feet below ground surface.
USEPA - United States Environmental Protection Agency.

References:
USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS): Volume 1, Human Health Evaluation Manual, Part A. EPA/5401-89/002. Office of Emergency and Remedial Response, Washington, DC. December.
USEPA. 2002. Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites. OSWER 9355.4-24. Office of Solid Waste and Emergency Response, Washington, DC. December.
USEPA. 2004. Risk Assessment Guidance for Superfund. Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment). Final. EPA/540/R/99/005. Office of Superfund Remediation and Technology Innovation, Washington, DC. July.
USEPA. 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment). Final. EPA-540-R-070-002. Office of Superfund Remediation and Technology Innovation, Washington, DC. January.
USEPA. 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. OSWER Directive 9200.1-120. Office of Solid Waste and Emergency Response, Washington, DC. February.
USEPA. 2019c. User's Guide. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-users-guide.

RAGS Part D Table 4.2
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: On-site Soil
Exposure Medium: Surface Soil (0-1 ft bgs)

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	Outdoor Maintenance Worker	Adult	Former Launch Area	EPCs	Exposure point concentration, soil	Chemical-specific	mg/kg	TBD	Chronic Daily Intake (mg/kg-day) =
				IRS	Soil ingestion rate	100	mg/day	USEPA 2014	EPCs × IRS × RBA × EF × ED × CF × 1/BW × 1/AT
				RBA	Relative bioavailability	Chemical-specific	unitless	USEPA 2019c	
				EF	Exposure frequency	225	days/year	USEPA 2014	
				ED	Exposure duration	25	years	USEPA 2014	
				CF	Conversion factor	1E-06	kg/mg	--	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	9,125	days	USEPA 1989	
Dermal	Outdoor Maintenance Worker	Adult	Former Launch Area	EPCs	Exposure point concentration, soil	Chemical-specific	mg/kg	TBD	Dermally Absorbed Dose (mg/kg-day) =
				DA_{event}	Absorbed dose per event	Chemical-specific	mg/cm^2-event	USEPA 2004	DA_{event} × EF × ED × EV × SA × 1/BW × 1/AT
				EF	Exposure frequency	225	days/year	USEPA 2014	where:
				ED	Exposure duration	25	years	USEPA 2014	Absorbed Dose per Event (DA_{event}) (mg/cm^2-event) =
				EV	Event frequency	1	events/day	Professional judgment	EPCs × CF × AF × ABS-d
				SA	Skin surface area available for contact	3,527	cm^2	USEPA 2014	
				BW	Body weight	80	kg	USEPA 2014	
				CF	Conversion factor	1E-06	kg/mg	--	
				AF	Soil-to-skin adherence factor	0.12	mg/cm^2-event	USEPA 2014	
				ABS-d	Dermal absorption fraction	Chemical-specific	unitless	USEPA 2004	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	9,125	days	USEPA 1989	
Inhalation	Outdoor Maintenance Worker	Adult	Former Launch Area	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	8	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	225	days/year	USEPA 2014	where:
				ED	Exposure duration	25	years	USEPA 2014	EPCa = EPCs × 1/PEF, for particulates
				PEF	Particulate emission factor due to wind under passive conditions	1.36E+09	m^3/kg	USEPA 2002	or
				VFs	Volatilization factor, soil	Chemical-specific	m^3/kg	USEPA 2002	EPCa = EPCs × 1/VFs, for volatile constituents
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	219,000	hours	USEPA 2009	

Notes:
ft bgs - feet below ground surface.
USEPA - United States Environmental Protection Agency.

References:

USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS): Volume 1, Human Health Evaluation Manual, Part A. EPA/5401-89/002. Office of Emergency and Remedial Response, Washington, DC. December.
USEPA. 2002. Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites. OSWER 9355.4-24. Office of Solid Waste and Emergency Response, Washington, DC. December.
USEPA. 2004. Risk Assessment Guidance for Superfund. Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment). Final. EPA/540/R/99/005. Office of Superfund Remediation and Technology Innovation, Washington, DC. July.
USEPA. 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment). Final. EPA-540-R-070-002. Office of Superfund Remediation and Technology Innovation, Washington, DC. January.
USEPA. 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. OSWER Directive 9200.1-120. Office of Solid Waste and Emergency Response, Washington, DC. February.
USEPA. 2019c. User's Guide. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-users-guide.

RAGS Part D Table 4.3
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: On-site Soil
Exposure Medium: All Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	Construction / Utility Worker	Adult	Former Launch Area	EPCs	Exposure point concentration, soil	Chemical-specific	mg/kg	TBD	Chronic Daily Intake (mg/kg-day) =
				IRS	Soil ingestion rate	330	mg/day	USEPA 2014	EPCs × IRS × RBA × EF × ED × CF × 1/BW × 1/AT
				RBA	Relative bioavailability	Chemical-specific	unitless	USEPA 2019c	
				EF	Exposure frequency	125	days/year	Professional judgment; Assumes 5 days per week for 25 weeks (approximately 6 months)	
				ED	Exposure duration	1	years	Professional judgment	
				CF	Conversion factor	1E-06	kg/mg	--	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	365	days	USEPA 1989	
Dermal	Construction / Utility Worker	Adult	Former Launch Area	EPCs	Exposure point concentration, soil	Chemical-specific	mg/kg	TBD	Dermally Absorbed Dose (mg/kg-day) =
				DA_{event}	Absorbed dose per event	Chemical-specific	mg/cm^2-event	USEPA 2004	DA_{event} × EF × ED × EV × SA × 1/BW × 1/AT
				EF	Exposure frequency	125	days/year	Professional judgment; Assumes 5 days per week for 25 weeks (approximately 6 months)	where:
				ED	Exposure duration	1	years	Professional judgment	Absorbed Dose per Event (DA_{event}) (mg/cm^2-event) =
				EV	Event frequency	1	events/day	Professional judgment	EPCs × CF × AF × ABS-d
				SA	Skin surface area available for contact	3,527	cm^2	USEPA 2014	
				BW	Body weight	80	kg	USEPA 2014	
				CF	Conversion factor	1E-06	kg/mg	--	
				AF	Soil-to-skin adherence factor	0.3	mg/cm^2-event	USEPA 2002	
				ABS-d	Dermal absorption fraction	Chemical-specific	unitless	USEPA 2004	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	365	days	USEPA 1989	
Inhalation	Construction / Utility Worker	Adult	Former Launch Area	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	8	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	125	days/year	Professional judgment; Assumes 5 days per week for 25 weeks (approximately 6 months)	where:
				ED	Exposure duration	1	years	Professional judgment	EPCa = EPCs × 1/PEF, for particulates
				PEF	Particulate emission factor for a construction scenario	Site-specific	m^3/kg	USEPA 2002	or
				VFs	Volatilization factor, soil	Chemical-specific	m^3/kg	USEPA 2002	EPCa = EPCs × 1/VFs, for volatile constituents
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	8,760	hours	USEPA 2009	

Notes:
USEPA - United States Environmental Protection Agency.

References:

USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS): Volume 1, Human Health Evaluation Manual, Part A. EPA/5401-89/002. Office of Emergency and Remedial Response, Washington, DC. December.
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RAGS Part D Table 4.4
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: On-site Shallow Groundwater
Exposure Medium: Indoor Air (Vapor Intrusion)

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation	Indoor Commercial / Industrial Worker	Adult	On-site Building	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	8	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	250	days/year	USEPA 2014	
				ED	Exposure duration	25	years	USEPA 2014	where, for volatile constituents:
				EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	EPCa = EPCgw × AFgw × CF × H'
				AFgw	Attenuation factor, generic	0.001	unitless	USEPA 2019a	
				CF	Conversion factor	1,000	L/m^3	--	
				H'	Henry's Law constant	Chemical-specific	unitless	USEPA 2019b	
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	219,000	hours	USEPA 2009	

Notes:
USEPA - United States Environmental Protection Agency.

References:
USEPA. 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment). Final. EPA-540-R-070-002. Office of Superfund Remediation and Technology Innovation, Washington, DC. January.
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USEPA. 2019a. Vapor Intrusion Screening Level Calculator. Available online: https://www.epa.gov/vaporintrusion/vapor-intrusion-screening-level-calculator.
USEPA. 2019b. Chemical Specific Parameters Table. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables.

RAGS Part D Table 4.5
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: On-site Shallow Groundwater
Exposure Media: Groundwater and Trench Air

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	Construction / Utility Worker	Adult	Utility Trench	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Chronic Daily Intake (CDI) (mg/kg-day)=
				IRW	Ingestion rate	0.02	liters/day	VDEQ 2019	EPCgw × IRW × EF × ED × 1/BW × 1/AT
				EF	Exposure frequency	125	days/year	Professional judgment; Assumes 5 days per week for 25 weeks (approximately 6 months)	
				ED	Exposure duration	1	years	Professional judgment	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	365	days	USEPA 1989	
Dermal	Construction / Utility Worker	Adult	Utility Trench	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Dermally Absorbed Dose (DAD) (mg/kg-day) =
				DA_{event}	Absorbed dose per event	Chemical-specific	mg/cm^2-event	USEPA 2004	DA_{event} × EV × ED × EF × SA × 1/BW × 1/AT
				EV	Event frequency	1	events/day	Professional judgment	
				ED	Exposure duration	1	years	Professional judgment	Where for inorganic chemicals:
				EF	Exposure frequency	125	days/year	Professional judgment; Assumes 5 days per week for 25 weeks (approximately 6 months)	Absorbed Dose per Event (DA_{event}) (mg/cm^2-event) = DA_{event} = Kp × EPCgw × t-event × CF
				SA	Skin surface area available for contact	3,527	cm^2	USEPA 2014	And where for organic chemicals:
				Kp	Permeability coefficient	Chemical-specific	cm/hour	USEPA 2019b	
				t-event	Event duration	8	hours/event	USEPA 2014	If t-event < t*, then: DA_{event} = 2FA × Kp × EPCgw × CF × SQRT{(6 × tau-event × t-event)/pi}
				CF	Volumetric conversion factor for water	0.001	L/cm^3	--	
				t*	Time to reach steady-state = 2.4×tau-event	Chemical-specific	hours	USEPA 2019b	If t-event > t*, then: DA_{event} = FA × Kp × EPCgw × CF × {(t-event/(1 + B)) + 2 × tau-event × ((1 + (3 × B) + (3 × B^2))/(1 + B)^2)}
				FA	Fraction absorbed	Chemical-specific	unitless	USEPA 2004	
				tau-event	Lag time per event	Chemical-specific	hours/event	USEPA 2019b	
				B	Ratio of permeability coefficient of a chemical through the stratum corneum relative to its permeability coefficient across the viable epidermis	Chemical-specific	unitless	USEPA 2019b	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	365	days	USEPA 1989	

RAGS Part D Table 4.5
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: On-site Shallow Groundwater
Exposure Media: Groundwater and Trench Air

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation	Construction / Utility Worker	Adult	Trench Air	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	8	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	125	days/year	Professional judgment; Assumes 5 days per week for 25 weeks (approximately 6 months)	
				ED	Exposure duration	1	years	Professional judgment	where, for volatile constituents:
				VF-trench	Volatilization factor for trench air	Chemical-specific	L/m^3	VDEQ 2019	EPCa = EPCgw × VF-trench
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	8,760	hours	USEPA 2009	

Notes:
USEPA - United States Environmental Protection Agency.
VDEQ - Virginia Department of Environmental Quality.

References:

USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS): Volume 1, Human Health Evaluation Manual, Part A. EPA/5401-89/002. Office of Emergency and Remedial Response, Washington, DC. December.
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VDEQ. 2019. Voluntary Remediation Program Risk Assessment Guidance. Accessed online: https://www.deq.virginia.gov/Programs/LandProtectionRevitalization/RemediationProgram/VoluntaryRemediationProgram/VRP-RiskAssessmentGuidance/Guidance.aspx.

RAGS Part D Table 4.6
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Current/Future
Medium: Shallow Groundwater
Exposure Medium: Indoor Air (Vapor Intrusion)

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation	Off-site Resident	Adult	Nearby Residence	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	24	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	350	days/year	USEPA 2014	
				ED	Exposure duration, cancer	20	years	USEPA 2014	where, for volatile constituents:
				EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	EPCa = EPCgw × AFgw × CF × H'
				AFgw	Attenuation factor, generic	0.001	unitless	USEPA 2019a	
				CF	Conversion factor	1,000	L/m^3	--	
				H'	Henry's Law constant	Chemical-specific	unitless	USEPA 2019b	
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	175,200	hours	USEPA 2009	
		Child	Nearby Residence	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	24	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	350	days/year	USEPA 2014	
				ED	Exposure duration, cancer	6	years	USEPA 2014	where, for volatile constituents:
				EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	EPCa = EPCgw × AFgw × CF × H'
				AFgw	Attenuation factor, generic	0.001	unitless	USEPA 2019a	
				CF	Conversion factor	1,000	L/m^3	--	
				H'	Henry's Law constant	Chemical-specific	unitless	USEPA 2019b	
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	52,560	hours	USEPA 2009	

Notes:
USEPA - United States Environmental Protection Agency.

References:
USEPA. 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment). Final. EPA-540-R-070-002. Office of Superfund Remediation and Technology Innovation, Washington, DC. January.
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USEPA. 2019b. Chemical Specific Parameters Table. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables.

RAGS Part D Table 4.7
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Hypothetical Future
Medium: On-site Groundwater
Exposure Medium: Potable Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	Indoor Commercial / Industrial Worker	Adult	Workplace Tapwater	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Chronic Daily Intake (CDI) (mg/kg-day)= EPCgw × IRW × EF × ED × 1/BW × 1/AT
				IRW	Ingestion rate	1.3	liters/day	Assumes half of the daily intake for an adult USEPA 2014; USEPA 1991	
				EF	Exposure frequency	250	days/year	USEPA 2014	
				ED	Exposure duration	25	years	USEPA 2014	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	9,125	days	USEPA 1989	
Dermal	Indoor Commercial / Industrial Worker	Adult	Workplace Tapwater	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Dermally Absorbed Dose (DAD) (mg/kg-day) = DA_{event} × EV × ED × EF × SA × 1/BW × 1/AT
				DA_{event}	Absorbed dose per event	Chemical-specific	mg/cm^2-event	USEPA 2004	
				EV	Event frequency	5	events/day	Professional judgment; Assumes dermal exposure during hand washing	
				ED	Exposure duration	25	years	USEPA 2014	Where for inorganic chemicals:
				EF	Exposure frequency	250	days/year	USEPA 2014	Absorbed Dose per Event (DA_{event}) (mg/cm^2-event) = DA_{event} = Kp × EPCgw × t-event × CF
				SA	Skin surface area available for contact	904	cm^2	Average of 50th percentile values for hands for adult males and females (USEPA 2004; Exhibit C-1).	
				Kp	Permeability coefficient	Chemical-specific	cm/hour	USEPA 2019b	And where for organic chemicals:
				t-event	Event duration	0.03	hours/event	Assumes 2 minutes per hand washing event	If t-event < t*, then: DA_{event} = 2FA × Kp × EPCgw × CF × SQRT{(6 × tau-event × t-event)/pi}
				CF	Volumetric conversion factor for water	0.001	L/cm^3	--	
				t*	Time to reach steady-state = 2.4×tau-event	Chemical-specific	hours	USEPA 2019b	If t-event > t*, then: DA_{event} = FA × Kp × EPCgw × CF × {(t-event/(1 + B)) + 2 × tau-event × ((1 + (3 × B) + (3 × B^2))/(1 + B)^2)}
				FA	Fraction absorbed	Chemical-specific	unitless	USEPA 2004	
				tau-event	Lag time per event	Chemical-specific	hours/event	USEPA 2019b	
				B	Ratio of permeability coefficient of a chemical through the stratum corneum relative to its permeability coefficient across the viable epidermis	Chemical-specific	unitless	USEPA 2019b	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	9,125	days	USEPA 1989	

RAGS Part D Table 4.7
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Hypothetical Future
Medium: On-site Groundwater
Exposure Medium: Potable Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation	Indoor Commercial / Industrial Worker	Adult	Workplace Indoor Air	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	8	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	250	days/year	USEPA 2014	
				ED	Exposure duration	25	years	USEPA 2014	where, for volatile constituents:
				VF-tap	Volatilization factor for tapwater	0.5	L/m^3	USEPA 2019c	EPCa = EPCgw × VF-tap
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	219,000	hours	USEPA 2009	

Notes:
USEPA - United States Environmental Protection Agency.
VDEQ - Virginia Department of Environmental Quality.

References:

USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS): Volume 1, Human Health Evaluation Manual, Part A. EPA/5401-89/002. Office of Emergency and Remedial Response, Washington, DC. December.
USEPA. 1991. "Standard Default Exposure Factors". Risk Assessment Guidance for Superfund, Volume I: Human Health Evaluation Manual Supplemental Guidance. Interim Final. OSWER Directive 9285.6-03. March 25, 1991.
USEPA. 2004. Risk Assessment Guidance for Superfund. Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment). Final. EPA/540/R/99/005. Office of Superfund Remediation and Technology Innovation, Washington, DC. July.
USEPA. 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment). Final. EPA-540-R-070-002. Office of Superfund Remediation and Technology Innovation, Washington, DC. January.
USEPA. 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. OSWER Directive 9200.1-120. Office of Solid Waste and Emergency Response, Washington, DC. February.
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USEPA. 2019c. User's Guide. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-users-guide.
VDEQ. 2019. Voluntary Remediation Program Risk Assessment Guidance. Accessed online: https://www.deq.virginia.gov/Programs/LandProtectionRevitalization/RemediationProgram/VoluntaryRemediationProgram/VRP-RiskAssessmentGuidance/Guidance.aspx.

RAGS Part D Table 4.8
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Hypothetical Future
Medium: Off-site Groundwater
Exposure Medium: Potable Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	Off-site Resident	Adult	Nearby Residence	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Chronic Daily Intake (CDI) (mg/kg-day)=
				IRW	Ingestion rate	2.5	liters/day	USEPA 2014	EPCgw × IRW × EF × ED × 1/BW × 1/AT
				EF	Exposure frequency	350	days/year	USEPA 2014	
				ED	Exposure duration	20	years	USEPA 2014	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	7,300	days	USEPA 1989	
		Child	Nearby Residence	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Chronic Daily Intake (CDI) (mg/kg-day)=
				IRW	Ingestion rate	0.78	liters/day	USEPA 2014	EPCgw × IRW × EF × ED × 1/BW × 1/AT
				EF	Exposure frequency	350	days/year	USEPA 2014	
				ED	Exposure duration	6	years	USEPA 2014	
				BW	Body weight	15	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	2,190	days	USEPA 1989	
Dermal	Off-site Resident	Adult	Nearby Residence	EPCgw	Exposure point concentration, groundwater	Chemical-specific	mg/L	TBD	Dermally Absorbed Dose (DAD) (mg/kg-day) =
				DA_{event}	Absorbed dose per event	Chemical-specific	mg/cm^2-event	USEPA 2004	DA_{event} × EV × ED × EF × SA × 1/BW × 1/AT
				EV	Event frequency	1	events/day	USEPA 2014	
				ED	Exposure duration	20	years	USEPA 2014	Where for inorganic chemicals:
				EF	Exposure frequency	350	days/year	USEPA 2014	Absorbed Dose per Event (DA_{event}) (mg/cm^2-event) = DA_{event} = Kp × EPCgw × t-event × CF
				SA	Skin surface area available for contact	19,652	cm^2	USEPA 2014	
				Kp	Permeability coefficient	Chemical-specific	cm/hour	USEPA 2019b	And where for organic chemicals:
				t-event	Event duration	0.71	hours/event	USEPA 2014	If t-event < t*, then: DA_{event} = 2FA × Kp × EPCgw × CF × SQRT{(6 × tau-event × t-event)/pi}
				CF	Volumetric conversion factor for water	0.001	L/cm^3	--	
				t*	Time to reach steady-state = 2.4×tau-event	Chemical-specific	hours	USEPA 2019b	If t-event > t*, then: DA_{event} = FA × Kp × EPCgw × CF × {(t-event/(1 + B)) + 2 × tau-event × ((1 + (3 × B) + (3 × B^2))/(1 + B)^2)}
				FA	Fraction absorbed	Chemical-specific	unitless	USEPA 2004	
				tau-event	Lag time per event	Chemical-specific	hours/event	USEPA 2019b	
				B	Ratio of permeability coefficient of a chemical through the stratum corneum relative to its permeability coefficient across the viable epidermis	Chemical-specific	unitless	USEPA 2019b	
				BW	Body weight	80	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	7,300	days	USEPA 1989	

RAGS Part D Table 4.8
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Hypothetical Future
Medium: Off-site Groundwater
Exposure Medium: Potable Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Dermal	Off-site Resident	Child	Nearby Residence	EPCgw	Exposure duration	Chemical-specific	mg/L	TBD	Dermally Absorbed Dose (DAD) (mg/kg-day) =
				DA_{event}	Absorbed dose per event	Chemical-specific	mg/cm^2-event	USEPA 2004	$DA_{event} \times EV \times ED \times EF \times SA \times 1/BW \times 1/AT$
				EV	Event frequency	1	events/day	USEPA 2014	
				ED	Exposure duration	6	years	USEPA 2014	Where for inorganic chemicals:
				EF	Exposure frequency	350	days/year	USEPA 2014	Absorbed Dose per Event (DA_{event}) (mg/cm^2-event) = $DA_{event} = Kp \times EPCgw \times t\text{-event} \times CF$
				SA	Skin surface area available for contact	6,365	cm^2	USEPA 2014	
				Kp	Permeability coefficient	Chemical-specific	cm/hour	USEPA 2019b	And where for organic chemicals:
				t-event	Event duration	0.54	hours/event	USEPA 2014	If t-event < t*, then: $DA_{event} = 2FA \times Kp \times EPCgw \times CF \times SQRT\{(6 \times tau\text{-event} \times t\text{-event})/pi\}$
				CF	Volumetric conversion factor for water	0.001	L/cm^3	--	
				t*	Time to reach steady-state = 2.4×tau-event	Chemical-specific	hours	USEPA 2019b	If t-event > t*, then: $DA_{event} = FA \times Kp \times EPCgw \times CF \times \{(t\text{-event}/(1 + B)) + 2 \times tau\text{-event} \times ((1 + (3 \times B) + (3 \times B^2))/(1 + B)^2)\}$
				FA	Fraction absorbed	Chemical-specific	unitless	USEPA 2004	
				tau-event	Lag time per event	Chemical-specific	hours/event	USEPA 2019b	
				B	Ratio of permeability coefficient of a chemical through the stratum corneum relative to its permeability coefficient across the viable epidermis	Chemical-specific	unitless	USEPA 2019b	
				BW	Body weight	15	kg	USEPA 2014	
				ATc	Averaging time, cancer	25,550	days	USEPA 1989	
				ATn	Averaging time, non-cancer	2,190	days	USEPA 1989	

RAGS Part D Table 4.8
Values Used for Daily Intake Calculations - Reasonable Maximum Exposure
Nike Antiaircraft Missile Battery BU-51/52
Town of Hamburg, Erie County, New York

Scenario Timeframe: Hypothetical Future
Medium: Off-site Groundwater
Exposure Medium: Potable Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation	Off-site Resident	Adult	Residence Indoor Air	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	24	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	350	days/year	USEPA 2014	
				ED	Exposure duration	20	years	USEPA 2014	where, for volatile constituents:
				VF-tap	Volatilization factor for tapwater	0.5	L/m^3	USEPA 2019c	EPCa = EPCgw × VF-tap
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	175,200	hours	USEPA 2009	
		Child	Residence Indoor Air	EPCa	Exposure point concentration, air	Chemical-specific	mg/m^3	Calculated	Exposure Concentration (mg/m^3) =
				ET	Exposure time	24	hours/day	USEPA 2014	EPCa × ET × EF × ED × 1/AT
				EF	Exposure frequency	350	days/year	USEPA 2014	
				ED	Exposure duration	6	years	USEPA 2014	where, for volatile constituents:
				VF-tap	Volatilization factor for tapwater	0.5	L/m^3	USEPA 2019c	EPCa = EPCgw × VF-tap
				ATc	Averaging time, cancer	613,200	hours	USEPA 2009	
				ATn	Averaging time, non-cancer	52,560	hours	USEPA 2009	

Notes:
USEPA - United States Environmental Protection Agency.

References:

USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS): Volume 1, Human Health Evaluation Manual, Part A. EPA/5401-89/002. Office of Emergency and Remedial Response, Washington, DC. December.
USEPA. 2004. Risk Assessment Guidance for Superfund. Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment). Final. EPA/540/R/99/005. Office of Superfund Remediation and Technology Innovation, Washington, DC. July.
USEPA. 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment). Final. EPA-540-R-070-002. Office of Superfund Remediation and Technology Innovation, Washington, DC. January.
USEPA. 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. OSWER Directive 9200.1-120. Office of Solid Waste and Emergency Response, Washington, DC. February.
USEPA. 2019b. Chemical Specific Parameters Table. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables.
USEPA. 2019c. User's Guide. Regional Screening Levels. Accessed online: https://www.epa.gov/risk/regional-screening-levels-rsls-users-guide.

ATTACHMENT B

Record of Phone Conversation (Arcadis and Town of Hamburg), 21 November 2019

RECORD OF CONVERSATION

Arcadis U.S., Inc.
17-17 Route 208 North
Suite 290 West
Fair Lawn
New Jersey 07410
Tel 201 797 7400
Fax 201 797 4399

Subject:

Future Land Use at Former Launch Area, Nike BU-51/52 Site

Department:

Human Health Risk Assessment

Arcadis Project No.:

30027953

Record by:

Julie Gillespie

Issue Date

November 21, 2019

SUMMARY OF PHONE CONVERSATION RE: FUTURE LAND USE

- Julie Gillespie (Arcadis) called the Town of Hamburg Building Department and asked to speak with Mike Quinn, the Town Engineer, who had previously been identified as the point of contact for the Nike BU-51/52 site (Nike Battery Site) in Hamburg, New York. Julie explained she had twice recently attempted to contact the Town Planner to inquire about future land use at the former Launch Area at the Nike Battery Site. She had left two voice messages but did not hear back. Mike stated the Town Planner was on vacation until Monday, November 25. He asked if there was something he could assist with.
- Julie indicated that as part of the risk assessment work plan for the remedial investigation of the former Launch Area, she needed to identify whether future residential development is a realistic scenario. The Town of Hamburg Master Plan depicts the Nike Battery Site, including the former Launch Area, as public land and indicates this general area of town contains rural residential development with open space, parks, and undeveloped lands. The former Launch Area is zoned for residential-agricultural use, and there are houses south of the former Launch Area, along Lakeview Road; however, given the current site ownership and use by the Town of Hamburg Highway Department, it seems unlikely the former Launch Area would ever be redeveloped for residential use.
- Mike stated the Nike site will never be redeveloped for residential use, and zoning has little meaning in terms of realistic future use. The Highway Department has used the former Launch Area for over 20 years and will likely do so in the future; and there are silos buried on that property. He offered to ask the Town Supervisor if he would put it in writing, but the former Launch Area will not be developed for residential use.

- Julie stated it's probably not necessary at this point and thanked Mike for the offer and for his time.

ACTION ITEMS

1. Julie Gillespie (Arcadis) to update the human health risk assessment work plan with details from phone conversation with Mike Quinn (Town Engineer, Hamburg, NY).

ATTACHMENT C

Ecological Soil Screening Tables

Table C-1. Nike BU-51/52 Soil Sampling Results (M&E, 1989) Compared to Ecological Screening Values

Sample Name:	Ecological Screening Value [1]		S-1	S-2	S-3	S-4	S-5
Sample Date:			3/29/1988	3/29/1988	3/29/1988	3/29/1988	3/29/1988
Volatile Organic Compounds (mg/kg)							
Methylene Chloride	0.21	[5],i	0.015 B	0.031 B	0.028 B	0.025 B	0.040 B
Acetone	1.2	[3],m	<0.011	0.024 B	0.016 B	0.017 B	0.013 B
Total Metals (mg/kg)							
Arsenic	18	[2],p	4.2	2.8	2.9	4.7	14
Barium	330	[2],i	40	37	39	29	29
Cadmium	0.36	[2],m	**0.51**	<0.48	<0.50	<0.49	<0.50
Chromium (Total)	23	[3],b	11	9.9	10	5.6	10
Lead	11	[2],b	11	9.2	8.5	9.3	**13**
Selenium	0.52	[2],p	0.17	<0.12	<0.13	0.24	**0.55**

Notes:
Green shaded and bolded values represent exceedance of Ecological Screening Values
"<" = not detected
B - compound detected in method blank
mg/kg = milligrams per kilogram
NA = Not Available

[1] Ecological Screening Values selected from USEPA Region 4 Guidance (2018) which include soil benchmarks for protection of plants (p), soil invertebrates (i), mammals (m) and birds (b) using the following hierarchy:
-USEPA Ecological Soil Screening levels (Eco-SSLs; USEPA 2007);
-Department of Energy Laboratories i.e., Los Alamos National Laboratory (LANL 2017); and
-Oak Ridge National laboratory (ORNL; Efroymson et al. 1997a, b).
-Equilibrium partitioning (EqP) methodology used to model values for organic chemicals.

[2] USEPA Eco-SSL (2007)

[3] LANL (2017)

[4] Efroymson et al. (1997a,b)

[5] USEPA (2018) Region 4 Eqp soil model

References:
Efroymson, R.A., M.E. Will, G.W. Suter, and A.C. Wooten. 1997a. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Oak Ridge National Laboratory, Oak Ridge, TN. ES/ER/TM-95/R4.
Efroymson, R.A., M.E. Will, and G.W. Suter. 1997b. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory, Oak Ridge, TN. ES/ER/TM-126/R2. https://rais.ornl.gov/documents/tm126r21.pdf
Los Alamos National Laboratory. 2017. ECORISK Database Release 4.1. September 2017. http://www.lanl.gov/environment/protection/eco-risk-assessment.php
USEPA. 2007. Ecological Soil Screening Levels. https://rais.ornl.gov/guidance/epa_eco.html
USEPA. 2018. Region 4 Ecological Risk Assessment Supplemental Guidance. March 2018 Update.

Table C-2. Nike BU-51/52 Surface Soil Sampling Results for the Launch Area (GZA, 1999) Compared to Ecological Soil Screening Values

Sample Name: / Sample Date:	Ecological Screening Value [1]		Upgradient of Launch Area NB-LA-1 3/24/1999	Downgradient of Launch Area NB-LA-2 3/24/1999	Downgradient of Launch Area NB-LA-3 3/24/1999	Downgradient of Launch Area NB-LA-4 3/24/1999	Lakeview Road, Downgradient of Launch Area NB-LA-5 3/24/1999	Lakeview Road, Downgradient of Launch Area NB-LA-6 3/25/1999
Volatile Organic Compounds (mg/kg)								
Chloromethane	NA		n/a	n/a	n/a	n/a	<0.013	n/a
Methylene Chloride	0.21	(5),i	n/a	n/a	n/a	n/a	0.025 B	n/a
Acetone	1.2	(3),m	n/a	n/a	n/a	n/a	0.029 B	n/a
2-Butanone	1	(5),i	n/a	n/a	n/a	n/a	<0.013	n/a
Toluene	0.15	(5),i	n/a	n/a	n/a	n/a	<0.013	n/a
Semivolatile Organic Compounds (mg/kg)								
Acenaphthene	See Total LMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Dibenzofuran	0.15	(5),i	n/a	n/a	<0.46	n/a	<0.44	n/a
Phenanthrene	See Total LMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Anthracene	See Total LMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Carbazole	0.07	(5),i	n/a	n/a	<0.46	n/a	<0.44	n/a
di-n-butylphthalate	0.011	(3),b	n/a	n/a	<0.46	n/a	<0.44	n/a
Fluoranthene	See Total HMW PAH		n/a	n/a	0.42 J	n/a	0.046 J	n/a
Pyrene	See Total HMW PAH		n/a	n/a	<0.46	n/a	0.077 J	n/a
Butylbenzylphthalate	0.59	(5),i	n/a	n/a	<0.46	n/a	0.065 J	n/a
Benzo(a)anthracene	See Total HMW PAH		n/a	n/a	0.1 J	n/a	<0.44	n/a
Chrysene	See Total HMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Benzo(b)fluoranthene	See Total HMW PAH		n/a	n/a	<0.46	n/a	0.52 J	n/a
Benzo(k)fluoranthene	See Total HMW PAH		n/a	n/a	<0.46	n/a	0.92 J	n/a
Benzo(a)pyrene	See Total HMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Ideno(1,2,3-cd)pyrene	See Total HMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Dibenzo(a,h)anthracene	See Total HMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a
Benzo(g,h,i)perylene	See Total HMW PAH		n/a	n/a	<0.46	n/a	<0.44	n/a

Table C-2. Nike BU-51/52 Surface Soil Sampling Results for the Launch Area (GZA, 1999) Compared to Ecological Soil Screening Values

Sample Name:	Ecological Screening Value [1]		Upgradient of Launch Area	Downgradient of Launch Area	Downgradient of Launch Area	Downgradient of Launch Area	Lakeview Road, Downgradient of Launch Area	Lakeview Road, Downgradient of Launch Area
			NB-LA-1	NB-LA-2	NB-LA-3	NB-LA-4	NB-LA-5	NB-LA-6
Sample Date:			3/24/1999	3/24/1999	3/24/1999	3/24/1999	3/24/1999	3/25/1999
Total LMW PAHs [6]	29.0	(2),i	n/a	n/a	0	n/a	0	n/a
Total HMW PAHs [6]	1.1	(2),m	n/a	n/a	0.52	n/a	**1.56**	n/a
Pesticides/PCBS (mg/kg)								
BHC (d-isomer)	NA		n/a	n/a	n/a	n/a	0.0014 J	n/a
Heptachlor	0.0016	(5),i	n/a	n/a	n/a	n/a	<0.00227	n/a
4,4'-DDT	0.021 (Total DDT)	(2),m	n/a	n/a	n/a	n/a	<0.0044	n/a
Methoxychlor	0.0021	(5),i	n/a	n/a	n/a	n/a	<0.0227	n/a
Total Metals (mg/kg)								
Aluminum	Narrative (pH based)	(2)	n/a	n/a	6,420	n/a	9,080	8,320
Antimony	0.27	(2),m	n/a	n/a	**4.6 B**	n/a	<4	<3.7
Arsenic	18.0	(2),p	<2.4	2.3	9.4	9.8	5.4	<2.5
Barium	330	(2),i	127	74.2	56	58.2	81.9	46.4 B
Beryllium	21	(2),m	2.6	1.0 B	<0.73	<0.78	<0.8	<0.74
Cadmium	0.36	(2),m	**1.5**	**1.7**	**1.6**	**1.7**	**2.0**	**1.4**
Calcium	NA		n/a	n/a	2,040	n/a	3,670	52,900
Chromium	23	(3),b	10.9	12.5	10.6	12.4	14.9	14.1
Cobalt	13	(2),p	n/a	n/a	8.1 B	n/a	9.6 B	10.8 B
Copper	28	(2),b	25.2	28.0	19.4	18.2	18.9	**30.1**
Iron	Narrative (pH based)	(2)	n/a	n/a	21,000	n/a	24,000	21,000
Lead	11	(2),b	**19.8**	**15.3**	**21.9**	**16.0**	**27.1**	**15.8**
Magnesium	NA		n/a	n/a	1,320	n/a	2,180	10,700
Manganese	220	(2),p	n/a	n/a	**295**	n/a	**611**	**482**
Mercury	0.013	(3),b	<0.12	<0.11	**0.20**	<0.13	**0.18**	<0.12
Nickel	38	(2),p	23.1	33.8	18.2	23.2	22.2	28.3
Potassium	NA		n/a	n/a	1,320	n/a	973 B	1,880
Selenium	0.52	(2),p	**47.6**	**31.0**	**2.50**	**3.80**	**6.70**	**36.6**
Sodium	NA		n/a	n/a	361 B	n/a	<267	<247
Thallium	0.05	(3),p	n/a	n/a	<2.4	n/a	<2.7	**13.60**
Vanadium	7.8	(2),b	n/a	n/a	**23.1**	n/a	**25.0**	**18.1**

Table C-2. Nike BU-51/52 Surface Soil Sampling Results for the Launch Area (GZA, 1999) Compared to Ecological Soil Screening Values

Sample Name:	Ecological Screening Value [1]		Upgradient of Launch Area	Downgradient of Launch Area	Downgradient of Launch Area	Downgradient of Launch Area	Lakeview Road, Downgradient of Launch Area	Lakeview Road, Downgradient of Launch Area
			NB-LA-1	NB-LA-2	NB-LA-3	NB-LA-4	NB-LA-5	NB-LA-6
Sample Date:			3/24/1999	3/24/1999	3/24/1999	3/24/1999	3/24/1999	3/25/1999
Zinc	46	[2],b	**112**	**87.5**	**103**	**114**	**278**	**74.2**
Total Cyanide	0.1	[3],b	<1.2	<1.2	<1.2	<1.2	<1.3	

Notes:

Green shaded and bolded values represent exceedance of Ecological Screening Values

"<" = not detected

B = compound detected in method blank

J = compound may be present at a concentration less than detection limit

mg/kg = milligrams per kilogram

NA = Not Available

n/a - not analyzed

[1] Ecological Screening Values selected from USEPA Region 4 Guidance (2018) which include soil benchmarks for protection of plants (p), soil invertebrates (i), mammals (m) and birds (b) using the following hierarchy:

-USEPA Ecological Soil Screening levels (Eco-SSLs; USEPA 2007);

-Department of Energy Laboratories i.e., Los Alamos National Laboratory (LANL 2017); and

-Oak Ridge National laboratory (ORNL; Efroymson et al. 1997a, b).

-Equilibrium partitioning (EqP) methodology used to model values for organic chemicals.

[2] USEPA Eco-SSL (2007)

[3] LANL (2017)

[4] Efroymson et al. (1997a,b)

[5] USEPA (2018) Region 4 Eqp soil model

[6] Total HMW PAHs and total LMW PAHs summed assuming each non-detect value is equal to 0.

References:

Efroymson, R.A., M.E. Will, G.W. Suter, and A.C. Wooten. 1997a. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Oak Ridge National Laboratory, Oak Ridge, TN. ES/ER/TM-95/R4.

Efroymson, R.A., M.E. Will, and G.W. Suter. 1997b. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory, Oak Ridge, TN. ES/ER/TM-126/R2. https://rais.ornl.gov/documents/tm126r21.pdf

Los Alamos National Laboratory. 2017. ECORISK Database Release 4.1. September 2017. http://www.lanl.gov/environment/protection/eco-risk-assessment.php

USEPA. 2007. Ecological Soil Screening Levels. https://rais.ornl.gov/guidance/epa_eco.html

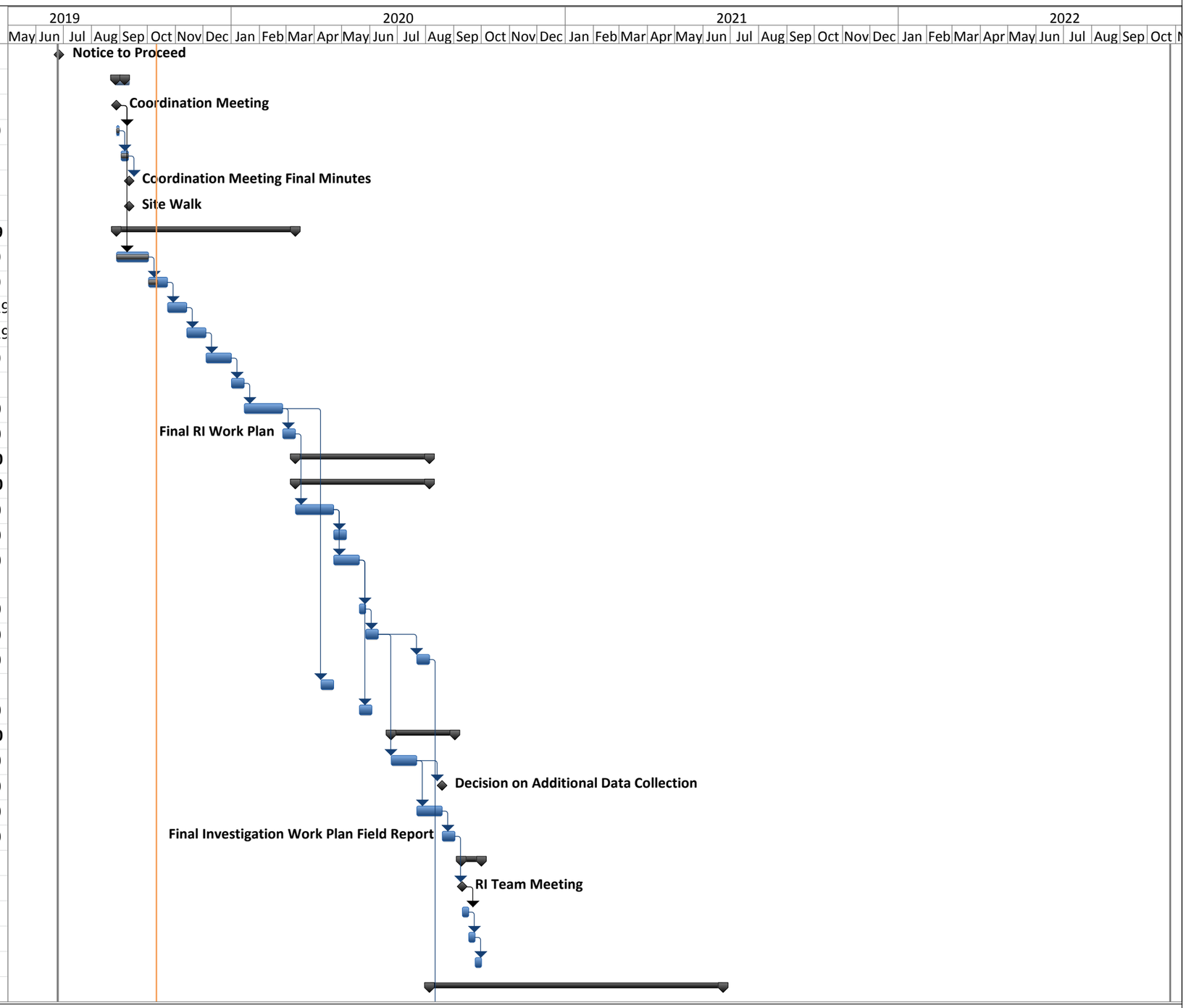
USEPA. 2018. Region 4 Ecological Risk Assessment Supplemental Guidance. March 2018 Update.



APPENDIX D
Schedule

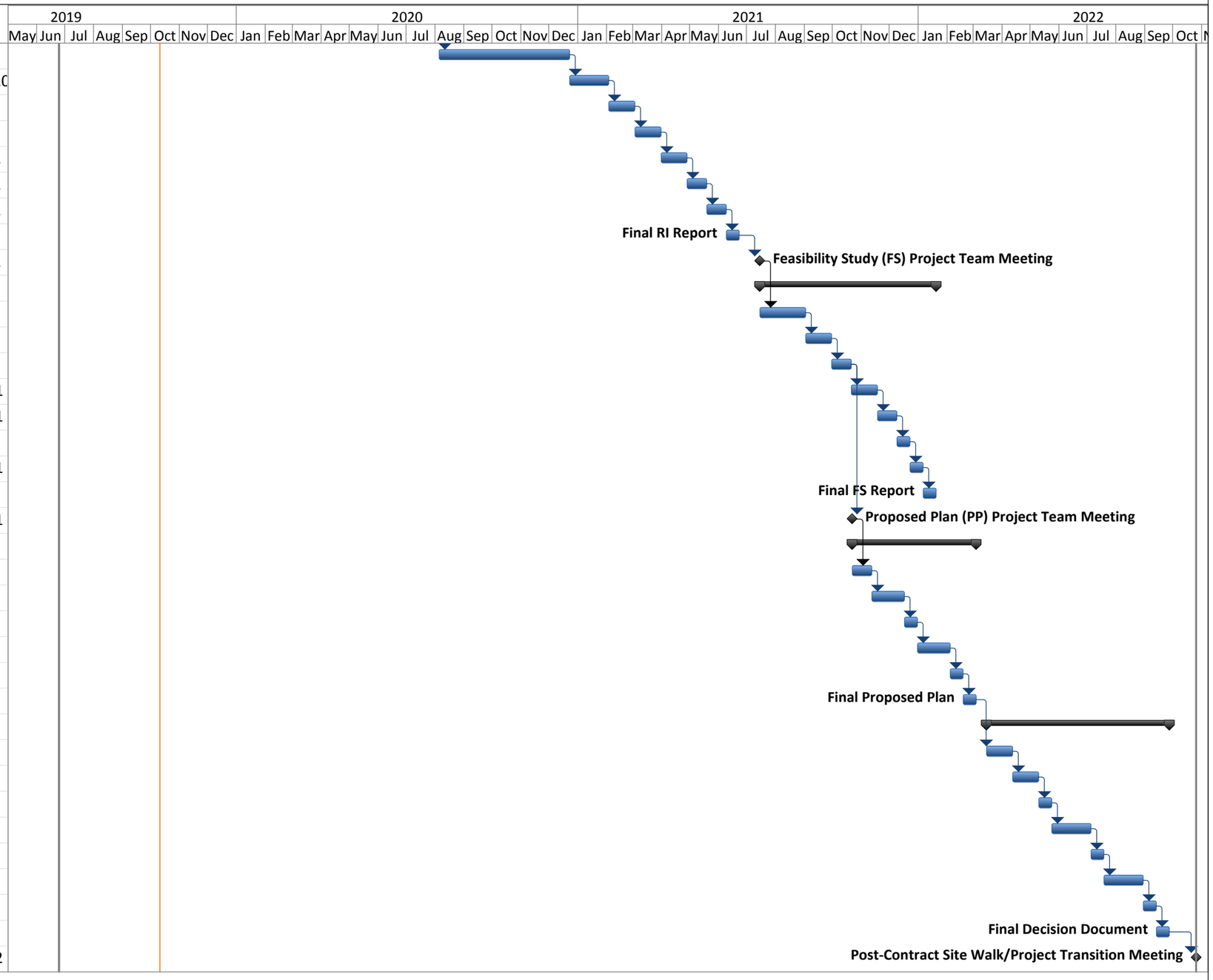
Project Schedule
Nike BU 51-52 FUDS Program RI/FS
Hamburg, New York

ID	Task Name	Duration	Start
1	***Notice to Proceed***	***1 day***	***Tue 6/25/19***
2	**Review Data/Coordination Meeting**	**8 days**	**Tue 8/27/19**
3	Coordination Meeting	1 day	Tue 8/27/19
4	Coordination Meeting Draft Minutes	3 days	Wed 8/28/19
5	RTCs	6 days	Mon 9/2/19
6	Coordination Meeting Final Minutes	1 day	Tue 9/10/19
7	Site Walk	1 day	Tue 9/10/19
8	**RI Work Plan Documents (WP, QAPP, APP, CRP)**	**140 days**	**Wed 8/28/19**
9	Draft Work Plan Documents	25 days	Wed 8/28/19
10	USACE Review	15 days	Wed 10/2/19
11	RTCs	15 days	Wed 10/23/19
12	Cx Review	15 days	Wed 11/13/19
13	RTCs	20 days	Wed 12/4/19
14	Draft Final RI Work Plan Documents Review	10 days	Wed 1/1/20
15	RTCs	30 days	Wed 1/15/20
16	Final RI Work Plan	10 days	Wed 2/26/20
17	**Execute RI Work Plan**	**105 days**	**Wed 3/11/20**
18	**Soil and Groundwater Investigation**	**105 days**	**Wed 3/11/20**
19	Utility Clearance	30 days	Wed 3/11/20
20	Surface Soil Sampling	10 days	Wed 4/22/20
21	Monitoring Well Installation/Subsurface Soil Investigation	20 days	Wed 4/22/20
22	Monitoring Well Development	5 days	Wed 5/20/20
23	Groundwater Sampling Event #1	10 days	Wed 5/27/20
24	Groundwater Sampling Event #2	10 days	Wed 7/22/20
25	Site Ecological Habitat Assessment	10 days	Wed 4/8/20
26	Site Topographical Survey	10 days	Wed 5/20/20
27	**RI Field Report**	**50 days**	**Wed 6/24/20**
28	Draft Investigation Work Plan Field Report	20 days	Wed 6/24/20
29	Decision on Additional Data Collection	20 days	Wed 7/22/20
30	RTCs & Groundwater Sampling Event #2 Data	20 days	Wed 7/22/20
31	Final Investigation Work Plan Field Report	10 days	Wed 8/19/20
32	**RI Project Team Meeting**	**16 days**	**Wed 9/9/20**
33	RI Team Meeting	1 day	Wed 9/9/20
34	Draft RI Project Team Meeting Minutes	5 days	Thu 9/10/20
35	RTCs	5 days	Thu 9/17/20
36	Final RI Project Team Meeting Minutes	5 days	Thu 9/24/20
37	**RI Report**	**230 days**	**Wed 8/5/20**



Project Schedule
Nike BU 51-52 FUDS Program RI/FS
Hamburg, New York

ID	Task Name	Duration	Start
38	Draft RI Report	100 days	Wed 8/5/20
39	USACE Review	30 days	Wed 12/23/20
40	RTCs	20 days	Wed 2/3/21
41	Cx Review	20 days	Wed 3/3/21
42	RTCs	20 days	Wed 3/31/21
43	Draft Final RI Report Review	15 days	Wed 4/28/21
44	RTCs	15 days	Wed 5/19/21
45	Final RI Report	10 days	Wed 6/9/21
46	Feasibility Study (FS) Project Team Meeting	1 day	Wed 7/14/21
47	**FS Report**	**135 days**	**Thu 7/15/21**
48	Draft FS Report	35 days	Thu 7/15/21
49	USACE Review	20 days	Thu 9/2/21
50	RTCs	15 days	Thu 9/30/21
51	Cx Review	20 days	Thu 10/21/21
52	RTCs	15 days	Thu 11/18/21
53	Draft Final FS Report Review	10 days	Thu 12/9/21
54	RTCs	10 days	Thu 12/23/21
55	Final FS Report	10 days	Thu 1/6/22
56	Proposed Plan (PP) Project Team Meeting	1 day	Thu 10/21/21
57	**Proposed Plan**	**95 days**	**Fri 10/22/21**
58	Draft PP	15 days	Fri 10/22/21
59	USACE Review	25 days	Fri 11/12/21
60	RTCs	10 days	Fri 12/17/21
61	Cx Review	25 days	Fri 12/31/21
62	RTCs	10 days	Fri 2/4/22
63	Final Proposed Plan	10 days	Fri 2/18/22
64	**Decision Document (DD)**	**140 days**	**Tue 3/15/22**
65	Draft DD	20 days	Tue 3/15/22
66	USACE Review	20 days	Tue 4/12/22
67	RTCs	10 days	Tue 5/10/22
68	CX Review	30 days	Tue 5/24/22
69	RTCs	10 days	Tue 7/5/22
70	Draft Final DD	30 days	Tue 7/19/22
71	RTCs	10 days	Tue 8/30/22
72	Final Decision Document	10 days	Tue 9/13/22
73	Post-Contract Site Walk/Project Transition Meeting	1 day	Tue 10/25/22



APPENDIX E
Field SOPs

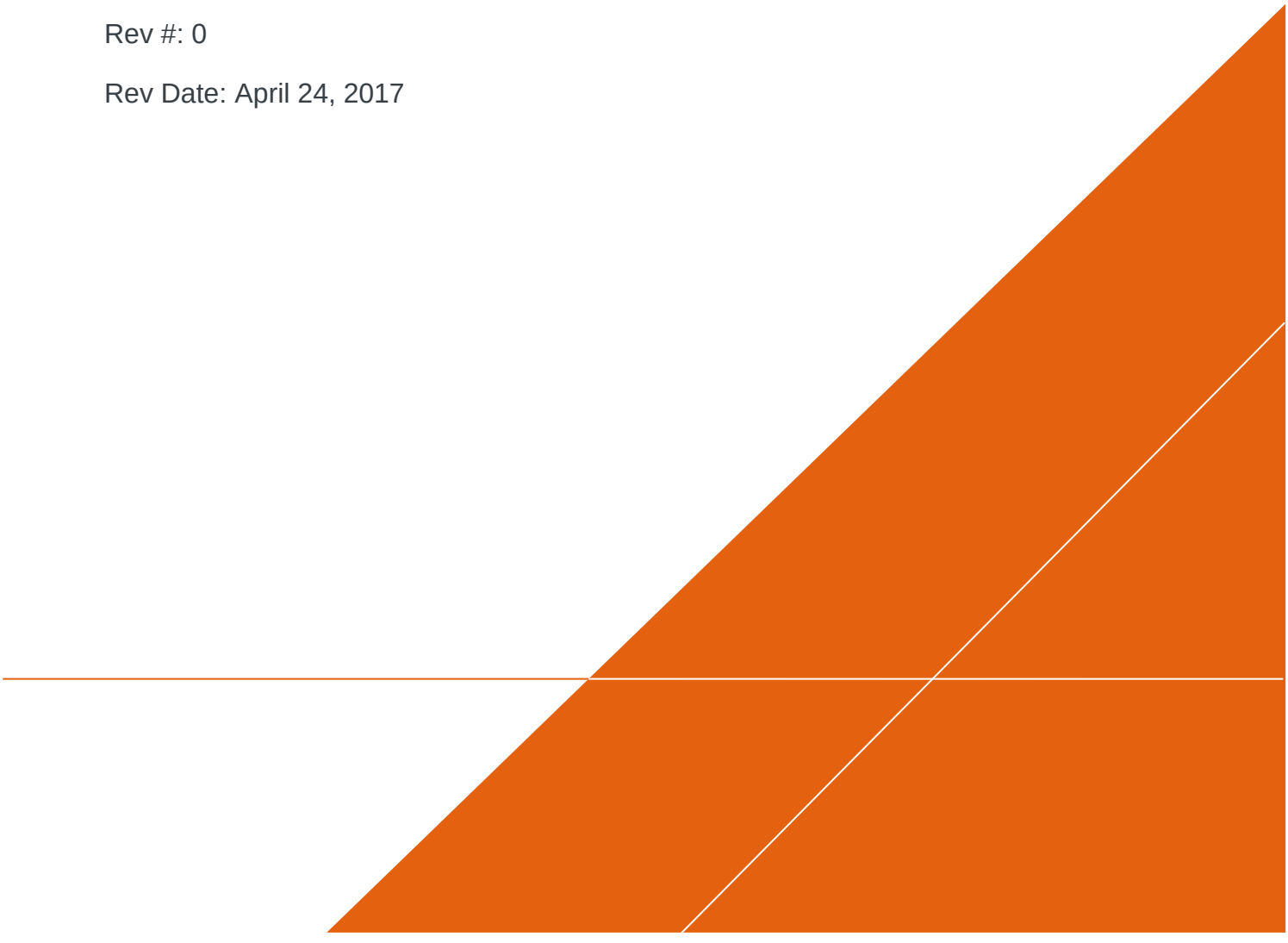
RI-SOP-1

ARCADIS Design & Consultancy for natural and built assets

TGI - MONITORING WELL INSTALLATION

Rev #: 0

Rev Date: April 24, 2017



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
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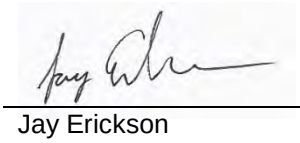
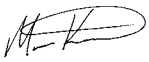
APPROVAL SIGNATURES

Prepared by:		4/20/17
	Jay Erickson	Date:
Technical Expert Reviewed by:		4/24/17
	Marc Killingstad	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) describes methods used to install groundwater monitoring wells in granular aquifers. It is assumed that the monitoring well has been properly designed, including sizing of the filter pack and screen, the length of the screen, total depth of the well, material strength and compatibility and surface completion. Typical monitoring wells are constructed of manufactured screen and engineered filter pack and are generally suitable for formations with granular materials having a grain size distribution with up to 50% passing a #200 sieve and up to 20% clay-sized material. Monitoring wells installed in formations finer than this may not be able to produce turbidity free water.

The monitoring well installation procedures set forth herein are consistent with the approach and methods presented in the American Society of Testing and Materials (ASTM) D5092 – *Standard Practice for Design and Installation of Groundwater Monitoring Wells* (ASTM D5092). As such, following this TGI in combination with proper well design (see appropriate TGI), well development (see appropriate TGI), groundwater sampling procedures (see appropriate TGI), and well maintenance and rehabilitation (see appropriate TGI), will result in a monitoring well suitable for: (1) collection of groundwater samples

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representative of the surrounding formation and free of artificial turbidity; (2) measurement of accurate groundwater levels; and (3) hydraulic conductivity testing of formation sediments immediately adjacent to the open interval of the well (e.g., slug testing).

Monitoring well boreholes in unconsolidated (overburden) materials are typically drilled using the hollow-stem auger drilling method. Other drilling methods that are also suitable for installing overburden monitoring wells, and are sometimes necessary due to site-specific geologic conditions or project objectives, include: drive-and-wash, spun casing, Rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and driven well points may also be used in some cases within the overburden. Monitoring wells to be installed within consolidated materials such as fractured bedrock are commonly drilled using water-rotary (coring or tri-cone roller bit), air rotary or Rotasonic methods. For guidance when installing monitoring wells in consolidated materials, please refer to the appropriate document. The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling/well depths, site or regional geologic knowledge, type of monitoring to be conducted using the installed well, project objectives, and cost.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). No polyvinyl chloride (PVC) glue/cement will be used in constructing or retrofitting monitoring wells that will be used for water-quality monitoring. No coated bentonite pellets will be used in the well drilling or construction process. Specifications of materials to be installed in the borehole will be obtained prior to mobilizing onsite; these materials generally include:

- Well casing (length, material, and diameter);
- Well screen (length, material, diameter, and slot size);
- Bentonite (type, as applicable, chips, non-coated and granular bentonite are acceptable);
- Filter pack (filter pack type and fine sand seal type, as applicable); and
- Grout (type, as applicable).

Well materials will be inspected and, if needed, cleaned or replaced prior to installation.

3 PERSONNEL QUALIFICATIONS

Monitoring well installation activities will be performed by persons who have been trained in proper well installation procedures under the guidance of an experienced field geologist, engineer, or technician. Where field sampling is performed for soil or bedrock characterization, field personnel will have undergone in-field training in soil or bedrock description methods, as described in the appropriate Standard Operating Procedures (SOPs) and/or TGIs for those activities.

4 EQUIPMENT LIST

The following materials will be available during soil boring and monitoring well installation activities, as required:

- Site Plan with proposed soil boring/well locations;

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- Work Plan (or equivalent), Field Sampling Plan (FSP), and site-specific Health and Safety Plan (HASP);
- Personal protective equipment (PPE), as required by the HASP;
- Traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area, if such are not provided by drillers;
- Appropriate soil sampling equipment (e.g., stainless steel spatulas, knife);
- Soil and/or bedrock logging equipment as specified in the appropriate project documents;
- Appropriate sample containers and labels;
- Drum labels as required for investigation derived waste handling;
- Chain-of-custody forms;
- Insulated coolers with ice, when collecting samples requiring preservation by chilling;
- Photoionization detector (PID) or flame ionization detector (FID);
- Ziplock style bags;
- Water level or oil/water interface meter;
- Locks and keys for securing the well after installation;
- Decontamination equipment (bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels);
- Engineer's tape/measuring wheel;
- Weighted tape;
- Disposable bailers;
- Digital camera (or phone with camera)
- Field notebook or Personal Digital Assistant (PDA); and
- Appropriate field forms, consider including a photo of the well head and a Google Earth map showing the well location.

Prior to mobilizing to the site, Arcadis personnel will contact the drilling subcontractor or in-house driller (as appropriate) to confirm that appropriate sampling and well installation equipment will be provided. Specifications of the sampling and well installation equipment are expected to vary by project, and so communication with the driller is necessary to ensure that the materials provided will meet the project objectives. Equipment/materials typically provided by the driller could include:

- Drilling equipment required by the ASTM standard guidance document D1586, when performing split-spoon sampling;
- Disposable plastic liners (when drilling with direct-push equipment);
- Drums for investigation derived waste;

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- Drilling and sampling equipment decontamination materials;
- Decontamination pad materials, if required; and
- Well construction materials.

5 CAUTIONS

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service. See appropriate guidance for proper utility clearance protocol.

Prior to beginning field work, contact the project technical team to ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members.

Some regulatory agencies require a minimum annular space between the well or permanent casing and the borehole wall. When specified, the minimum clearance is typically 2 inches on all sides (e.g., a 2-inch diameter well requires a 6-inch diameter borehole). In addition, some regulatory agencies have specific requirements regarding grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the drilling and well installation plan.

If dense non-aqueous phase liquids (DNAPL) are known or expected to exist at the site, refer to the project specific documents for additional details regarding drilling and well installation to reduce the potential for inadvertent DNAPL remobilization.

Similarly, if light non-aqueous phase liquids (LNAPLs) are known or expected to be present as “perched” layers above the water table, refer to the DNAPL Contingency Plan. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Similarly, consider the compatibility between the well materials and the surrounding environment. For example, PVC well materials are not preferred when DNAPL is present. In addition, some groundwater conditions leach metals from stainless steel or are corrosive to metal well materials. If questions arise, contact the CPM and/or project technical lead to discuss.

Water used for drilling and sampling of soil or bedrock, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply should be considered.

Specifications of materials used for backfilling the borehole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPLs are likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.

As noted above, coated bentonite pellets will not be used in monitoring well construction, as the coating could impact the water quality in the completed well.

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Heat of hydration during neat cement grout curing must be considered to avoid damage to PVC well materials. The annular space for a typical monitoring well is small enough that heat of hydration should not create excessive temperature increases which may damage PVC well material. However, washouts in the borehole can lead to thick accumulations of grout which can produce enough heat during curing to weaken and potentially damage PVC casing. If heat of hydration is a concern, contact the project technical lead to address the issue.

6 HEALTH AND SAFETY CONSIDERATIONS

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

7 PROCEDURE

The procedures for installing groundwater monitoring wells are presented below:

Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

1. Prior to monitoring well installation, determine the expected volumes of filter pack and seal materials including bentonite (if applicable) and grout (neat cement or cement-bentonite).
2. Locate boring/well location, establish work zone, and set up sampling equipment decontamination area.
3. Advance boring to desired depth. Collect soil and/or bedrock samples at appropriate interval as specified in the Work Plan (or equivalent) and/or FSP. Collect, document, and store samples for laboratory analysis as specified in the Work Plan and/or FSP. Decontaminate equipment between samples in accordance with the Work Plan (or equivalent) and/or FSP. A common sampling method that produces high-quality soil samples with relatively little soil disturbance is described in ASTM D1586 – *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils* (ASTM D1586). Split-spoon samples are obtained during drilling using hollow-stem auger, drive-and-wash, spun casing, and fluid/mud rotary. Rotasonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks. Dual-rotary removes cuttings by compressed air or water/mud and allow only a general assessment of geology.
4. Describe each soil sample as outlined in the appropriate project records. Record descriptions in the field notebook and/or personal digital assistant (PDA). It is also beneficial to photo document the samples. It should be noted that PDA logs must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data. During soil boring advancement, document all drilling events in field notebook, including blow counts (number of blows required to advance split-spoon sampler in 6-inch increments) and work stoppages. Blow counts will not be available if Rotasonic, dual-rotary, or direct-push methods are used.

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5. If it is necessary to install a monitor well into a permeable zone below a confining layer, particularly if the deeper zone is believed to have water quality that differs significantly from the zone above the confining layer, then a telescopic well construction should be considered. In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer, and a permanent casing (typically PVC, black steel or stainless steel) is installed into the socket drilled into the top of the confining layer. The casing is then grouted in place. The preferred methods of grouting telescoping casings include: pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole. In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Site-specific criteria and work plans should be created for the completion of non-standard monitoring wells, including telescopic wells.

6. Before installing a screened, it is important to confirm that the borehole has been advanced into the targeted saturated zone. This is particularly important for wells installed to monitor the water table and/or the shallow saturated zone, as the capillary fringe may cause soils above the water table to appear saturated. If one or more previously installed monitoring wells exist nearby, use the depth to water at such well(s) to estimate the water-table depth at the new borehole location.

   To verify that the borehole has been advanced into the saturated zone, it is necessary to measure the water level in the borehole. For boreholes drilled without using water (e.g., hollow-stem auger, cable-tool, air rotary, air hammer), verify the presence of groundwater (and /or LNAPL, if applicable) in the borehole using an electronic water level probe, oil-water interface probe, or a new or decontaminated bailer. For boreholes drilled using water (e.g., drive and wash, spun-casing with roller-bit wash, Rotasonic, or water rotary with core or roller bit), monitor the water level in the borehole as it re-equilibrates to the static level. In low-permeability units like clay, fine-grained glacial tills, shale and other bedrock formations, it may be necessary to wait overnight to allow the water level to equilibrate. Document depth to water in the borehole on the appropriate field forms and field notebook. If there are questions concerning the depth of the well/screen interval, consult with the project technical lead prior to finalizing well depth/screen interval. To the extent practicable, ensure that the depth of the well below the apparent water table is deep enough so that the installed well can monitor groundwater year-round, accounting for seasonal water-table fluctuations. When in doubt, err on the side of slightly deeper well installation.

7. Upon completing the borehole to the desired depth, if a screened well construction is desired, install the monitoring well by lowering the screen and casing assembly with sump through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter (although sometimes 4-inch), flush-threaded PVC or stainless steel slotted or wire wrapped well screen and blank riser casing. Smaller diameters may be used if wells are installed using direct-push methodology or if multiple wells are to be installed in a single borehole. The screen length will be specified in the Work Plan (or equivalent) or FSP based on regulatory requirements and specific monitoring objectives. Monitoring well screens are usually 5 to 10 feet long, but may be up to 25 feet long in very low permeability, thick

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geologic formations. The screen length will depend on the purpose for the well and the objectives of the groundwater investigation and will (in most cases) be determined prior to the field mobilization.

The slot size and filter pack gradation should be predetermined in the Work Plan (or equivalent) or FSP and based on site-specific grain-size analysis (sieve analysis) or other geologic considerations or monitoring objectives. Typically, slot sizes for monitoring wells will range from 0.010 inches to 0.020 inches while the filter pack will be 20-40, Morie No. 0, or equivalent. In very fine-grained formations where sample turbidity needs to be minimized, it may be preferred to use a 0.006-inch slot size and 30-65, Morie No. 00, or equivalent filter pack. Alternatively, where monitoring wells are installed in coarse-grained deposits and higher well yield is required, a 0.020-inch slot size and 10-20, Morie No. 1, or equivalent filter pack may be preferred. If the screen slot size and filter pack have not been based on site-specific grain-size analysis, consider collecting soil samples during well installation so future wells can be properly designed.

A blank sump may be attached below the well screen if the well is being installed for DNAPL recovery/monitoring purposes. If so, the annular space around the sump may be backfilled with neat cement grout using a tremie to the bottom of the well screen prior to placing the filter pack around the screen. A blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well. For wells greater than 50 feet deep, centralizers may be desired to assist in centering the monitoring well in the borehole during construction.

8. When the monitoring well assembly has been set in place and the grout has been placed around the sump (if any), place a washed silica filter pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen (following specifications in the Work Plan) using a tremie. The filter pack is placed and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth. Verify that the expected volume of filter pack matches with the actual amount installed. There can be differences due to irregularities in the borehole. Washout of the borehole will result in the need for greater than calculated well materials. If a difference of more than 10% is noted, consult with the project technical team. The filter pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan (or equivalent) or FSP. The well should be gently surged to prevent filter pack material bridging and to settled the filter pack prior to well seal installation.
9. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack (alternatively, in some cases a fine sand seal may be installed instead of bentonite—follow the specifications in the Work Plan). If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding. *No coated bentonite pellets will be used in monitoring well drilling or construction*. Potable water may be added to hydrate the bentonite if the seal is above the water table. Monitor the placement of the sand pack and bentonite with a weighted tape measure.
10. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 ft. below groundwater surface (bgs) or as specified in the Work Plan (or equivalent). As with the filter pack, it is recommended that seal material be placed with a tremie pipe. Ensure that seal materials are mixed at the proper ratios with water following manufacturer's recommendations.

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11. Install the monitoring well completion as specified Work Plan (or equivalent). Typical completions are a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing, and should slope gently away to promote drainage away from the well.
12. Monitoring wells should be labeled using indelible ink or paint with the appropriate designation on both the inner and outer well casings or inside of the curb box lid.
13. When an above-grade completion is used, the riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a small-diameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flush-mount installation is used, the riser will be sealed using an unvented, expandable locking plug.
14. During well installation, record construction details and actual measurements relayed by the drilling contractor and tabulate materials used (e.g., screen and riser footages; bags of bentonite, cement, and sand) in the field notebook as well as appropriate field forms.
15. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 7 below.

Direct-Push Method

The direct-push drilling method may also be used to complete soil borings and install monitoring wells. Examples of this technique include the Diedrich ESP vibratory probe system, GeoProbe®, or AMS Power Probe® dual-tube system. Environmental probe systems typically use a hydraulically operated percussion hammer. Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual-tube liner for sampling soil. The outside diameter (OD) of the outer casing ranges from 1.75 to 2.4 inches and the OD of the inner sampling tube ranges from 1.1 to 1.8 inches. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. The double-rod system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, the inside diameter (ID) of the steel casing provides clearance for the installation of small-diameter (e.g., 0.75- to 1-inch ID) micro-wells. The procedures for installing monitoring wells in soil using the direct-push method are described below.

1. Locate boring/well location, establish work zone, and set up sample equipment decontamination area.
2. Advance soil boring to designated depth, collecting samples at intervals specified in the Work Plan (or equivalent). Samples will be collected using dedicated, disposable, plastic liners. Describe samples in accordance with the procedures outlined in Step 3 above. Collect samples for laboratory analysis as specified in the Work Plan (or equivalent) and/or FSP.
3. Upon advancing the borehole to the desired depth, install the micro-well through the inner drill casing. The micro-well will consist of approximately 1-inch ID PVC or stainless steel slotted screen and blank riser. The sand pack, bentonite seal, and cement/bentonite grout will be installed as described, where applicable, in Steps 9 through 11 above.

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4. Install protective steel casing or flush-mount, as appropriate, as described in Step 12 above. During well installation, record construction details and tabulate materials used in field notebook as well as appropriate field forms.
5. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 8 below.

Driven Well Point Installation

Well points will be installed by pushing or driving using a drilling rig or direct-push rig, or hand-driven where possible. The well point construction materials will consist of a 1- to 2-inch-diameter threaded steel casing with either 0.010- or 0.020-inch slotted stainless steel screen. The screen length will vary depending on the hydrogeologic conditions of the site. The casings will be joined together with threaded couplings and the terminal end will consist of a steel well point. Because they are driven or pushed to the desired depth, well points do not have annular backfill materials such as sand pack or grout.

8 WASTE MANAGEMENT

Investigation-derived wastes (IDW), including soil cuttings and excess drilling fluids (if used), decontamination liquids, and disposable materials (well material packages, PPE, etc.), will be placed in clearly labeled, appropriate containers, or managed as otherwise specified in the Work Plan (or equivalent), FSP, and/or IDW management guidance document.

9 DATA RECORDING AND MANAGEMENT

Drilling activities should be documented on appropriate field/log forms as well as in a proper field notebook and/or PDA. Additionally, all documents (and photographs) should be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of well installation activities, soil descriptions, well construction specifications (screen and riser material and diameter, sump length, screen length and slot size, riser length, sand pack type), and quantities of materials used. In addition, the locations of newly-installed wells will be documented photographically or in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

The well location, ground surface elevation, and inner and outer casing elevations will be surveyed using the method specified in the site Work Plan (or equivalent). Generally, a local baseline control will be set up. This local baseline control can then be tied into the appropriate vertical and horizontal datum, such as the National Geodetic Vertical Datum of 1929 or 1988 and the State Plane Coordinate System. At a minimum, the elevation of the top of the inner casing used for water-level measurements should be measured to the nearest 0.01 foot. Elevations will be established in relation to the National Geodetic Vertical Datum of 1929. A permanent mark will be placed on top of the inner casing to mark the point for water-level measurements.

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10 QUALITY ASSURANCE

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate SOP. Well materials will also be cleaned prior to well installation.

11 REFERENCES

American Society for Testing Materials (ASTM) D5092 - *Standard Practice for Design and Installation of Ground Water Monitoring Wells*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

American Society of Testing and Materials (ASTM) D1586 - *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

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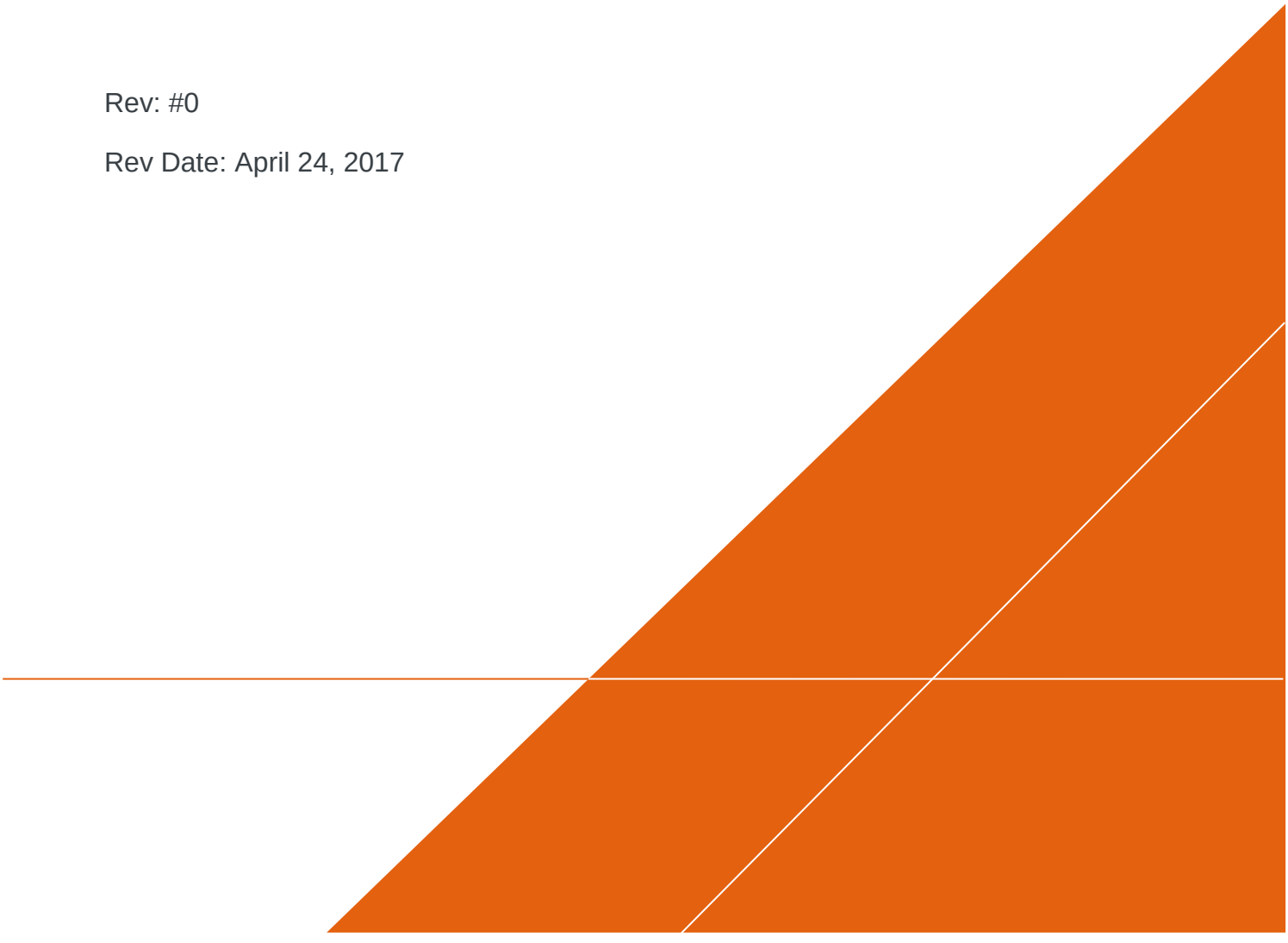
TECHNICAL GUIDANCE INSTRUCTION - MONITORING WELL DEVELOPMENT

Rev: #0

Rev Date: April 24, 2017



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	4/24/2017	All	Re-written as TGI	Marc Killingstad

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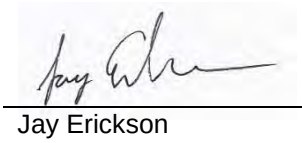
APPROVAL SIGNATURES

Prepared by:		4/24/2017
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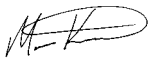
Technical Expert Reviewed by:		4/24/2017
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2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) covers the development of screened wells used for obtaining representative groundwater information and samples from granular aquifers (i.e., monitoring wells). Note that this TGI only applies to monitoring well development and not remediation (injection/extraction) well development.

The purposes of Monitoring Well Development are:

1. Repair damage to the borehole wall from drilling that can include clogging, smearing or compaction of aquifer materials;
2. Remove fine grained sediment from the formation and filter pack that may result in high turbidity levels in groundwater samples;
3. To re-sort formation and filter pack material adjacent to the well screen;

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4. To recover any drilling fluids (if used) that may affect the permeability of the formation and filter pack or alter the water quality around the well; and
5. To optimize the well efficiency and hydraulic communication between the well screen and the formation.

Successful monitoring well development is dependent on the following:

1. Hydrostratigraphy – Permeable formations containing primarily sand and gravel are more easily developed due to lower percentages of silt and clay material. Water in permeable formations can be moved in and out of the screen and/or through the formation easier than in less permeable deposits
2. Well Diameter – Development tooling including brushes, surge blocks, pumps and jetting tools are more readily available for wells 4 inches in diameter and greater.
3. Well Design – Wells with filter packs and screens designed to match the formation through the analysis of formation sieve samples are easier to develop. An important aspect to well design is to minimize the size of the annular space between the formation and well screen. Adequate room must be allowed for the proper installation of well materials, but not too large as to prevent/reduce communication with the surrounding formation.
4. Drilling Methods – Different drilling methods result in varying amount of borehole damage and, therefore, impact the degree to which development will be successful.

Well development methods for monitoring wells include the following:

1. Bailing – use of a bailer to remove water and sediment from the well casing. This technique does little to remove fines from the filter pack and may lead to bridging of sediment since the flow in only in one direction, toward the well screen.
2. Pumping/overpumping – use of a pump to remove water and sediment from the well casing, overpumping involves pumping the well at a rate that exceeds the design capacity of the well. Similar to bailing, this technique does little to remove fines from the filter pack and may lead to bridging of sediment since the flow in only in one direction, toward the well screen. Small diameter monitoring wells have the additional constraint on pump size and flow rates.
3. Backwashing (rawhiding) – consists of starting and stopping a pump intermittently to produce rapid pressure changes in a well. This method can produce better results than pumping alone since the procedure involves movement of the water in and out of the screen and formation. However, in many cases the surging action is not rigorous enough to fully develop the well.
4. Surging/swabbing – use of a mechanical surge block or swabbing tool to operate like a piston with an up and down motion. The downstroke causes a backwash action that breaks up bridged sediment and the upstroke pulls the dislodged sediment into the well. This method works well for small and large diameter wells. Care should be taken on the downstroke so as not to force fines back into the formation, frequent pumping/purging during surging help to keep fines out of the well. Double surge blocks are recommended.
5. Jetting – use of a tool fitted with nozzles that direct streams of water horizontally into well screens at high velocity. Due to the size of the tooling, this method is better suited for wells 4 inch in diameter and larger. The method is also more effective with wire-wrapped/continuous slot screens due to the

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increased open area. Jetting requires specialized equipment and concurrent pumping to prevent reintroducing fines into the filter pack. Additionally, jetting requires subsequent surging to remove fines dislodged in the filter pack and formation.

For most situations, gentle surging coupled with bailing or pumping to remove dislodged materials is recommended.

Well development for properly designed and constructed monitoring wells may begin after the annular seal materials have been installed and allowed to cure, since these wells are designed to retain 90-99% of the filter pack material. This cure time is typically at least 24 to 48 hours after the sealing materials have been installed.

This TGI is meant to provide a general guide for proper monitoring well development. A site-specific field implementation plan for well installation and development detailing the specific methods and tools should be developed to provide site-specific instruction and guidance.

3 PERSONNEL QUALIFICATIONS

Monitoring well development activities will be performed by persons who have been trained in proper well development procedures under the guidance of an experienced field geologist, engineer, or technician.

4 EQUIPMENT LIST

Required equipment depends on the selected method and should be detailed in the site-specific field implementation plan. However, the following are typically required.

- Health and safety equipment, as required by the site Health and Safety Plan (HASP):
- Cleaning equipment
- Field notebook and/or personal digital assistant (PDA)
- Monitoring well keys
- Water level indicator
- Field parameter meter (YSI)
- Well Development Logs
- Well construction logs/diagrams
- Weighted tape (measure depth)
- Turbidity meter
- Camera
- Watch/timing device.

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5 CAUTIONS

Where surging is performed to assist in removing fine-grained material from the sand pack, surging must be performed in a gentle manner. Excessive suction could promote fine-grained sediment entry into the outside of the sand pack from the formation.

Avoid using development fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

In some cases, it may be necessary to add potable water to a well to allow surging and development, especially for new monitoring wells installed in low permeability formations. Before adding potable water to a well, the Certified Project Manager (CPM) and/or Project Hydrogeologist must be notified and the CPM shall make the decision regarding the appropriateness and applicability of adding potable water to a well during well development procedures. If potable water is to be added to a well as part of development, the potable water source should be sampled and analyzed for constituents of concern, and the results evaluated by the CPM prior to adding the potable water to the well. If potable water is added to a well for development purposes, at the end of development the well will be purged dry to remove the potable water, or if the well no longer goes dry then the well will be purged to remove at least three times the volume of potable water that was added.

6 HEALTH AND SAFETY CONSIDERATIONS

Field activities associated with monitoring well development will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

7 PROCEDURE

As indicated above, for most monitoring wells, gentle surging coupled with bailing or pumping to remove dislodged sediment is recommended.

1 Ensure sufficient time has passed to allow for proper curing of the well seal.

2 Don appropriate PPE (as required by the site-specific HASP).

3 Place plastic sheeting around the well.

4 Clean all equipment entering each monitoring well, except for new, disposable materials that have not been previously used.

5 Open the well cover while standing upwind of the well, remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field notebook. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don the appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings.

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6 Obtain an initial measurement of the depth to water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book. It is recommended to use a weighted tape for the total well depth measurement.

7 The depth to the bottom of the well should be sounded and then compared to the completion form or construction diagram for the well. Any discrepancies should be reported immediately to the CPM and/or Project Hydrogeologist. If sand or sediment is present inside the well, it should first be removed by bailing. Do not insert bailers, pumps, or surge blocks into the well if obstructions, parting of the casing, or other damage to the well is suspected. Instead report the conditions to the CPM and/or Project Hydrogeologist and obtain approval to continue or cease well development activities.

8 Lower a double surge block into the screened portion of the well. Starting from the bottom of the screen using 2 foot throws, gently raise and lower the surge block to force water in and out of the screen slots and sand pack. Continue surging for 15 to 30 minutes.

9 Lower a bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well and gently bounce on the bottom of the well to collect/remove accumulated sediment, if any. Remove and empty the bailer, if used. Repeat until the bailed/pumped water is free of excessive sediment and contact at the bottom of the well feels solid. Alternatively, measurement of the well depth with a weighted tape can be used to verify that sediment and/or silt has been removed to the extent practicable, based on a comparison with the well installation log or previous measurement of total well depth.

10 After surging the well for a minimum of two cycles and removing excess accumulated sediment from the bottom of the well, re-measure the depth-to-water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.

11 Remove formation water by pumping/bailing. Where pumping is used, measure and record the pre-pumping water level. Operate the pump at a relatively constant rate. Measure the pumping rate using a calibrated container and stop watch, and record the pumping rate in the field log book. Measure and record the water level in the well at least once every 5 minutes during pumping. Note any relevant observations in terms of water color, visual level of turbidity, sheen, odors, etc. Pump or bail until termination criteria specified in the Site-Specific Field Implementation plan are reached. Note: the project-specific field implementation plan may also specify a maximum turbidity requirement for completion of development. Unless otherwise specified the maximum turbidity should be 50 NTUs or less. Record the total volume of water purged from the well.

12 While developing, take periodic water level measurements (at least one every five minutes) to determine if drawdown is occurring and record the measurements on the Well Development Log.

13 While developing, calculate the rate at which water is being removed from the well. Record the volume on the Well Development Log.

14 While developing, water is also periodically collected directly from the well or bailer discharge and readings taken of the indicator parameters: pH, specific conductance, and temperature. Development is considered complete when the indicator parameters have stabilized (i.e., three consecutive pH, specific conductance, and temperature readings are within tolerances specified in the project work plans or within 10% if not otherwise specified), the extracted water is clear and free

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of fine sediment and most importantly, when acceptable volume of water has been removed and/or a sufficient amount of surging has been performed.

15 In certain instances, for slow recharging wells, the parameters may not stabilize. In this case, well development is considered complete when minimal amounts of fine-grained sediments are recovered and acceptable volume of water has been removed.

16 If the well goes dry, stop pumping or bailing. Note the time that the well went dry. After allowing the well to recover, note the time and depth to water. Resume pumping or bailing when sufficient water has recharged the well.

17 Contain all development water in appropriate containers.

18 When complete, secure the lid back on the well.

19 Place disposable materials in plastic bags for appropriate disposal and decontaminate reusable, downhole pump components and/or bailer

8 WASTE MANAGEMENT

Materials generated during monitoring well installation and development will be placed in appropriate labeled containers and disposed of as described in the Work Plan/Field Implementation Plan or Field Sampling Plan.

9 DATA RECORDING AND MANAGEMENT

All well development activities should be documented on appropriate log forms as well as in a proper field notebook and/or PDA. Additionally, all documents (and photographs) should be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site; times of arrival and departure; significant weather conditions; timing of well development activities; development method(s); observations of purge water color, turbidity, odor, sheen, etc.; purge rate; and water levels before, during, and after pumping.

10 QUALITY ASSURANCE

All reused, non-disposable, downhole well development equipment should be cleaned in accordance with the procedures outlined in the project documents.

11 REFERENCES

American Society for Testing Materials (ASTM), Designation D5521-05. *Standard Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

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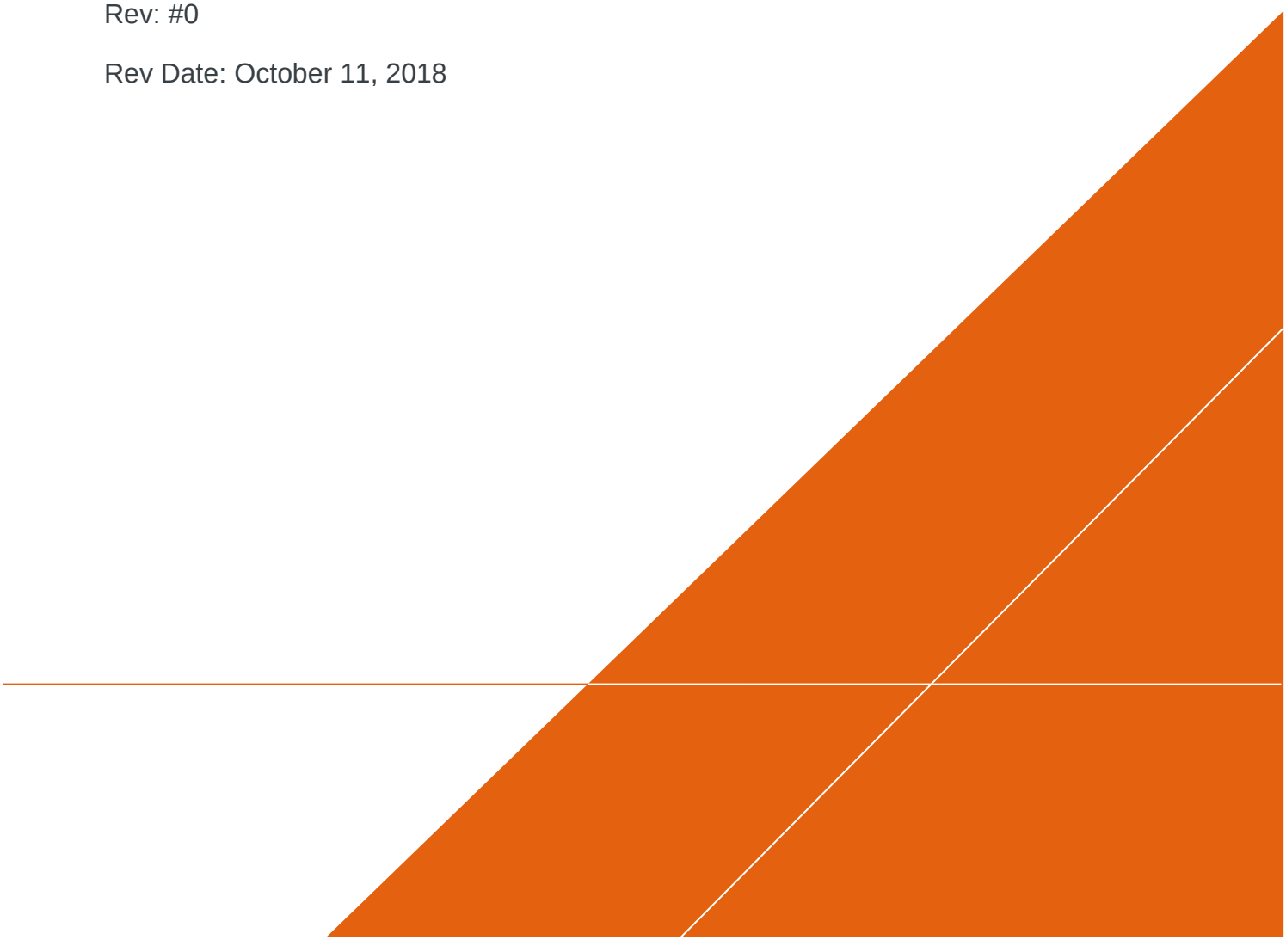
TGI – MANUAL WATER-LEVEL MONITORING

Rev: #0

Rev Date: October 11, 2018



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 11, 2018	All	Updated and re-written as TGI	Marc Killingstad Trey Fortner

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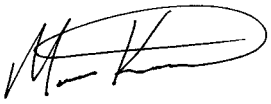
APPROVAL SIGNATURES

Prepared by:		10/11/2018
	Everett H. Fortner III, PG	Date:
Technical Expert Reviewed by:		10/11/2018
	Marc Killingstad (Technical Expert)	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

The objective of this Technical Guidance Instruction (TGI) is to describe procedures to measure and record water-levels (groundwater and surface-water) using manual water-level meters. Water levels may be measured using an electronic water-level probe or an oil-water level indicator from established reference points (e.g. top of casing). Reference points must be surveyed to evaluate fluid elevations relative to mean sea level (msl). This TGI describes the equipment, field procedures, materials, and documentation procedures to measure and record water-levels using the aforementioned equipment.

Surface water elevations can be determined from stilling wells or fixed points (bridges, walls, etc.) and measuring from an established point of reference using a water-level meter. In some cases, surface water elevations will be determined from a graduated stream gauge, attached to a pole located in open water with known elevation, without the use of a water-level meter.

The use of pressure transducers or other automated device for the collection of groundwater elevation data will be subject of *TGI – Water-Level Monitoring using Pressure Transducers and TGI – Water-Level Measurements using Sonic Meters*.

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3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements or access control requirements.

4 EQUIPMENT LIST

The following field equipment is required for water-level measurements:

- Site-specific Health and Safety Plan (HASP)
- Appropriate personal protective equipment (PPE) as specified in the HASP
- Electronic water-level indicator graduated in 0.01 ft. increments
- Electronic oil-water level indicator graduated in 0.01 ft. increments, if necessary
- Non-phosphate laboratory soap (Alconox or equivalent), brushes, clean buckets or clean wash tubs. New buckets or tubs should be purchased if it cannot be determined if the present items are clean
- Distilled or de-ionized (required for sites with metals) water for equipment decontamination
- Photoionization detector (PID) and/or organic vapor analyzer (optional)
- 150-foot measuring tape (or sufficient length for the maximum site depth requirement)
- Solvent (methanol/acetone) rinse - optional
- Plastic drop cloth (e.g. Weatherall Visqueen) to place beneath the buckets or tubs to reduce potential for contamination of the tape or probe
- Tools and/or keys required for opening wells
- Well construction summary table and/or well construction logs
- Summary table of previous water-level measurements
- Field notebook and/or smart device (phone or tablet) or appropriate field forms (see **Attachment 1**).
- Indelible ink pen

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5 CAUTIONS

Electronic water-level probes and oil-water interface probes can sometimes produce false-positive readings. For example, if the inside surface of the well or stilling tube has condensation above the water level, then an electronic water-level probe may produce a signal by contacting the side of the well rather than the true water level in the well. For accuracy, the electronic water-level probe and/or interface probe should be raised and lowered several times at the approximate depth where the instrument produces a tone indicating a fluid interface to verify consistent, repeatable results. Additionally, some wells may be constructed with a sump. If local/regional groundwater levels have declined such that the water table is below the base of the well screen, a sump may still contain water and provide an erroneous measurement. Therefore, possessing and checking against either a well construction summary table or well construction log is vital for proper measurements and reporting.

If the presence of a non-aqueous phase liquid (NAPL) is known or suspected at the site or within specific wells, *do not use* an electronic water-level indicator. Use an oil-water interface meter instead.

When measuring well depths with an electronic water-level indicator, staff performing the measurement must measure and add the length of the probe beneath the circuit closing electrodes (if applicable to the instrument) to the depth measured to obtain the true depth. This is necessary because the tape distance markings are referenced to the electrodes, rather than the end of the probe. Some newer instruments do not have offset electrodes and this correction would not be needed.

If the depth markings on the tape or cable are worn or otherwise difficult to read, extra care must be taken in obtaining the depth readings.

Ensure that the type of electronic water-level probe is compatible with the depth and diameter of the wells to be measured. Some smaller piezometers or production well stilling tubes will accommodate only smaller diameter probes.

Tapes missing length (too short) or have been repaired should be discarded because the accuracy of the tape is lost.

6 HEALTH AND SAFETY CONSIDERATIONS

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or oil. Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well head), and potentially the use of sharp cutting tools (scissors, knife). Appropriate personal protective equipment (PPE) will be worn during these activities. Only use non-toxic peppermint oil spray for stinging insect nests. Open well caps slowly and keep face and body away to allow to vent any built-up pressure. Field personnel will thoroughly review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives.

Obtaining measurements from active pumping wells requires knowledge of the construction and design as the indicator probe and tape can be intertwined within the pump propellers causing a serious health and safety hazard and equipment damage. Ensure that stilling wells have a perforated end and capped bottom to inhibit tape from extending into the downhole pump depth. If a stilling tube is not present or the

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still tube construction is not known, determine a conservative “not to exceed” measurement depth based on the top of pump depth with an added safety factor. If all information is not known, a water-level should not be taken from the pumping well until clarification on depths are available.

7 PROCEDURE

Calibration procedures and groundwater level measurement procedures for electronic water-level indicators and oil-water indicators are described in the sections below.

Calibration Procedures

The indicator probe must be tested to verify that the meter has been correctly calibrated by the manufacturer. The following steps will be used to verify the accuracy of the indicator:

- Measure the lengths between each increment marker on the indicator with a measuring tape. The appropriate length of indicator measuring tape, suitable to cover the depth range for the wells of interest, will be checked for accuracy.
- If the indicator measuring tape is inaccurate, the probe should be sent back to the manufacturer or rental company. If a replacement can’t immediately be available, then an offset can be measured to correct the measurements.
- If multiple water level meters are being used for an event, calibration of the multiple devices should occur by taking a water level at a single well contemporaneously with all indicators to be used and determining appropriate corrections (normally corrections are small ranging from 0.01 to 0.03 feet).
- Equipment calibration will be recorded in the field logbook and/or smart device.

Water-Level Measurement Procedures

The general procedures to be followed for the collection of groundwater level measurements and well depths from the monitoring wells are as follows:

- Check that the water-level indicator battery is functional, before leaving for site and prior to each day onsite (e.g. turn power on and check that meter sounds when probe is lowered into a bucket of water – note that water-level meters will not work with distilled water).
- Record instrument make, model, serial number and (if present) Arcadis ID number in the field book.
- Don disposable nitrile gloves. Decontaminate the water-level indicator, any attached tape and the spool with laboratory-grade soap and distilled water (see *Initial Decontamination Procedures* below). The spool requires extra care because it may not be water-proof and the electrical parts inside may be damaged during cleaning.
- The top of the monitoring well will be cleaned with a clean rag to prevent loose particulate matter from falling into the well.
- Perform a well inspection (note that for some sites a well inspection form is required to be filled out along with a photo to document the conditions).
- Place clean plastic sheeting on the ground next to the well.

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- Unlock and/or open the monitoring well cover while standing upwind from the well (note that some wells may be under pressure and precaution should be taken with opening well caps
- Measure the volatile organics present in the monitoring well head space with a PID and record the PID reading in the field logbook (if applicable and requirement for the site).
- Allow the water-level in the well to equilibrate with atmospheric pressure for a few minutes (check previous field forms/logs for equilibration time if noted).
- Locate the measuring reference point that correlates to the survey point on the monitoring well casing. If one is not found, create a reference point by notching the highest and/or north point on the inner casing (or outer if an inner casing is not present) with a hacksaw. All downhole measurements will be taken from the reference point. Document the creation of any new reference point or alteration of the existing reference point.
- Measure to the nearest 0.01 of a foot and record the height of the inner well casing and outer protective casing to ground level (note that some well pads are raised and are not at true ground surface).
- Lower the indicator probe into the center of the well until a contact with the water surface is indicated by either audible alarm or light. Use and install tape guide (Solinst products) to help with accuracy and provide protection with damaging the measurement tape. If a tape guide is not available, make sure that the tape does not drag on the inner or outer casing which could fray and damage the tape.
- Hold the tape at the measuring point (tip of well casing) and repeat the measurement two more times.
- Read and record measurement to the nearest 0.01 ft. Check against previous measurements and note any anomalies/discrepancies; if significant, contact technical lead for project.
- Measure and record total depth of well (see *Total Depth Measurement Procedures* below).
- Record all measurements (**with date and time collected**) and note any inconsistencies/anomalies and relevant observations in the field notebook and/or smart device or appropriate field forms.
- Follow decontamination procedure outlined below before gauging the next well (see Decontamination after *Water Level and Total Depth Measurements* below).
- Lock the well when all activities are completed.

Total Depth Measurement Procedures

- Weighted tape or electronic water-level indicator can be used to determine the total well depth.
- Follow initial procedures noted above in *Water-Level Measurements* above.
- Lower indicator probe (or tape) until weighted end is felt resting on the bottom of the well. Raise indicator slowly until there is no slack in the tape. Gently "feel" the bottom of the well by slowly raising and lowering the indicator: great care should be taken to avoid damaging the sensor on the probe. The operator may find it easier to allow the weight to touch bottom and then detect the ‘tug’ on the tape while lifting the weight off the well bottom.

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- Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the tape end is touching the bottom of the well and sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements.
- If total depth measurements will be collected during low-flow sampling events, "Bottom of well" measurements will be made only after low-flow sampling has been completed or at least 12 hours prior to initiating sample collection from the well, in order to minimize: 1) mixing of the stagnant water at the top of the well column with potential formation water underneath; and/or 2) agitation and subsequent entrainment of possible sediment collected at the well bottom).
- Read and record measurement to the nearest 0.1 ft. Please refer to the note regarding total depth measurements described in *Section 5 Cautions* above.
- Follow decontamination procedure outlined below before gauging the next well (see Decontamination after *Water Level and Total Depth Measurements* below).

Initial Decontamination

- Set up a decontamination station consisting of three **clean** buckets (e.g. 5-gallon buckets). The buckets should not be used to containerize purge water; they will be used for decontamination purposes only.
- Fill the first bucket with one gallon of distilled water (use deionized water if metals are a contaminant at the site) and add non-phosphate laboratory-grade soap. Fill the second bucket with distilled water (use deionized water if metals are a contaminant at the site) and leave the third bucket empty. Place the drop cloth underneath.
- Unwind the entire tape from the spool into a bucket with non-phosphate laboratory-grade soap and distilled water; Brush the tape carefully to remove dirt and possible contamination, using a brush dedicated to the wash bucket.
- Carefully brush all dirt of the spool and wipe down with a soapy cloth or paper towel.
- Transfer the tape into the second bucket containing rinse water. Carefully brush the tape using a second brush, dedicated to the rinse bucket. Lift the tape out of the bucket and allow rinse water to drip off the tape.
- Transfer the tape to the third bucket. Wind the tape onto the spool while wiping excess water off the tape using a paper towel.

Decontamination after Water Level and Total Depth Measurements

- Set up a decontamination station consisting of three **clean** buckets, fill according to the initial decon procedure.
- Unwind the only the length of tape used for gauging from the spool into a bucket with laboratory-grade soap and distilled water. Brush the tape carefully to remove dirt and possible contamination, using a brush dedicated to the wash bucket.
- Continue as described above.

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- Extra care should be taken to clean the probe after a total depth measurement. All sediment or dirt needs to be removed during decontamination.

Notes:

- Collect decon blanks per work plan (minimum 1 per 20 samples or 1 per sampling event)
- Prepare new wash solution and rinse water when necessary but at least every 10 to 20 wells. The spend wash and rinse solution should be discharged according to site requirements.
- The decontamination station can be expanded by adding extra rinse and/or detergent stations (i.e. solvent wash station) to the set up. The addition of more stations depends on the requirements of the work plan or the site-specific Field Sampling and Quality Assurance Plan and need to be discussed in the project kick-off meeting.
- Small crates or washtubs are a possible substitute for the buckets. In any case, it is recommended to use containers with a lid.

8 WASTE MANAGEMENT

Decontamination fluids, PPE, and other disposable equipment will be properly stored on site in labeled containers and disposed of properly. Be certain that waste containers are properly labeled and documented in the field log book. Review *TGI – Investigation Derived Waste Handling and Storage*, for additional information and state- or client-specific requirements.

9 DATA RECORDING AND MANAGEMENT

Groundwater level measurements as well as all relevant observations should be documented in the field logbook, field forms and/or PDA as appropriate. The following information must be documented:

- Well or location identification;
- Measurement time;
- Total well depth or depth of the water body at the location;
- Depth to water and, where necessary, depth to product.

Once all the data has been collected and recorded, all notes/forms/data must be uploaded to the appropriate project directory on the Arcadis server, and an email should be sent to the Task Manager and/or Technical Lead for notification. A summary of the work completed that day and any relevant observations noted (such as well inspections) during the daily activities as well as copies of the data mentioned above should be included with the email. The appropriate team member will review the data for accuracy and provide feedback.

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10 QUALITY ASSURANCE

- As described in the detailed procedure, the electronic water-level meter and/or oil-water interface probe will be calibrated prior to use versus an engineer's rule to ensure accurate length demarcations on the tape or cable. The results will be recorded.
- Measurements will be conducted three times and the final measurement will be recorded.
- Fluid interface measurements will be verified by gently raising and lowering the instrument through each interface to confirm repeatable results.
- Field notes will be reviewed by the project team once the field data has been delivered.

11 REFERENCES

Cunningham, W.L., and Schalk, C.W., comps., 2011. *Groundwater technical procedures of the U.S. Geological Survey: U.S. Geological Survey Techniques and Methods 1–A1, 151 pp*.

U.S. Environmental Protection Agency, 2013. *SESD Operating Procedure, Groundwater level and Well Depth Measurement.* January 29.

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ATTACHMENT A

Water-Level Measurement Form

[Ctrl+Click on image to open form in Excel]

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Water-Level Measurement Form

Project No.: _____ Field Personnel: _____
Site Location: _____ Date: _____
Instrument Model: _____ Instrument Serial No.: _____

Well Number	Time	W.L. Measurements TD (feet)	DTW (feet)	DTP (feet)	Comments Well Locked	Lock Condition	Other Comments

W.L. Water Level
TD Total Depth
DTW Depth To Water
DTP Depth To Product

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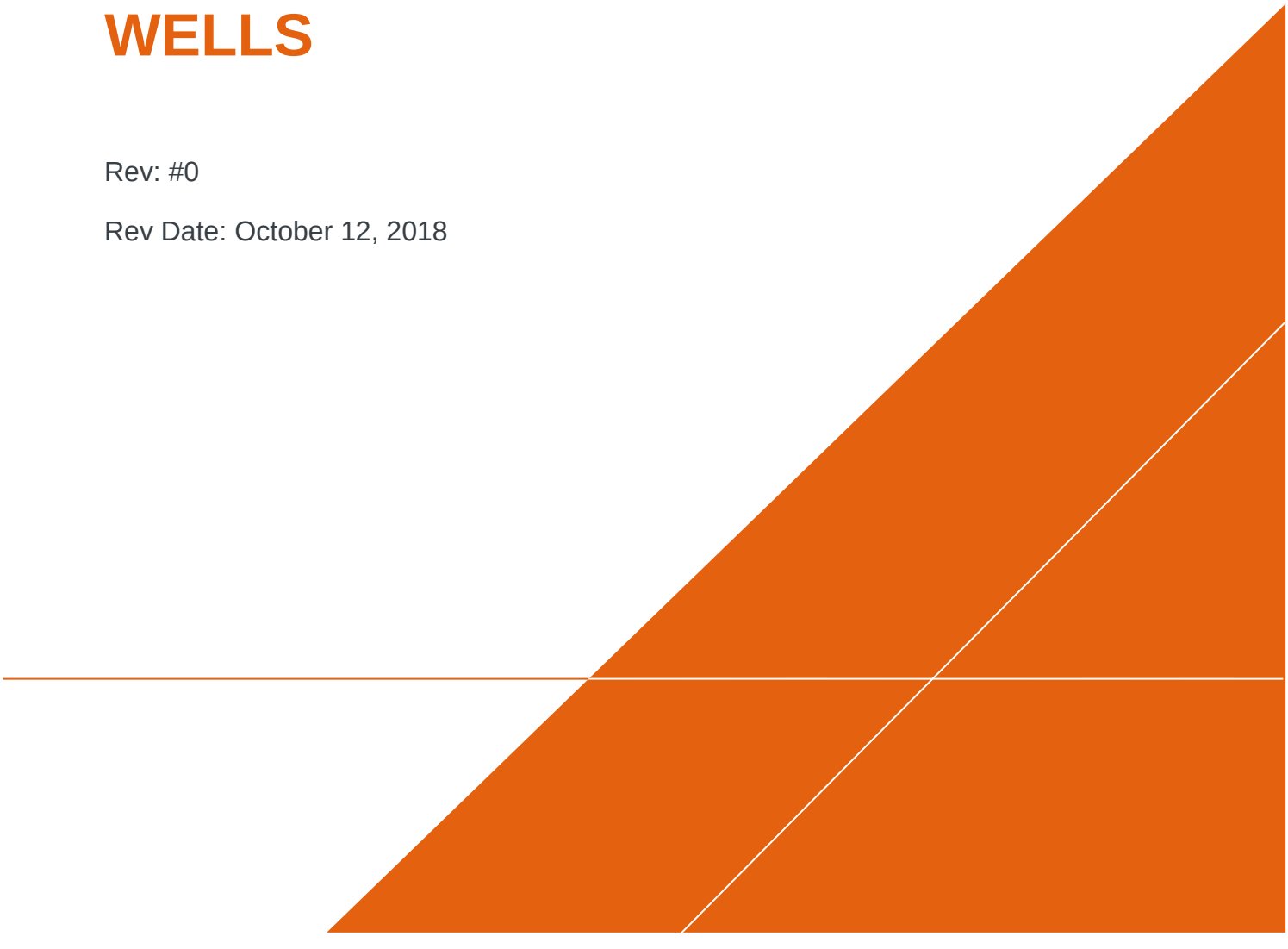
TGI - LOW-FLOW GROUNDWATER PURGING AND SAMPLING PROCEDURES FOR MONITORING WELLS

Rev: #0

Rev Date: October 12, 2018



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 12, 2018	All	Updated and re-written as TGI with new branding and content	Marc Killingstad

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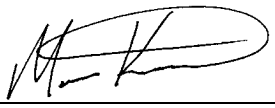
APPROVAL SIGNATURES

Prepared by:		10/12/2018
	Ryan McKinney	Date:
Technical Expert Reviewed by:		10/12/2018
	Marc Killingstad (Technical Expert)	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

Groundwater samples are collected from monitoring wells to evaluate groundwater quality. The protocol presented in this Technical Guidance Instruction (TGI) describes the procedures to purge monitoring wells and collect groundwater samples using the low flow purging/sampling methodology. This protocol has been developed in accordance with the United States Environmental Protection Agency (USEPA) Region I *Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells* (EQASOP-GW4; September 19, 2017).

Both filtered and unfiltered groundwater samples may be collected using this low-flow sampling method. Filtered samples will be obtained using a 0.45-micron disposable filter. Project teams will evaluate the last time the monitoring wells were developed and determine if additional development might be necessary. Water samples will not be taken immediately following well development. Sufficient time will be allowed for the groundwater flow regime in the vicinity of the monitoring well to stabilize and to approach chemical equilibrium with the well construction materials. This lag time will depend on site conditions and methods of installation but often exceeds one week.

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3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the groundwater sampling team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the groundwater sampling team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

4 EQUIPMENT LIST

Specific to this activity, the following materials (or equivalent) will be used:

- Site-specific HASP and health and safety documents identified in the HASP
- Field Implementation Plan (FIP) that includes site map, well construction records, sampling plan (sample analyses, sample volume required, and sample holding time), and prior groundwater sampling records (if available)
- Field notebook and/or smart device (phone or tablet)
- Low-flow sampling field forms (**Attachment A**)
- Appropriate personal protective equipment (PPE) (e.g., latex or nitrile gloves, safety glasses, etc.) as specified in the HASP
- Well keys and other tools to remove manhole covers (manual torque wrench with 9/16” socket and flat head screwdriver typical)
- Photoionization detector (PID) or Flame ionization detector (FID) (as appropriate, depending on site-specific constituents of concern)
- Electronic water-level indicator (e.g., Solinist Model 101) or oil/water interface probe with 0.01-foot accuracy (oil/water as appropriate, note that sampling will not be performed when sheen or light non-aqueous phase liquid [LNAPL] is present)
- Down-hole multiparameter water-quality sonde (temperature/pH/specific conductivity/oxidation reduction [ORP]/turbidity/dissolved oxygen) meter and flow-through measurement cell; for example:

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    - YSI 6-Series Multi-Parameter Instrument
    - Horiba U-22 Multi-Parameter Instrument.
    - Hydrolab Series 3 or Series 4a Multiprobe and Display.
- Plastic sheeting (e.g., Weatherall Visqueen) to protect all down-hole sampling equipment from contact with potential sources of contamination.
- Decontamination equipment
    - Non-phosphate laboratory soap (Alconox or equivalent), brushes, clean buckets or clean wash tubs—new buckets or tubs will be purchased if it cannot be determined if the present items are clean
    - Distilled or de-ionized water for equipment decontamination
- Indelible ink pen
- 150-foot measuring tape (or sufficient length for the maximum site depth requirement)
- Sampling pump, which may consist of one or more of the following:
    - Submersible pump (e.g., Grundfos Redi-Flo 2)
    - Peristaltic pump (e.g., ISCO Model 150)
    - Bladder pump (e.g., Marschalk System 1, QED Micropurge, Geotech)
- Appropriate controller and power source for pump:
    - Submersible and peristaltic pumps require electric power from either a generator or a deep cell battery
    - Submersible pumps such as Grundfos require a pump controller to run the pump
    - Bladder pumps require a pump controller and a gas source (e.g., air compressor or compressed N2 or CO2 gas cylinders)
- Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being used
    - For peristaltic pumps, dedicated Tygon® tubing (or other type as specified by the manufacturer) will be used through the pump apparatus
    - Teflon® will not be used when sampling for per- and polyfluoroalkyl substances (PFAS)
- Graduated cylinder and stop watch or other device to measure time to determine pumping rate
- Appropriate water sample containers (supplied by the laboratory)
- Appropriate blanks (trip blank supplied by the laboratory)
- Sample labels and Chain-of-Custody forms (COC)
- 0.45-micron disposable filters (if field filtering is required)

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- A supplemental turbidity meter (e.g., Horiba U-10, Hach 2100P, LaMotte 2020) may be required for specific projects and will be specified in the project FIP/ work plan and the kick-off notes.
    - If used, in-line 'T' and valve allows for collection of water for turbidity measurements before the pump discharge enters the flow-through cell

NOTE: *The maintenance requirements for the above equipment generally involve decontamination or periodic cleaning, battery charging, and proper storage, as specified by the manufacturer. For operational difficulties, the equipment will be serviced by a qualified technician.*

5 CAUTIONS

Different USEPA regions and/or state regulatory agencies may stipulate deviations from this document. It is the responsibility of the Project Team (Project Manager and Technical Lead) to be fully aware of the requirements from the applicable regulatory framework.

Weather

- If heavy precipitation occurs, and no cover over the sampling area and monitoring well can be erected, sampling may be discontinued until adequate cover is provided. Rain water could compromise groundwater samples.
- Avoid extreme weather situations. Be aware that thermal currents and vertical mixing of cold and warm water inside the well casing could create a convection cell within the well and compromise data collection (e.g., biological mechanisms).
    - Direct sunlight and hot ambient temperatures may cause the groundwater in the tubing or flow-through-cell to heat up and de-gas. This may result in the loss of volatile organic compounds (VOCs) and dissolved gases. Shade the equipment from direct sunlight, keep the tubing as short as possible, and avoid the hottest times of the day.
    - Sampling during freezing conditions may adversely impact the data quality objectives. USEPA recommends low-flow sampling be conducted at air temperatures above 32°F (0°C) or taking special precautions to prevent groundwater from freezing in the equipment.

Cross-Contamination

- To mitigate potential cross-contamination, groundwater samples are to be collected in a pre-determined order from least impacted to impacted based on previous analytical data. If no analytical data are available, collect samples in order of up-gradient, then furthest down-gradient to source area locations.
- Note that permanent markers could introduce volatile constituents into the samples; *therefore, indelible ink is recommended* to be used for labels on sample containers or sample coolers.
- When using a gasoline generator, this power source will be set-up at least 30 feet downwind from the well to avoid exhaust fumes to contaminate samples.

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Pumps

- Preferred methods of extracting groundwater are adjustable rate, submersible pumps - such as centrifugal pumps or bladder pumps – constructed of stainless steel or polytetrafluoroethylene (PTFE, i.e. Teflon®). However, *PTFE will not be used when sampling for per- and polyfluoroalkyl substances (PFAS). PTFE could contain PFAS*.
- When using a bladder pump for collecting VOCs and dissolved gases, “best practice” is to set-up the pump to deliver sufficient water to fill a 40 mL VOC vial.
- The use of peristaltic pumps will be based on the type of data to be collected. *Because the use a peristaltic pump can result in de-gassing of VOC and / or dissolved gases from groundwater, a different type of pump will be considered if these compounds are of concern.*
- *Manual or motor driven inertial pumping devices are not recommended because they cause greater disturbance during purging and pumping than regular pumps and are less easily controlled. This could cause a higher degree of data variability.*

Tubing

- When sampling for VOCs, SVOCs, pesticides, PCBs and inorganics, use of PTFE (Teflon®) or PTFE-lined tubing is preferred. However, PTFE tubing will not be used when sampling for PFAS.
- PVC, polypropylene or polyethelene tubing may be used when sampling for metals or other inorganics.
- Tubing with inside diameters of 1/4 or 3/8 inch is recommended because this will help ensure tubing remains water filled when operating at very low pumping rates.

General Precautions

- Store and/or stage empty and full sample containers and coolers out of direct sunlight.
- It may be necessary to field filter the groundwater for some parameters (e.g., metals) during collection, depending on preservation, analytical method, and project quality objectives. The task-kick-off notes and the FIP/work plan will list the samples that require field filtering.
- Be careful not to overtighten lids with Teflon® liners or septa (e.g., 40 mL vials). Over-tightening can cause the glass to shatter or impair the integrity of the Teflon® seal.

6 HEALTH AND SAFETY CONSIDERATIONS

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or non-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include pressurized wells, stinging

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insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well head), and potentially the use of sharp cutting tools (scissors, knife)—open well caps slowly and keep face and body away to allow to vent any built-up pressure; only use non-toxic peppermint oil spray for stinging insect nests; review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives, and use appropriate hand protection.

Generators and cord and plug equipment will employ an overcurrent protection device such as an integrated ground fault circuit interrupter (GFCI) cord. Grundfos pump controllers will not run properly with a GFCI, so the power source will be equipped with other overcurrent protection means.

Overtightening of lids with Teflon® liners can cause the glass to shatter and create a risk for hand injuries.

7 PROCEDURE

Field personnel will set up and perform low-flow sampling in accordance with the following procedures.

1. Review FIP and groundwater sampling records from previous sampling events (if available) prior to mobilization to estimate the optimum pumping rate and anticipated drawdown for each well to perform sampling as efficiently as possible (i.e., reach a stabilized pumping condition)
2. Calibrate field instruments according to manufacturer procedures for calibration and record calibration procedure and results in field log
3. All equipment will either be new or decontaminated in accordance with appropriate guidance document (*TGI – Groundwater and Soil Sampling Equipment Decontamination)* prior to use
4. Visually inspect the well to ensure that it is undamaged, properly labeled and secured
    a) Damage or other conditions that may affect the integrity of the well will be recorded in the Field Activity Daily Log and brought to the attention of the designated Field Manager and/or Project Manager
    b) Record well construction and conditions on the Low-Flow Sampling Field Form (**Attachment A**)
5. Place clean plastic sheeting on the ground near the well to keep monitoring and sampling equipment off the surface unless the equipment is elevated above the ground (e.g. on a table).
6. Open the well cover while standing upwind of the well. Remove the well cap and place it on the plastic sheeting. If appropriate or required for site-specific conditions, insert the photoionization detector (PID) probe approximately 4 to 6 inches into the casing or the well headspace and cover it with a gloved hand. Record the PID reading in the field log. Perform air monitoring in the breathing zone according to the HASP and/or JSA.
7. Measure and record the initial depth to groundwater prior to placing the pumps
8. Prepare and install the pump in the well

NOTE: *Groundwater will be purged from the wells using an appropriate pump. If the depth to water is below the sampling range of a peristaltic pump (approximately 25 feet below ground surface), a*

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submersible or bladder pump will be used, provided that the well is constructed with a casing diameter of at least two (2) inches (the minimum well diameter capable of accommodating such pumps). For smaller diameter wells, where the depth to water is below the sampling range of a peristaltic pump, alternative sampling methods (i.e., bailing or small diameter bladder pumps) will be used to purge and sample the groundwater. Bladder pumps are preferred over peristaltic and submersible pumps to prevent volatilization if sampling of VOCs and/or dissolved gasses is required. Purge water will be collected and containerized according to the direction of the project team.

a) For submersible and non-dedicated bladder pumps, decontaminate the pump according to site decontamination procedures. Non-dedicated bladder pumps will require a new bladder and attachment of an air-line, sample discharge line, and safety cable prior to placement in the well. Attach the air-line tubing to the air-port on the top of the bladder pump. Attach the sample discharge tubing to the water port on the top of the bladder pump. Take care not to reverse the air and discharge tubing lines during bladder pump setup, as this could result in bladder failure or rupture. Attach and secure a safety cable to the eyebolt on the top of pump (if present, depending on pump model used). Slowly lower the pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the approximate center of the saturated screen section of the well. Avoid twisting and tangling of safety cable, tubing, and electrical lines while lowering the pump into the well; twisted and tangled lines could result in the pump becoming stuck in the well casing. Also, make sure to keep tubing and lines from touching the ground or other surfaces while introducing them into the well, as this could lead to unintended contamination.

b) If using a bladder pump, connect the air-line to the pump controller output port. The pump controller will be connected to a supply line from an air compressor or compressed gas cylinder using an appropriate regulator and air hose. Tighten the regulator connector onto the gas cylinder (if used) to prevent leaks. Teflon® tape may be used on the threads of the cylinder to provide a tighter seal. Once the air compressor or gas cylinder is connected to the pump controller, turn on the compressor or open the valve on the cylinder to begin the gas flow. Turn on the pump controller power (if an on/off switch is present) and verify that all batteries are charged and fully functioning before starting the pump.

c) If a peristaltic pump is being used, slowly lower the sampling tubing into the well to a depth corresponding to the approximate center of the saturated screen section of the well. The pump intake or sampling tube must be kept at least two (2) feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.

d) If using an in-line ‘T’ and valve, install between pump discharge water line and the bottom inlet port of the flow-through cell. Attach a short piece of tubing to the outlet. This set-up will be used to collect samples for turbidity readings.

9. Connect the pump discharge water line to the bottom inlet port on the flow-through cell connected to the water quality meter.

10. Before starting the pump, ensure that the water level inside the well has stabilized (i.e., measure the water level multiple times after deploying the pump in the well)

11. Start pumping the well at 200 to 500 milliliters (mL) per minute (or at lower site-specific rate if specified) and adjust the pumping rate to cause little or no water level drawdown in the well (less

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than 0.3 feet below the initial static depth to water measurement): the water level should stabilize, however, this is not always possible.

12. If the well diameter is of sufficient size, measure the water level every 3 to 5 minutes (or as appropriate, lower flow rates may require longer time between readings) during pumping

13. Maintain a steady flow rate to the extent practicable and do not break pump suction or cause entrainment of air in the sample

14. Record pumping rate adjustments and depths to water

    If necessary, reduce pumping rates to the minimum capabilities of the pump to avoid pumping the well dry and/or to stabilize indicator parameters; if the recharge rate of the well is very low, use alternative purging techniques, which will vary based on the well construction and screen position:

    For wells screened across the water table, the well may be pumped dry and sampling can commence as soon as the volume in the well has recovered sufficiently to permit collection of samples

    For wells screened entirely below the water table, the well can be pumped until a stabilized level (which may be greater than the maximum displacement goal of 0.3 feet) is maintained and monitoring for stabilization of field indicator parameters can commence; if a lower stabilization level cannot be maintained, the well may be pumped until the drawdown is at a level slightly higher than top of the well screen

15. The sampling process may commence after one well volume has been removed and the well has recovered sufficiently to permit collection of samples

16. After water levels have stabilized and a sufficient volume has been purged, continue pumping and begin monitoring field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, ORP, and dissolved oxygen [DO]) every 3 to 5 minutes (or after each volume of the flow-through cell has been purged or other appropriate interval) using a flow-through analytical cell or a clean container such as a glass beaker and collect samples for turbidity readings using the T-valve

17. Record field indicator parameters on the groundwater sampling log

18. The well is considered stabilized and ready for sample collection when three consecutive readings are within the following limits:

    - **Turbidity** within ± 10% for values greater than 5 nephelometric turbidity unit [NTU] or if three turbidity values are less than 5 NTU, consider the values as stabilized;
    - **Dissolved Oxygen (DO)** within ± 10% for values greater than 0.5 mg/L or if three DO values are less than 0.5 mg/L, consider the values as stabilized;
    - **Specific Conductance** within ± 3%;
    - **Temperature** within ± 3%;
    - **pH** within ± 0.1 unit;
    - **Oxidation/Reduction Potential (ORP)** within ±10 millivolts (mV);

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NOTE: *Alternate stabilization goals may exist in different geographic regions, consult the site-specific FIP/work plan for stabilization criteria).*

NOTE: *If the parameters have stabilized but turbidity remains relatively high (e.g., greater than 50 NTU), the pump flow rate may be decreased to a minimum rate of 100 mL/min to reduce turbidity levels as low as possible.*

NOTE: *If field indicator parameters do not stabilize within one (1) hour of the start of purging, but the groundwater turbidity has been minimized (i.e., within ± 10%) and the values for all other parameters are within ± 10%, the well may be sampled; however, consult specifications in the FIP/work plan and/or the project technical lead.*

NOTE: *DO is extremely susceptible to various external influences (including temperature or the presence of bubbles on the DO meter); therefore, great care will be taken to minimize the agitation or other disturbance of water within the flow-through cell while collecting these measurements. If air bubbles are present on the DO probe or in the discharge tubing, remove them before taking a measurement. If DO values are not within acceptable range for the temperature of groundwater, again check for and remove air bubbles on the probe before re-measuring. The table below may be used as a general guide for DO values under various temperatures; however, understand that groundwater contaminants may affect oxygen solubility. If DO value is 0.00 or less, then the meter will be serviced and re-calibrated. If DO values are above possible results, then the meter will be serviced and re-calibrated.*

NOTE: *During extreme weather conditions, stabilization of field indicator parameters may be difficult to attain. Modifications to the sampling procedures to alleviate these conditions (e.g., measuring the water temperature in the well adjacent to the pump intake) will be documented in the field logbook/on the sampling form.*

NOTE: *If other field conditions are suspected of preventing stabilization of certain parameters, observations will be documented in the field logbook/on the sampling form.*

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Oxygen Solubility in Fresh Water

Temperature (degrees C)	Dissolved Oxygen (mg/L)
0	14.6
1	14.19
2	13.81
3	13.44
4	13.09
5	12.75
6	12.43
7	12.12
8	11.83
9	11.55
10	11.27
11	11.01
12	10.76
13	10.52
14	10.29
15	10.07
16	9.85
17	9.65
18	9.45
19	9.26
20	9.07
21	8.9
22	8.72
23	8.56
24	8.4
25	8.24
26	8.09
27	7.95
28	7.81
29	7.67
30	7.54
31	7.41
32	7.28
33	7.16
34	7.05
35	6.93

Reference: Vesilind, P.A., *Introduction to Environmental Engineering*, PWS Publishing Company, Boston, 468 pages (1996).

19. Complete the sample label(s) and cover the label(s) with clear packing tape to secure the label onto the container
20. After the indicator parameters have stabilized, collect groundwater samples by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container
    a) If a flow-through analytical cell is being used to measure field parameters, the flow-through cell will be disconnected after stabilization of the field indicator parameters and prior to groundwater sample collection
    b) Under no circumstances will analytical samples be collected from the discharge of the flow-through cell

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c) If an in-line 'T' and valve are used, the valve needs to be removed as well

d) Samples will be collected in the following order: VOCs, total organic carbon (TOC), semi-volatile organic compounds (SVOCs), metals and cyanide, and others (or other order as defined in the site-specific FIP/work plan)

e) When the container is full, tightly screw on the cap

21. If sampling for total and filtered metals and/or polychlorinated biphenyls (PCBs), a filtered and unfiltered sample will be collected

    a) Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected

    b) Continue to run the pump until an initial volume of "flush" water has been run through the filter in accordance with the manufacturer's directions (generally 100 to 300 mL)

    c) Collect the filtered groundwater sample by diverting flow out of the filter into the appropriately labeled sample container

    d) When the container is full, tightly screw on the cap

22. Secure with packing material and store the samples on ice (targeting 4° Celsius) in an insulated transport container provided by the laboratory and include a temperature blank in each container to be shipped

23. Record on the Low-Flow Sampling Field Form (and bound field logbook) the time at which sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance and the presence or lack of odors or sheens), and the values of the stabilized field indicator parameters as measured during the final reading during purging (see **Attachment A**)

24. Turn off the pump and air compressor or close the gas cylinder valve if using a bladder pump setup

25. Slowly remove the pump, tubing, lines, and safety cable from the well

    a) If using dedicated tubing, do not allow the tubing or lines to touch the ground or any other surfaces which could contaminate them

    b) If using dedicated tubing, it will be folded - without pinching it - to a length that will allow the well to be capped and also facilitate retrieval of the tubing during later sampling events.

    c) Use a length of rope or string to tie the tubing to the well cap

    d) Alternatively, if tubing and safety line are to be saved and reused for sampling the well at a later date, coil the tubing neatly and placed in a clean plastic bag that is clearly labeled with the well ID ensuring the bag is tightly sealed before placing it in storage

26. Secure the well and properly dispose of personal protective equipment (PPE) and disposable equipment

27. Complete the procedures for packaging, shipping, and handling with the associated Chain-of-Custody

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28. Complete decontamination for flow-through analytical cell and submersible or bladder pump, as appropriate (*TGI – Groundwater and Soil Sampling Equipment Decontamination*)
29. At the end of each day of the sampling event, perform calibration check of field instruments and record procedure and results in field log

8 WASTE MANAGEMENT

Materials generated during groundwater sampling activities, including disposable equipment and excess purge water, will be stored on site in appropriately labeled containers and disposed of properly. Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log book.

9 DATA RECORDING AND MANAGEMENT

Management of the original documents from the field will be completed in accordance with the site-specific QAPP.

In general, forms (e.g., Low-Flow Sampling Field Forms), logs/notes (including daily field and calibration logs), digital records, and Chain-of-Custody records will be maintained by the field team lead.

Field logs and Chain-of-Custody records will be transmitted to the Arcadis Project Manager and/or Task Manager, as appropriate, at the end of each day unless otherwise directed. Electronic data files will be sent to the project team and uploaded to the electronic project folder daily.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

10 QUALITY ASSURANCE

Quality assurance procedures will be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

Unless described otherwise in the project-specific FIP/work plan, QAPP, or Sampling and Analysis Plan, quality assurance/quality control samples will be collected as follows:

- One duplicate for every 10 samples
- One laboratory matrix/matrix spike sample for every 20 samples

In addition to the quality control samples to be collected in accordance with this TGI, the following quality control procedures will be observed in the field:

- Collect samples from monitoring wells, in order of increasing concentration, to the extent known based on review of historical site information if available
- Equipment blanks will include the pump and tubing (if using disposable tubing) or the pump only (if using tubing dedicated to each well)

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- Collect equipment blanks after wells with higher concentrations (if known) have been sampled
- Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures—calibrate instruments at the beginning of each day, verify the calibration at the end of each day, and record all calibration activities in the field notebook
- Clean all groundwater sampling equipment prior to use in the first well and after each subsequent well following the procedure for equipment decontamination

11 REFERENCES

USEPA. 1986. *RCRA Groundwater Monitoring Technical Enforcement Guidance Document* (September 1986).

USEPA. 1991. *Handbook Groundwater, Volume II Methodology*, Office of Research and Development, Washington, DC. USEPN62S, /6-90/016b (July 1991).

USEPA Region I. 2017. *Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells* (EQASOP-GW4; September 19, 2017).

U.S. Geological Survey (USGS). 1977. *National Handbook of Recommended Methods for Water-Data Acquisition: USGS Office of Water Data Coordination*. Reston, Virginia.

12 ATTACHMENTS

Attachment A - Low-Flow Sampling Field Form

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ATTACHMENT

Attachment A - Low-Flow Sampling Field Form

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LOW-FLOW GROUNDWATER SAMPLING FORM

ARCADIS

Project No. _____ Well ID _____ Date _____

Project Name/Location _____ Weather _____

Measuring Pt. Description _____ Screen Setting (ft-bmp) _____ Casing Diameter (in.) _____ Well Material ____ PVC ____ SS

Static Water Level (ft-bmp) _____ Total Depth (ft-bmp) _____ Water Column (ft) _____ Gallons in Well _____

MP Elevation _____ Pump Intake (ft-bmp) _____ Purge Method: _____ Sample Method _____

Pump On/Off _____ Volumes Purged _____ Centrifugal _____ Submersible _____ Other _____

Sample Time: Label _____ Gallons Purged _____

Purge Start _____ Replicate/

Purge End _____ Code No. _____ Sampled by _____

Time	Minutes Elapsed	Rate (gpm)/(mL/min) 200mL/min +	Depth to Water (ft) -0.3	Gallons Purged	pH ± 0.1	Cond. (µMhos)/(mS/cm) ± 3%	Turbidity (NTU) ± 10%	DO (mg/L) ± 10%	Temp. (°C)/(°F) ± 3%	Redox (mV) ± 10mV	Appearance	
											Color	Odor
Stabilization Calculations (±)												
Stabilization Criteria					**± 0.1 s.u.**	**±3%**	**± 10% or within 1 NTU (1)**	**± 10%**	**±3%**	**±10 mV**		

(1) **Turbidity** < 50 NTU and ±10% or within 1 NTU of a previous reading when <10 NTU

Constituents Sampled	Container	Number	Preservative

Comments _____

Well Casing Volumes

Gallons/Foot	*1" = 0.04*	*1.5" = 0.09*	*2.5" = 0.26*	*3.5" = 0.50*	*6" = 1.47*
	1.25" = 0.06	*2" = 0.16*	*3" = 0.37*	*4" = 0.65*	

Well Information

Well Location: _____ Well Locked at Arrival: Yes / No

Condition of Well: _____ Well Locked at Departure: Yes / No

Well Completion: Flush Mount / Stick Up Key Number To Well: _____

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RI-SOP-5

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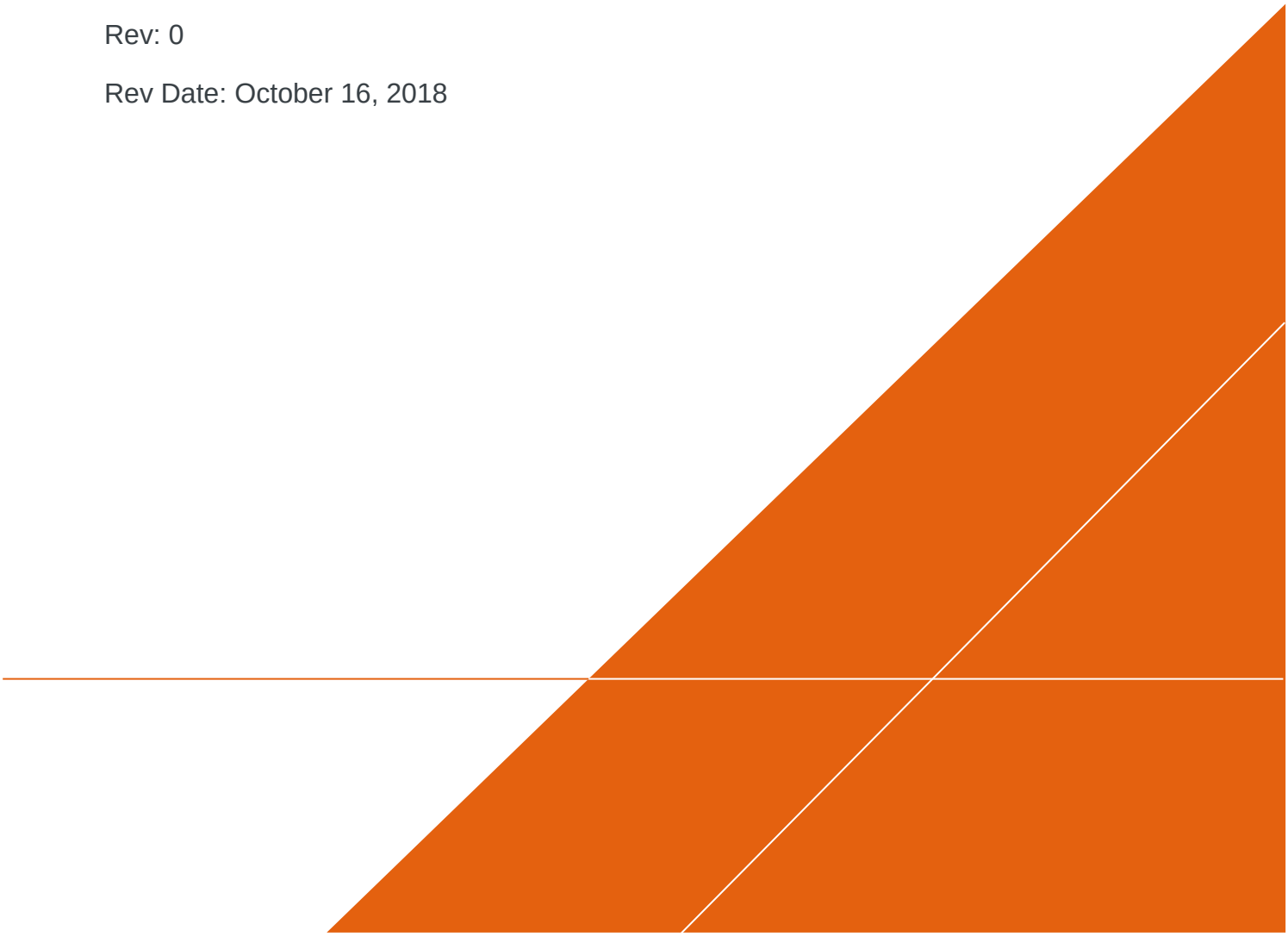
TGI - IN-SITU AND EX-SITU WATER QUALITY PARAMETERS

Rev: 0

Rev Date: October 16, 2018



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 16, 2018	All	Updated and re-written as TGI (in-situ and ex-situ adaptation)	Marc Killingstad

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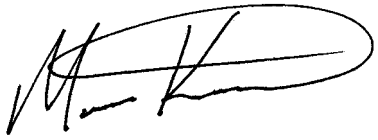
APPROVAL SIGNATURES

Prepared by:		10/16/2018
	Chris Shepherd	Date:
Technical Expert Reviewed by:		10/16/2018
	Marc Killingstad (Technical Expert)	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

Water quality parameters (e.g., turbidity, specific conductivity, pH, and temperature) of natural waters are usually measured in the field. The temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation reduction potential (ORP) and turbidity of surface water will be measured in-situ or ex-situ with a multiparameter water quality meter (i.e., multimeter).

Conductivity is the ability of a solution to pass an electric current. This current is carried by inorganic dissolved solids. The measurement is useful to quantify the chemical purity of the water relative to the amount of dissolved solids in a solution. Generally, temperature-compensated conductivity, termed specific conductivity, is measured by a water quality meter. Specific conductivity can also be used to estimate total dissolved solids (TDS) in solution (grams per liter or g/L) by multiplying the specific conductivity (measured in millisiemens per centimeter or mS/cm) by 0.65, which assumes that sodium chloride is a reasonable surrogate for the dissolved solids in solution.

Measuring the concentration of DO in water is an important component in evaluating the quality of natural as well as contaminated waters. The effects of wastes on rivers/streams, the suitability of water for fish

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and other organisms, as well as the effects of remediation efforts, can often be ascertained from the DO content. Dissolved oxygen levels generally range from 5 to 9 milligrams per liter (mg/L) when measured in water that is in equilibrium with air, depending on the temperature and barometric pressure; however, levels may be lower in a 'reducing' environment where anoxic conditions exist in water (e.g., gasoline plume undergoing bioremediation).

The measurement from an ORP sensor represents the net status of all the oxidation and reduction reactions in the sample being measured. Positive results indicate an oxidizing environment while negative results indicate a reducing environment. ORP is determined by measuring the potential of a chemically-inert electrode which is immersed in the solution. The sensing electrode potential is read relative to the reference electrode of the pH probe and the value is presented in millivolts (mV).

ORP meter results can indicate possible contamination and can be particularly useful if it is known that one component of the sample is primarily responsible for the observed value. For example, excess chlorine in a sample will result in a large positive ORP value while the presence of hydrogen sulfide will result in a large negative ORP value. Together with pH, temperature, and knowledge of the dominant species in a sample, ORP results help predict the oxidation state of ions in solution and whether certain reactions may take place.

Temperature readings will be taken at each water sampling location to assist understanding the water quality as well as compensating measurements. A thermometer is typically part of the multimeter. If not, a temperature probe or thermometer will be used.

The measurement of turbidity is useful in that it expresses the relative amount of suspended particles in the water column. Turbidity cannot be measured in-situ.

Refer to the following documents for procedures of groundwater sample collection:

TGI – Standard Groundwater Sampling for Monitoring Wells (describes the methods to be used to collect groundwater samples using traditional purging and sampling techniques)

TGI - Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells (describes low-flow purging and sampling techniques)

TGI – Passive Diffusion Bag Sampling, TGI – Groundwater Sampling with HydraSleeves™, and *TGI - Bailer-Grab Groundwater Sampling* (describes no-purge/passive sampling techniques)

3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the sampling team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan

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(FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the field sampling team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

4 EQUIPMENT LIST

The following materials, as required, will be available during field measurement of water quality parameters:

- Site-specific HASP and health and safety documents identified in the HASP (e.g., job safety assessments [JSAs])
- Field Implementation Plan (FIP) that includes site map with sampling locations, well construction records (table or logs), sampling plan, and prior groundwater sampling records (if available)
- Field notebook, pen(s) (indelible ink) and/or smart device (smart phone or tablet)
- Field laptop / data manager
- Water Quality Measurement Log (**Attachment 1**)
- Water Quality Meter Calibration Log (**Attachment 2**)
- Appropriate personal protective equipment (PPE) (e.g., latex or nitrile gloves, safety glasses, etc.) as specified in the HASP
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified in the Traffic Safety Plan (TSP)
- Well key(s)
- Photoionization detector (PID), flame ionization detector (FID) or other air monitoring equipment, as needed, in accordance with the HASP
- Dedicated plastic sheeting (e.g., Weatherall Visqueen) or other clean surface to prevent sampling equipment from coming in contact with the ground
- Electronic water-level indicator (e.g., Solinist Model 101) or oil/water interface probe with 0.01-foot accuracy (oil/water as appropriate, note that sampling will not be performed when sheen or light non-aqueous phase liquid [LNAPL] is present)
- Multiparameter (temperature/pH/specific conductivity/ORP/turbidity/DO) water quality meter or sonde with flow-through cell (as appropriate) plus reader and protective housing; for example:
    - YSI Professional Plus Multiparameter Instrument
    - YSI EX01 or EX02 Multiparameter Sonde

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  - Horiba W-22 XD Multiparameter Instrument
  - Hydrolab DS5 Multiprobe and Display
- Operation manual(s) for multiparameter meter
- Water quality meter extension cable (as needed for in-situ readings of deepest well)
- Standard solutions for calibration
- Multimeter maintenance kit and extra DO membranes if using amperometric DO probe
- Extra batteries for the various instruments
- Tape measure and/or masking tape for in-situ readings
- Decontamination equipment (buckets, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels)

5 CAUTIONS

NOTE: *Proper operation and maintenance are very important for a functioning meter and accurate readings. Carefully follow manufacturer's instructions for operation (including down-hole use), maintenance, and calibration as each manufacturer's instructions will vary.*

Verify the meter is intended for in-situ measurements and that all necessary parts are available prior to field mobilization.

Check and record the expiration dates of calibration fluids. Verify that calibration readings are reasonable and correspond to previous calibration readings when available. Expired calibration fluids may result in erroneous results. If accurate measurements of TDS are required, site-specific calibration will be necessary in accordance with the manufacturer's specifications.

Turbidity meters will be stored and used in dry locations.

When taking measurements in the field, verify that readings make sense and compare to historical readings when possible.

6 HEALTH AND SAFETY CONSIDERATIONS

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP. Also, at a minimum, wear latex gloves and safety glasses when working with calibration solutions.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or non-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well

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head), and potentially the use of sharp cutting tools (scissors, knife)—open well caps slowly and keep face and body away to allow to vent any built-up pressure; only use non-toxic peppermint oil spray for stinging insect nests; review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives, and use appropriate hand protection.

If working at a site with hazardous levels of volatile organic compounds, headspace and/or breathing zone readings will be measured prior to working at/in the well, in accordance with the HASP.

7 PROCEDURE

The procedure is organized into the following sections:

A. Standard Procedures – calibration, operation, and maintenance procedures that apply to all methods and meters

B. Calibration – general calibration procedures for:
    a. Multimeter
    b. Turbidity meter

C. Operation – general measurement operations for:
    a. Ex-situ measurements
        i. Multimeter
        ii. Turbidity meter
    b. In-situ measurements (downhole multimeter only)

D. Maintenance
    a. Multimeter
    b. Turbidity meter

7.1 Standard Procedures

Calibration

- The meter will be calibrated following the manufacturer's instructions as calibration steps vary by manufacturer.
- Calibration information will be recorded in the calibration log (Attachment 2) and the field notebook.
- Check batteries/power levels during calibration.
- More frequent calibration may be necessary in harsh conditions or per project plans.

Operation

- The meter will be operated following the manufacturer's instructions.

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- Mutimeters can be operated in-situ (down-hole, in water, etc.) or ex-situ (water is transferred to container for measurement).
- Two readings will be made after stabilization (minimum 2 minutes) and the average will be recorded in the field notebook or both measurements will be recorded in the water quality measurement log and/or logger.
- Additional details for pH, conductivity, temperature, dissolved oxygen, and turbidity operation are presented in section **7.3 Operation** below.

Maintenance

- The meter will be maintained according to the manufacturer's instructions.
- Maintenance information will be recorded in the field notebook or instrument log.
- Manufacturer recalibration or replacement probes may be required on a routine basis.
- A replacement meter and probes will be available onsite or ready for overnight shipment, as necessary.
- Additional details for pH, conductivity, temperature, dissolved oxygen, and turbidity maintenance are presented in section **7.4 Maintenance** below.
- More frequent maintenance (probe replacement, sensor reconditioning, etc.) may be necessary in harsh conditions or per project plans.

7.2 Calibration

Multimeter Calibration

1) Connect cables from meter to reader
2) Check probes and ensure they are clean
3) Switch on instrument and allow to warm-up
4) Check battery life and replace, if needed
5) Adjust date/time, if needed
6) Soak all probes in distilled or de-ionized water for at least 5 minutes (some probes may require longer soak times, see below and manufacturer instructions) and then shake off excess liquid.
    i. NOTE: *If the probes are recently reconditioned or have slight build-up, allow for longer soaking period.*
7) Navigate to calibration display/mode
8) **pH Calibration** – when in use, the pH meter will be calibrated daily, at a minimum.
    i. Connect electrode (if applicable) or remove protective cap from electrode
    ii. Rinse end of electrode in distilled/deionized water and shake off excess water

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iii. Measure and record temperature of buffer solutions

iv. Immerse pH electrode in pH buffer 7.00, set the temperature to that of the buffer 7.00, and allow sufficient time for the electrode to stabilize

v. Adjust the calibration for the correct readout and temperature

vi. Confirm/press the calibration button

vii. Remove electrode from buffer and rinse with distilled/deionized water

viii. Immerse pH electrode in buffer 4.00, set the temperature control to that of the buffer 4.00, and allow sufficient time for the electrode to stabilize

ix. Adjust the calibration for the correct readout and temperature

x. Confirm/press the calibration button

xi. Rinse electrode with distilled/deionized water

xii. A pH 10 calibration will also be performed if high pH is anticipated, following manufacturers procedures

xiii. The pH meter is calibrated

9) **Conductivity Calibration** – when in use, the conductivity meter will be calibrated daily, at a minimum.

i. Conductivity is generally measured in specific conductivity (temperature compensated), verify with the FIP/work plan

ii. Soak the probe in distilled/deionized water for at least 30 minutes

iii. Remove the probe from the water and fling out drops clinging inside

iv. Immerse the probe to or beyond the vent holes in a beaker containing 1.413 mS/cm standard solution and gently agitate vertically to remove entrapped air

v. Repeat Steps 3 and 4 at least once more

vi. Press calibration button

10) **Temperature calibration** – temperature will be verified according to FIP/work plan, if applicable.

11) **Dissolved Oxygen (DO) calibration** – when in use, the DO meter will be calibrated daily using the air calibration method or less frequently if using an optical sensor (see below).

i. Preparation

1. Polarographic sensor – periodically recondition sensor, replace fluids, and Teflon® membrane, per manufacturer, and air bubbles should not be present

2. Galvanic sensor – Periodically recondition sensor, replace fluids, and membrane, per manufacturer, and air bubbles should not be present

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3. Optical sensor – Per manufacturer, most will only require field checks and maintenance, however, weekly checks are recommended to verify the accuracy, at a minimum

ii. Obtain a barometric pressure reading from a daily weather report or from the instrument, as required by instrument

iii. Keep instrument upright and vent cap/cover while retaining a small amount of clean water (do not cover probes) for non-optical sensors only

iv. Allow 5 to 15 minutes for optimum probe stabilization and polarization, for non-optical sensors (reading will range between 5 to 9 mg/L depending on temperature and barometric pressure)

v. Press calibration button, if reading is more than 2% off of the standard

12) **Oxidation reduction potential (ORP) calibration** – when in use, the ORP meter will be calibrated daily, if required.

i. Rinse the probe in distilled/deionized water

ii. Remove the probe from the water and fling out drops clinging inside

iii. Immerse the probe in the ORP solution. Gently agitate vertically to remove entrapped air

iv. Repeat Steps 3 and 4 at least once more

v. Press calibration button, if reading is more than 2% off of the standard

13) Adjust the instrument to READ or LOGGING mode, per instruction manual and project plans; the instrument is now ready for use

Turbidity Calibration (if required)

Turbidity meters are generally calibrated weekly, unless required otherwise. However, daily checks are recommended to verify the accuracy. The turbidity meter will be calibrated per steps below:

1) Switch on instrument and allow time to warm-up

2) Check battery life and replace, if needed

3) Turbidity sample tubes will always be washed prior to use, but not the calibration standard tubes, using a mild detergent to remove any dirt or finger prints

4) Dry the outside of the sample turbidity tubes with a clean, lint-free cloth or disposable wipe and allow the turbidity tubes to air-dry in an inverted position to prevent dust from entering the tube, as allowable (but, not the calibration standard tubes)—dirt or fingerprints can lead to inaccurate results

5) Wipe the 1 Nephelometric turbidity unit (NTU) standard and place in the chamber after aligning the arrows (manufacturer dependent)

6) Place the light shield over the turbidity standard and allow the meter to stabilize

7) Press the read and/or CAL button, per instructions

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8) Repeat steps 3 thru 5 above

9) Adjust to READ mode and the instrument is now ready for use.

7.3 Operation

Water quality parameters can be measured ex-situ or in-situ in accordance with the FIP/work plan. The operating procedures for each method are described below. It is also possible to collect a depth-specific sample from a well or water body for ex-situ measurement.

Ex-situ Operation

Multimeter

1) Fill two 100-mL plastic disposable beakers or clean beakers with water from the sample
2) Insert the probe into the first beaker immersing all sensors and allow to stabilize (2 minutes at minimum)
3) Record readings
    i. Temperature – in degrees Celsius or Fahrenheit
    ii. pH – in standard units between 0 and 14, in 0.01 increments
    iii. Specific conductivity – in mS/cm or microsiemens per centimeter (uS/cm)
    iv. DO – in mg/L and percent (will typically read between 0 and 15 mg/L)
    v. ORP – in millivolts (mV; will typically read between -1,400 mV and + 1,400 mV), if required
4) Rinse probe off with distilled/deionized water
5) Repeat Steps 2 thru 4 for the other beaker
6) Log results on the water quality measurement log and/or in field notebook - the average will be the actual result
7) Rinse probe off with distilled/deionized water

NOTE: *If a flow through cell is used during low flow groundwater sampling, a single reading can be obtained from the multimeter during each step of the wells purging in accordance with TGI - Low Flow Groundwater Purging and Sampling.*

Turbidity Meter

1) Fill the two cleaned, manufacturer provided sample tubes with water from the sample
2) Wipe off and dry the outside of the sample turbidity tubes with a clean, lint-free cloth or disposable wipe
3) Insert the first sample tube and close the cover
4) Push the READ button
5) Record turbidity reading in NTUs (0 to 1,100 NTUs)

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i. If readings are over the instrument limit, a single 2X dilution can be run by mixing the sample with equal parts distilled or de-ionized water and gently mixing

ii. The resulting value will need to be doubled (for example, if the diluted reading is 750 NTUs, the estimated turbidity would be 750 x 2 = 1,500 NTUs. Diluted samples will be qualified as estimated)

6) Repeat steps 2 thru 5 with the second sample tube

7) Log the results on the water quality measurement log and/or in field notebook - the average will be the actual result

8) Rinse sample tubes with distilled/deionized water

NOTE: *If a flow through cell is used during low flow groundwater sampling, a single reading can be obtained from the multimeter during each step of the wells purging in accordance with TGI - Low Flow Groundwater Purging and Sampling. The turbidity sample is recommended to be collected prior to passing through the flow through cell.*

In-situ Operation

1) Connect extension cable and protective housing to meter

2) Measure water level from reference point

3) Lay out plastic sheeting as needed to keep the multimeter clean

4) Clean multimeter and cable per decontamination procedures and instruction manual and rinse with distilled/deionized water using caution, as most meters can only handle light washing with a mild soap and warm water

5) Organize work area to prevent dirt or objects from falling in the well

6) Measure and mark extension cable at planned measurement intervals

7) Slowly lower the probe into the well or water body to the desired measurement interval using caution to prevent rubbing of cable on the well and minimize water and sediment disturbance

8) Allow readings to stabilize (typically 1 to 3 minutes)

i. For many instruments, the instrument may need to be slowly oscillated up and down a few inches to circulate water around the probes

9) Record readings

i. Temperature – in degrees Celsius or Fahrenheit

ii. pH – in standard units between 0 and 14, in 0.01 increments

iii. Specific conductivity – in mS/cm or uS/cm

iv. DO – in mg/L and percent (will typically read between 0 and 15 mg/L)

v. ORP – in mV (will typically read between -1,400 mV and +1,400 mV), if required

10) Repeat steps 4 through 9 to complete targeted in-situ measurements

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11) Log results on the water quality measurement log and/or in field notebook

12) Slowly retrieve the multimeter

13) Clean multimeter and cable per decontamination procedures and instruction manual and rinse with distilled/deionized water using caution, as most meters can only handle light washing with a mild soap and warm water

7.4 Maintenance

Multimeter

1) After use, the meter will be inspected and maintained according to the manufacturer's specifications and the inspection/maintenance activities will be recorded in the field notebook

2) Keep records of usage, maintenance, calibration, problems, and repairs

3) Recharge/replace batteries on a regular basis

4) Store meters or electrodes in protective casing when not in use

5) DO membranes will be stored moist, unless specified otherwise by manufacturer

6) Focused robe cleaning may be necessary in accordance with the operation manual and may be needed more frequently in harsh conditions

7) A replacement meter will be available onsite or ready for rapid delivery/shipment

8) Periodic manufacturer calibration may be necessary per operation manual and/or field plans

Turbidity Meter

1) Recharge battery on a regular basis

2) Store in protective casing when not in use

3) Keep records of usage, maintenance, calibration, problems, and repairs

4) After use, the meter will be inspected with results recorded in the field notebook

5) Keep sample tubes clean inside and out replacing them when they become scratched or etched and avoid handling the tubes in the region where the light beam enters them.

6) Clean lens periodically per instruction manual

7) Turbidity meter will be sent back to the manufacturer for service when needed

8 WASTE MANAGEMENT

Investigative-Derived Waste (IDW), including rinse water, excess sample water, spent calibration solutions, and disposable materials (plastic sheeting, PPE, etc.) generated during the procedures outlined in this TGI will be collected and stored on site in appropriately labeled containers (disposable materials will be contained separately) and disposed of properly. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained

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(e.g., soil, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log book.

PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures activities will be placed in plastic bags. These bags will be disposed of as general waste unless specified otherwise.

9 DATA RECORDING AND MANAGEMENT

Management of the original documents from the field will be completed in accordance with the site-specific QAPP. Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

In general, documentation of the following information is required:

- Calibration – calibration information will be recorded on a calibration form, field log, or electronic device per project plans
    - Meter manufacturer and model
    - Serial number
    - Calibration personnel
    - Calibration date/time
    - Standard value, initial and final reading
    - Observations, if applicable
- Readings - data will be recorded on a field log, sampling form, or electronic device per project plans
    - Instrument model
    - Measurement date/time
    - Field personnel
    - Weather
    - Measurement location and depth, if applicable
    - Value of readings and average reading, if applicable
    - Units of readings
    - In-situ vs ex-situ measurement method
    - Key observations

All records will be provided to the project manager and retained in the project files. Any maintenance needs will be communicated to the project manager promptly.

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QUALITY ASSURANCE

Quality assurance procedures will be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

Refer to the QAPP or FIP/sampling plan/work plan for the frequency of calibrations.

10 REFERENCES

Not applicable.

11 ATTACHMENTS

Attachment 1 - Water Quality Measurement Log

Attachment 2 - Water Quality Meter Calibration Log

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ATTACHMENT 1

Water Quality Measurement Log

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WATER QUALITY MEASUREMENT LOG

GENERAL INFORMATION

Date _____ Weather _____

Project No _____ Sample Personnel _____

Site Name _____ Sample ID _____

Site Location _____ Start reading time _____

Site/Well No. _____ End reading time _____

MEASUREMENT DATA

Measuring Point (MP) description _____

Depth to Water (ft)/Time _____ Instrument model/serial no. _____

Well Screen Interval (ft) _____ Instrument model /serial no. 2 _____

Casing Diameter (in) _____ ☐ In-situ ☐ Ex-situ reading

Meters calibrated (Y/N) _____ (see calibration log, if applicable)

Water Quality Parameters

Time	Depth (ft)	pH (S.U.)	Spec. Cond. (mS/cm or uS/cm)	Temp (°C or °F)	DO (mg/L)	DO (%)	ORP (mV)	Turbidity (NTU)	Observations (Odor, clarity, etc., if applicable)

REMARKS

_____

_____

_____

_____

ATTACHMENT 2

Water Quality Meter Calibration Log

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WATER QUALITY METER CALIBRATION LOG

PROJECT NAME	
PROJECT NO.	
MODEL	
SERIAL #	
SAMPLER	
DATE	

TURBIDITY CALIBRATION

CAL. READING		CAL. READING		CAL. RANGE	TIME
(LOT #)		(LOT #)			
(EXP. DATE)		(EXP. DATE)			
PRE-CAL. / POST-CAL		PRE-CAL. / POST-CAL			
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	

AUTOCALIBRATION

AUTOCAL SOLUTION		CAL. READING	CAL. RANGE	TIME	CALIBRATION RANGES (1)	
(LOT #)						
(EXP. DATE)						
CALIBRATED PARAMETERS		PRE-CAL. / POST-CAL				
☐	pH	/	☐ WITHIN RANGE		pH	± 0.2 S.U.
☐	CONDUCTIVITY	/	☐ WITHIN RANGE		COND	± 1% OF CAL. STANDARD
☐	ORP	/	☐ WITHIN RANGE		ORP	± 25 mV
☐	DO	/	☐ WITHIN RANGE		DO	Atmospheric
☐	TURBIDITY	/	☐ WITHIN RANGE		TURB	± 5% OF CAL. STANDARD
☐		/	☐ WITHIN RANGE			
☐		/	☐ WITHIN RANGE			

(1) CALIBRATION RANGES ARE SPECIFIC TO THE MODEL OF THE WATER QUALITY METER

NOTES:

SIGNED DATE

CHECKED BY DATE

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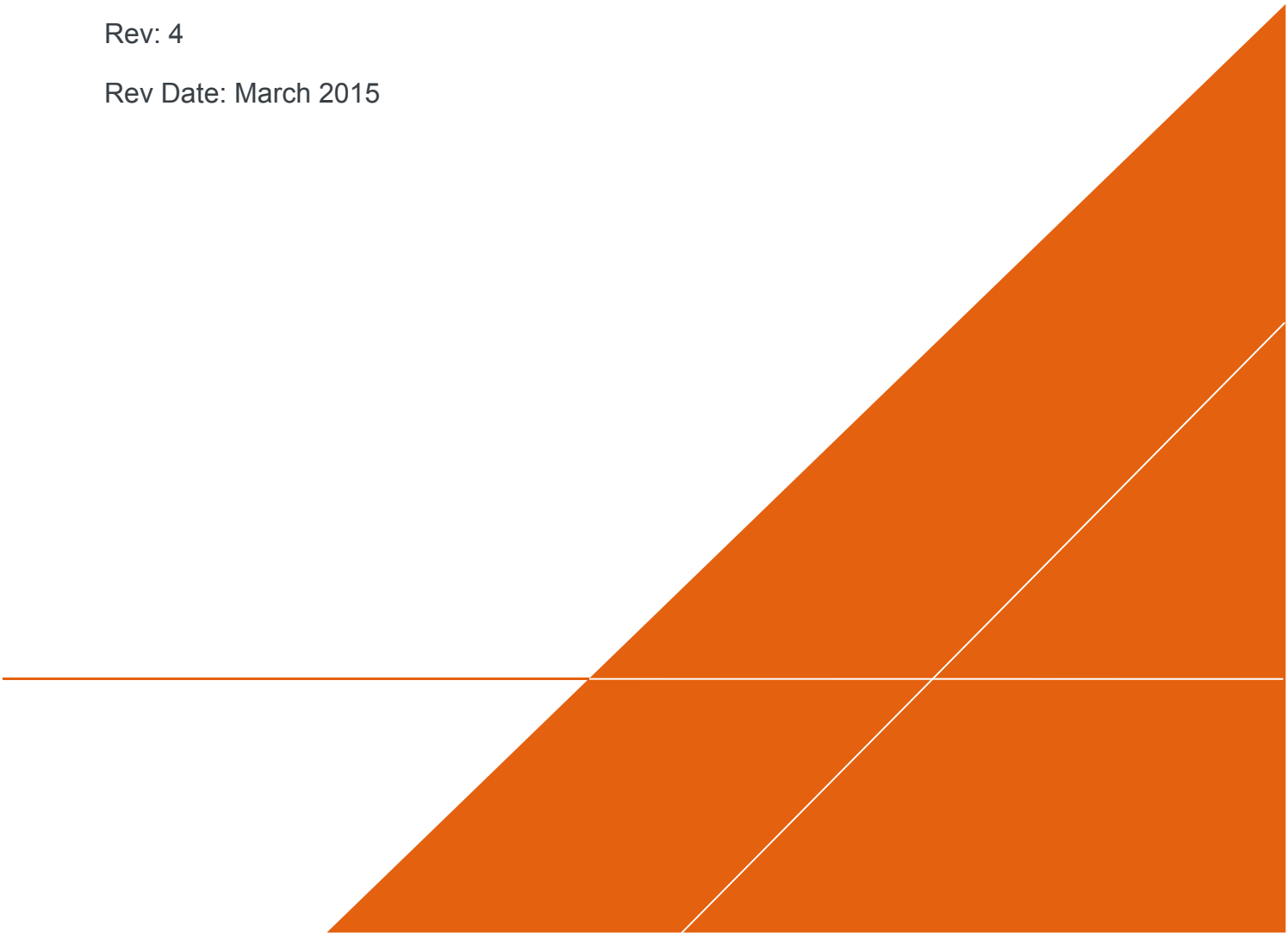
TECHNICAL GUIDANCE INSTRUCTIONS FOR SLUG TESTING

Rev: 4

Rev Date: March 2015



TGI VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by

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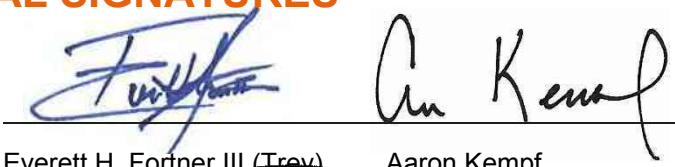
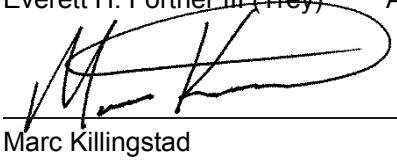
Prepared by:  Date: March 5, 2015

Everett H. Fortner III (Trey) Aaron Kempf

Reviewed by:  Date: March 5, 2015

Marc Killingstad

ABOUT THIS TGI

This Technical Guidance Instruction for Slug Testing is intended as general guidance for testing design that applies to all forms of slug testing. Guidance for the specific slug testing method selected based on this overall guidance is attached in four separate documents. The four detailed methods of slug testing covered include:

A TGI: Solid Slug Testing

B TGI: Water Slug (Inflow) Testing

C TGI: Baildown Testing

D TGI: Pneumatic Slug Testing

Each of these attachments should be used in conjunction with this general guidance. For more information or questions, please contact the TKI technical lead for Aquifer Testing.

1 SCOPE AND APPLICATION

The objective of this Technical Guidance Instruction (TGI) is to establish uniform procedures for slug testing to estimate the hydraulic conductivity of the groundwater zone near a well. A slug test is completed by "instantaneously" inducing an artificial change in hydraulic head and measuring the rate of the groundwater return to equilibrium (static) conditions. This guidance document provides detailed information on test methodology, planning, and application. Field forms and procedures for conducting tests using solid slugs, inflow (water slug), baildown, and pneumatic testing methods are attached. Also attached is a parts list and as-built drawing for the pneumatic testing manifold.

Please note that the data analysis portion of slug testing is not covered in this guidance.

It is strongly advised that, prior to conducting any aquifer testing, the project team contact the Technical Knowledge and Innovation (TKI) Aquifer Testing Focus Group to receive specific technical guidance regarding the design, execution, and analysis phases of the proposed testing.

Slug tests are used as an economic, simple, and rapid way to obtain data needed to estimate near-well hydraulic conductivity (Butler 1998). The advantages of slug testing over other testing methods (e.g. pumping tests) is that they are relatively easy and inexpensive to implement and generate little to no investigation-derived waste. These advantages allow for several tests to be completed at a site over a relatively short period of time, which provides a better understanding of the spatial distribution of hydraulic conductivity both horizontally and vertically across the groundwater zone(s) (i.e., aquifer heterogeneity). However, slug tests should not be viewed as a replacement for the larger scale estimates of hydraulic conductivity derived from multi-well pumping tests (Kruseman and de Ridder 1994). The shorter time frame and limited stress on the groundwater zone from slug tests provides a smaller scale (near-well) hydraulic conductivity estimate than pumping tests. Due to this localized scale of slug testing, the effects of the well filter pack, well development, and well skin are more significant than during pumping tests, and have the potential to limit water-level change during testing. Wells that are insufficiently developed can adversely affect the results of the slug test and generally lead to a lower hydraulic conductivity estimate than other conventional methods. Therefore, careful consideration should be given prior testing wells that are expected to have skin damage and where the development history is unknown. Varying the direction and magnitude of the stress and inducing a relatively large, measurable head change can facilitate determination of skin effects and increase the probability that the selected test response is a result of groundwater zone hydraulics and not the well construction hydraulics (Butler 1998).

Slug Test Design

The initial step during the planning stage should consist of determining whether or not slug tests will provide adequate aquifer characterization to meet the specific project objectives. For example, slug testing may be useful in mapping relative changes in hydraulic conductivity (heterogeneities) or used to calibrate high resolution hydraulic testing results provided by direct-push tools. However, slug testing is not an appropriate testing method to obtain aquifer storage estimates. In addition, slug testing may not be appropriate for testing of bedrock, particularly when there is incomplete understanding of the fracture network. A pumping test, packer test, or open borehole test may be more appropriate in bedrock. However, weathered bedrock zones can often be considered hydraulically equivalent to a porous medium and, therefore, slug testing may be appropriate.

The design phase includes several elements as outlined below. Please note, there may be exceptions, as design specifications need to be tailored to the conceptual site model (CSM) and project objectives.

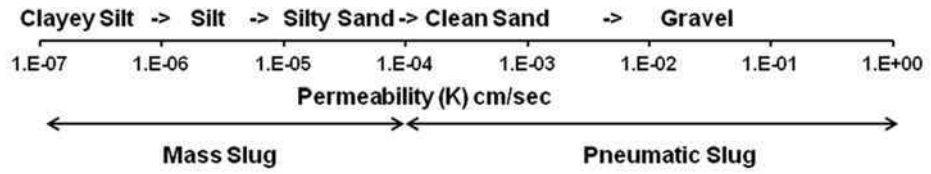
1. Review of the CSM and site data. This should include a review of boring logs (key elements – lithology and drilling method utilized), well construction details, well development logs, cross-sections, constituent plume information, hydrostratigraphic zones, previous estimates of hydraulic conductivity, and regional estimates from published sources. Special attention should be given to well screen intervals in order to ensure that the tests are conducted in the targeted groundwater zone. If a well is screened across multiple hydrostratigraphic zones, uncertainty associated with estimated hydraulic conductivity values for a given zone is introduced. However, there are exceptions if the permeability of the zones that are crossed by the well screen differ significantly.
2. Review of regulatory program rules or guidance to ensure proper compliance with the criteria outlined in the regulatory documents. In most cases, slug testing using pneumatic or solid slug methods will not require permitting; however, use of in-flow procedures, although unexpected, may require a permit (e.g., Underground Injection Control [UIC] permit).
3. Compile detailed information for each proposed test well:
    a. Well construction information
        i. Well casing/screen diameters and lengths
        ii. Screen type (material and slot design) and slot size
        iii. Total depth – reviewing the measurement of the test well total depth aids in development assessment. If the total depth has decreased substantially since installation, sediment buildup may be occurring and redevelopment should be completed before testing
        iv. Filter pack construction (thickness and material)
    b. Historical and current groundwater levels – this will aid in the determination of the appropriate slug test type (e.g., is the screen submerged)
    c. Well development record – testing improperly developed wells will result in anomalous results
    d. Recent well sampling logs – historic sampling data will provide information on the potential yield of the well and groundwater quality (turbidity)
4. Determine appropriate testing method(s): In light of the information researched in items 1 through 3, slug test method(s) should be selected based on the groundwater zone information and data needs for proper analysis:

    - Solid Slug – used for rising and falling head tests in wells of adequate water column length with fully or partially submerged screens. If screen is partially submerged (i.e., intersects the groundwater table), then a rising head test is the most appropriate test method. Adequate length of water column would allow for placement of transducer at least 1 foot below the anticipated submersion depth of the slug or length of water column to accommodate the just the slug when using manual measurements.

    - Inflow – used for falling head tests where the water column is too small to accommodate typical slug test equipment or applied as part of development procedures.

    - Baildown – used for rising head tests.

- Pneumatic – used for rising head tests with fully submerged well screens. The well is pressurized to depress the water-level and then allowed to equilibrate. The recovery to static conditions is measured after the pressure is released.
- Aquifer information: The initial hypotheses of the groundwater zone characteristics and permeability based on either previous testing, qualitative information (boring logs), or other studies in the area is also an important consideration when determining the testing methodology. The chart below depicts of test type to be applied in relation to permeability for guidance.



Note: For instance, if boring logs indicate the groundwater zone is predominantly sand and/or gravel and groundwater sampling logs indicate good well yield, then a hypothesis may be made that the formation may have has a relatively high permeability. Therefore, if the well screens are fully submerged, pneumatic slug testing would be appropriate.

- Additional data needs: Additional data may be required or appropriate in order to determine the most appropriate test method (Butler 1998) and to understand external influences on the test. This includes duplicate testing, rising and/or falling head tests, multiple tests with varying initial displacements, and background data to assess background water level trends. Generally, three tests per well is recommended with two duplicate tests at the same displacement and one with double the original displacement. Special cases with expected external influences that induce water level changes (tidal, atmospheric or pumping) may require determination of pre-test water level trends. Arrange to have nearby active pumping wells shut down for at least 48 hours prior to testing or constant rate extraction during testing, if applicable and possible. Finally, testing should not be completed during periods of significant precipitation.

5. Data acquisition: To ensure high resolution data acquisition, data-logging pressure transducers should be used to collect water level data. Change in head measurements should be collected at a frequency of 0.5 second or less for high permeability groundwater zones (e.g., gravels and sands). A lower frequency can be selected for lower permeability groundwater zones (e.g., fine sands, silts, and clays). Ideally, frequencies should be in the seconds range depending on the permeability and the length of the test. To optimize data collection and test quality decisions, the transducer can be used in real-time viewing. Manual water-level measurements should also be collected to verify and back-up the electronic data collected using pressure transducers. For low permeability formations with fully submerged well screens, manual data acquisition may be appropriate (i.e., where groundwater recovery occurs at a rate slow enough such that frequent data collection is not required).

2 PERSONNEL QUALIFICATIONS

Field personnel performing the testing should have the following qualifications:

- Sufficient "hands-on" experience necessary to successfully complete the slug test field work. Training requirements for conducting slug tests include reviewing this guidance and other applicable documents and instrument calibration.
- Demonstrated familiarity with the electronic data logging equipment.
- Completed current health and safety training in accordance with the project health and safety plan (e.g., 40-hour Hazardous Waste Operations training and site-specific training, as appropriate)

Additional Information – Key Considerations

- Pressure Transducers/Data Loggers
    - Ensure that all rental instruments and tapes have been calibrated.
    - Small-diameter pressure transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Install the pressure transducer at a reasonable distance below the targeted drawdown estimated for the well to prevent noise. Do not install the transducer closer than 6 inches from the base of the well to eliminate the possibility of fouling the transducer with debris/mud accumulated at the bottom of the well. To prevent pressure transducer malfunction or damage, do not submerge pressure transducers in excess of the operating range, and do not insert objects in the sensor opening unless directed by the manufacturer.
    - For vented pressure transducers/data loggers, test functionality with a field test of readings using a bucket or barrel filled with water. Submerge pressure transducer, accurately measure the water head above the pressure transducer, and compare the measurement to the reading.
    - Non-vented transducers, which record a combined pressure of barometric and the water column above the pressure transducer, can be tested in the same fashion as the vented pressure transducer (outlined above). The water column above the pressure transducer can be checked by subtracting out current atmospheric pressure.
    - In general, when testing the pressure transducers, check the pressure transducer response to changing heads by raising the pressure transducer a certain distance, observing the change in head, and then measuring the distance manually. Additionally, water level meters should be in good working condition and calibrated to true depth and ensuring that there are no breaks or splices in the cable.
    - Pressure transducers should be set in the well at least 10 minutes prior to testing to allow to the transducer to thermally equilibrate with groundwater and ensure that the pressure transducer cable will not stretch during testing.
    - Logarithmic or head-change settings should not be used to log data, only linear.

  - Prior to testing, secure pressure transducer cables at the wellhead to prevent movement that would affect measurements. Mark a reference point on the down-hole transducer cable or securing line and check regularly to detect slippage.

- Data Management
  - Data management is crucial to prevent any loss. Use caution not to overwrite any previously recorded files and remember, data backup is always necessary. A job loss would occur if data would be accidently lost. Always back up data on a laptop computer and a flash drive and keep at different spots (e.g., back pack and glove compartment) to reduce the risk of data loss (e.g., computer failure).
- Slug Volume
  - Solid slugs should be calibrated to determine their accurate volume(s) for theoretical displacement. In most cases, rental slugs offer economic and data quality benefits over field-built slugs.
  - When completing baildown or inflow testing, purge or injected volume should be measured accurately.
- Initial Displacement and Recovery
  - When performing slug tests, the general rule of thumb for initial displacement is between 1 and 6 feet and/or generally less than 25% of the effective screen length.
  - Water levels should be recorded to within 80% to 95% recovery. In addition, duplicate tests should be completed only after the first test has recovered by at least 95%.
- Investigative-Derived Waste (IDW)
  - Containerize all purged water as specified in the project plans. Discharge water should be disposed of according to all applicable laws, regulations, and project guidelines. Contact the governing agencies to determine which restrictions apply. Arcadis should not be responsible for signing manifests and should not "take possession" of purged water.
- Equipment Care
  - Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place pressure transducers near electric power generators or electric pump motors. Likewise, radio signals may cause pressure transducers or computers to malfunction.
- Decontamination
  - Make sure all equipment that enters the test well (slug, water-level meter, pressure transducer) is decontaminated before use. If testing multiple wells, start with the least contaminated and progress to the most contaminated.
- Non-Aqueous Phase Liquids (NAPL)
  - Slug tests should not be conducted in wells where Non-Aqueous Phase Liquids are present. Consult TKI Aquifer Testing Focus Group lead for guidance.

3 HEALTH AND SAFETY CONSIDERATIONS

The site-specific HASP will be used to ensure that the tests are conducted in a safe manner, and should include a Job Safety Analysis (JSA). The following specific health and safety issues should be considered when conducting slug tests:

- Appropriate PPE with minimum of Level D should be worn to avoid contact with site chemicals of concern during slug test.
- Well covers should be carefully removed to avoid potential contact with insects or animals. Well caps should be vented or tethered to avoid potential eye injury in case of gas buildup in the well.
- Pressurization or vacuum hazards associated with pneumatic slug testing should be considered during test planning and implementation.

4 WASTE MANAGEMENT

Rinse water, PPE, and other waste materials generated during equipment decontamination should be placed in appropriate containers and labeled. Containerized waste should be disposed of, consistent with appropriate waste management procedures for investigation-derived waste.

5 DATA RECORDING AND MANAGEMENT

Field personnel will complete a Slug Test Field Log form for each test. As previously noted, it is generally recommended to conduct three tests per well (the original displacement, a duplicate, and double original displacement); therefore, one field log should be completed for each test. It is strongly recommended that data be copied to a flash drive and transmitted to the project team as soon as possible to ensure no data loss. Field equipment calibration, decontamination activities, and waste management activities should be recorded in the field logbook.

6 QUALITY ASSURANCE

Review data collected during field testing to determine reasonableness/quality given site-specific conditions. Again, this can also be completed using the transducer in real-time viewing mode as the test progresses. Compare the theoretical head displacement calculated from the slug volume or pressure to the observed displacement. If the data are questionable, the field equipment should be checked to confirm proper working order and the test may be repeated, if possible. Consult with the project hydrogeologist to work through issues encountered in the field and to help determine test validity.

Any issues that may affect the data should be recorded in the field logbook for consideration by the project hydrogeologist.

7 REFERENCES

Butler, J.J., Jr., 1998. The Design, Performance, and Analysis of Slug Tests, Lewis Publishers, New York, 252p.

Kruseman, G.P. and N.A. de Ridder, 1994. Analysis and Evaluation of Pumping Test Data (2nd ed.), Publication 47, Intern. Inst. for Land Reclamation and Improvement, Wageningen, The Netherlands, 370p.

ATTACHMENT A

TGI: Solid Slug Testing

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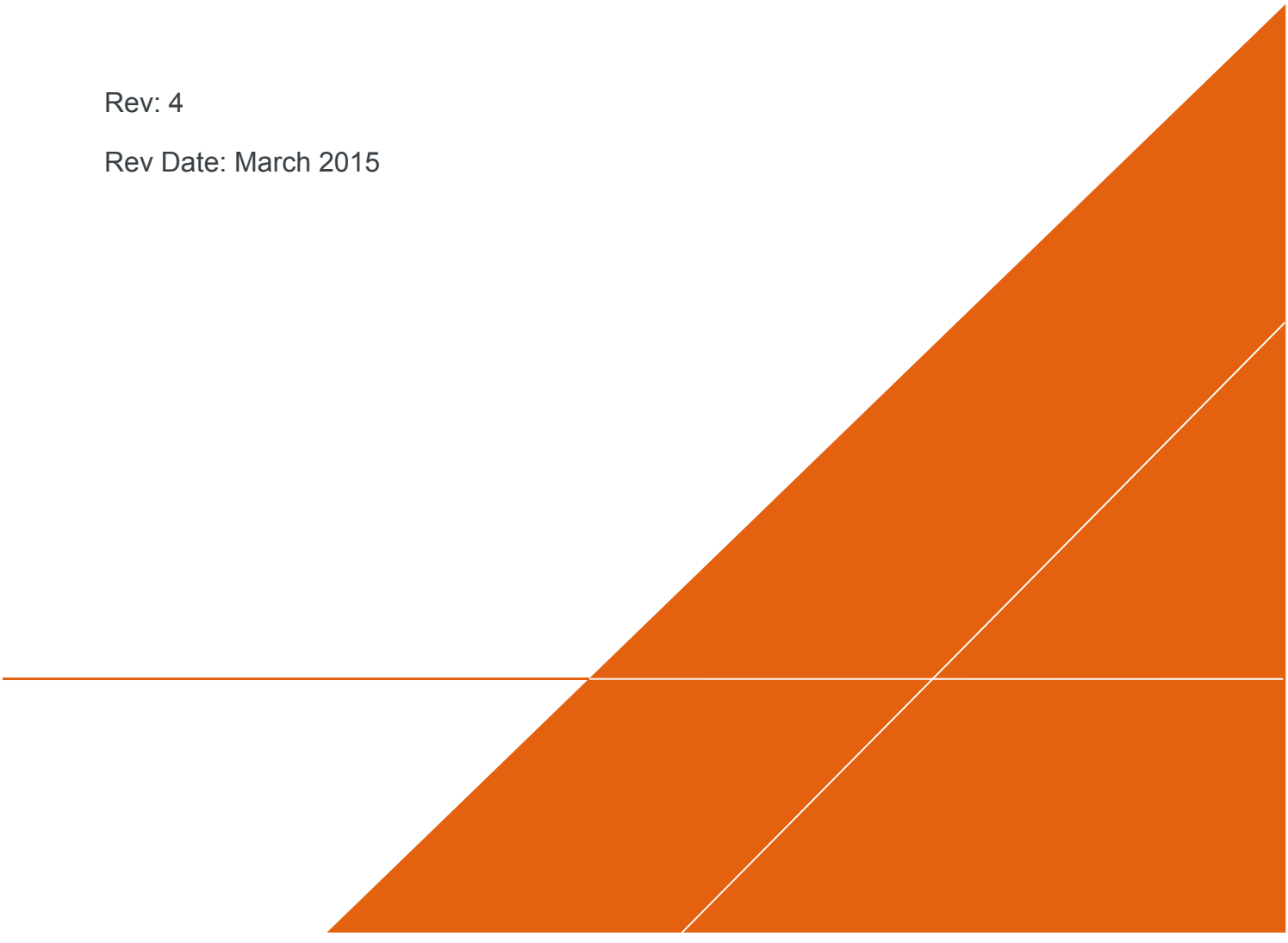
Slug Testing TGI Attachment A

TECHNICAL GUIDANCE INSTRUCTIONS FOR SOLID SLUG TESTING

Rev: 4

Rev Date: March 2015



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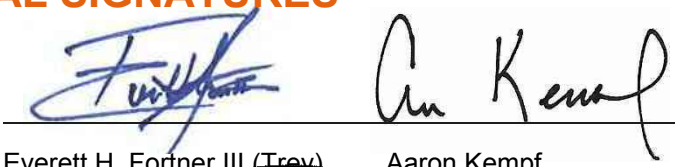
Prepared by:  Date: March 5, 2015

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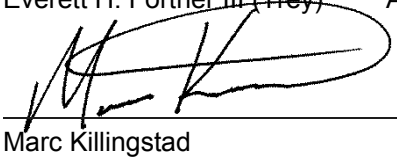
Reviewed by:  Date: March 5, 2015

Marc Killingstad

1 SCOPE AND APPLICATION

The use of a solid slug allows for both falling- and rising-head slug tests to be completed. Solid slug(s) of a known volume are inserted and removed from the water column in a well in a near-instantaneous manner. The water level response is observed using a data-logging pressure transducer with manual measurement backup or using just a manual water level meter for slow recovering wells with fully submerged screens.

2 EQUIPMENT LIST

The following materials should be available, as required, during slug testing using a solid slug:

1. Job safety analysis and site Health and Safety Plan
2. Related project-specific requirements and plans
3. Personal protective equipment, as required by the site Health and Safety Plan
4. Solid slug(s) of known volume
5. Pressure transducer and barologger
6. Pressure transducer software
7. Laptop computer and/or data transfer device
8. Rope or cables (for deep wells) (chemical resistant, low stretch is optimal)
9. Water level meter
10. Measuring tape
11. Spring-loaded clamps and zip ties
12. Decontamination equipment
13. Slug test field form
14. Field notebook
15. Waterproof marker.

3 PROCEDURE

1. Decontaminate all down-well equipment: pressure transducer and cable, slug(s), rope or cable, water level meter in accordance with project specific requirements. In general, wells should be tested from least contaminated to more contaminated, if possible or applicable.
2. Measure depth to water and well total depth. Determine the water column length.
    a. Multiple depth to waters should be measured and any trends should be noted.
    b. The "static" depth to water should be representative of the water level after the well equalizes with atmosphere.
3. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
4. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory should be used to select the highest measurement frequency possible.

   a. In hydrologic settings where high hydraulic conductivity is expected, water levels should be measured at 0.5-second intervals or the highest frequency available. This measurement frequency should be selected for gravels and sands.
   b. In hydrologic settings where low hydraulic conductivity is expected, water levels should be measured at 1- to 2-second intervals. This measurement frequency should be selected for silts and clays.

5. If applicable, program the barologger to record barometric pressure. The barologger should be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.
6. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally the transducer should be 5 to 10 feet lower than the maximum depth of the slug testing equipment not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom as silt can clog the pressure transducer. Clamp the pressure transducer cable to the well casing or other static object.
7. View the measured water level in real time on the laptop computer. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer should be permitted to equilibrate to groundwater temperatures to ensure accurate water-level measurements.
8. Re-measure the depth to water.
9. Measure the slug and rope assembly length and mark the rope at a length as follows:

   Rope Mark #1 = Depth to Potentiometric Surface from TOC

   Rope Mark #2 = Depth to Potentiometric Surface from TOC + Length of Slug + Safety Factor (Safety Factor = 10% of the Length of Slug)

   When deployed, Rope Mark #2 should be at the well top of casing, and the slug should be totally submerged. If insufficient water column is available to cover the slug assembly top, note the theoretical length of the slug to be inserted into the water column. Upon removal, measure the wet slug length.

10. Slowly insert the slug assembly into the well and stop just above the potentiometric surface Rope Mark #1.
11. With slack in the rope and the slug being suspended above the water column, place the Rope Mark #2 at the top of casing. Clamp the non-slug end of the rope to a static object.
12. Quickly drop the slug into the water column.
13. Observe the water level response on the laptop computer and/or measure depth to water, being careful not to interfere with the pressure transducer cable. Several manual depth to water measurements should be made throughout the test (typically 2 to 3 in the first minute, one reading a minute for the next 5 to 10 minutes, and every 2 to 5 minutes thereafter). If the water level meter is used as the primary measurement technique, the measurement frequency should be increased as practicable.
14. Allow sufficient time for water level to recover to static level. If completing one test (just a falling head test or just a rising head test), then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test should be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.

15. Quickly remove the slug assembly from the water column. The slug assembly should be left in the well above the static water level in order to limit pressure cable disturbance until the testing is complete and the water levels have equilibrated to the target level.
16. Repeat both the falling- and rising-head slug tests for data reproducibility by repeating steps 12 and 13, if applicable. If possible, complete a third test with a slug or combination of slugs that equates to twice the volume as the original.
17. Save all data files to the laptop, backup on flash drive or by emailing, and finalize any field notes.
18. Review the data collected to determine the reasonableness of the preliminary results and compare the pressure transducer results to the water level meter results. The observation of apparently anomalous results should be discussed with senior project staff prior to additional testing or leaving the field site. The water level record for each test should show static conditions, the insertion or removal of the slug(s), and the water level response. Make notes on the field form and notebook concerning any irregularities.
19. Decontaminate all down-well equipment in accordance with project plans.

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ATTACHMENT B

TGI: Water Slug (Inflow) Testing

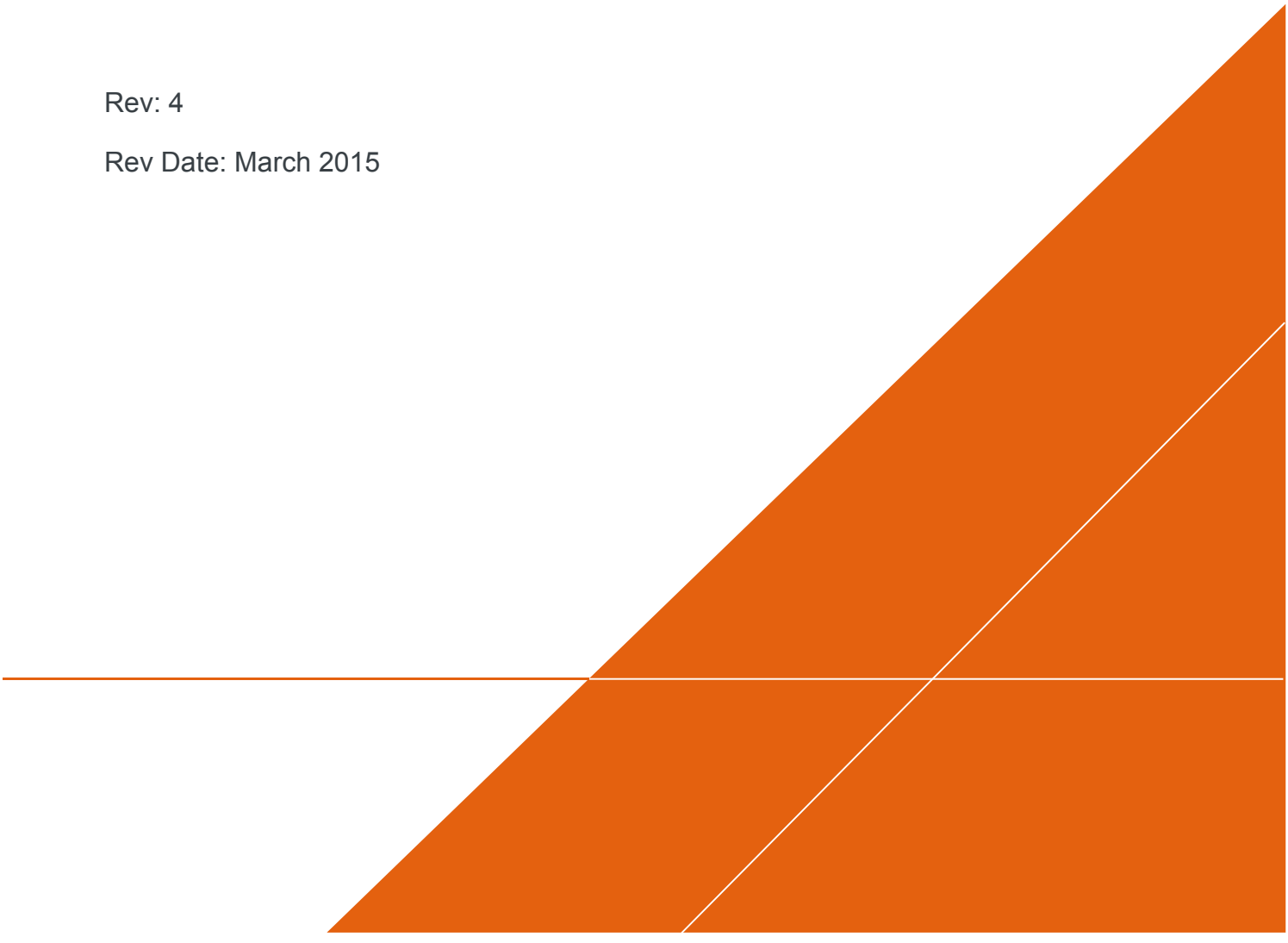
ARCADIS Design & Consultancy for natural and built assets

Slug Testing TGI Attachment B

TECHNICAL GUIDANCE INSTRUCTIONS FOR WATER SLUG (INFLOW) TESTING

Rev: 4

Rev Date: March 2015



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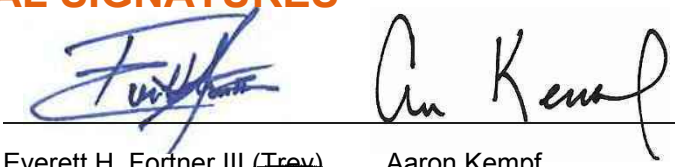
Prepared by:  Date: March 5, 2015

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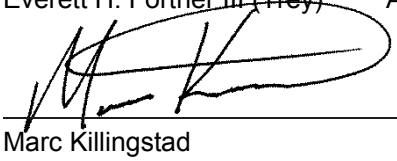
Reviewed by:  Date: March 5, 2015

Marc Killingstad

1 SCOPE AND APPLICATION

A known volume of potable water (slug) may be used to complete falling-head slug tests. Water of a known volume is poured into a well in a near-instantaneous manner. The water level response is observed using a pressure transducer. Slug tests using a water slug are most appropriate for fully submerged well screen (i.e. water table above the well screen top), and where a relatively slow response is anticipated. These constraints limit the probability of slug water entering the filter pack and vadose zone, thereby ensuring slug insertion to be near instantaneous relative to the observed response. This type of test is sometimes used to evaluate well development by performing a slug test before and after well development.

Consult with local regulatory requirements concerning underground injections. The injection volume and injectate are typically innocuous enough that a permit-by-rule authorization is granted in lieu of an underground injection control permit.

2 EQUIPMENT LIST

The following materials should be available during slug testing using a water slug:

- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Potable water (do not use distilled water, as it is not conductive and will not work with electronic water level meters)
- Pressure transducer and cable
- Pressure transducer software
- Laptop computer and/or data transfer device
- Graduated cylinder or similar measuring device
- Funnel with large neck and opening (wide mouth)
- Water level meter
- Spring-loaded clamp and zip ties
- Decontamination equipment
- Slug test field form
- Field notebook.

3 PROCEDURE

1. Decontaminate all down-well equipment: pressure transducer, pressure transducer cable, and water level meter in accordance with project specific requirements. In general, wells should be tested from least contaminated to more contaminated, if possible or applicable.
2. Measure depth to water and well total depth. Determine the water column length.
    a. Multiple depth to waters should be measured and any trends should be noted.
    b. The "static" depth to water should be representative of the water level after the well equalizes with atmosphere.
3. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
4. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory should be used to select the highest measurement frequency possible.
    a. In hydrologic settings where high hydraulic conductivity is expected, water levels should be measured at 0.5-second intervals, or the highest frequency available. This measurement frequency should be selected for gravels and sands.
    b. In hydrologic settings where low hydraulic conductivity is expected, water levels should be measured at 1 to 2 second intervals. This measurement frequency should be selected for silts and clays.
5. If applicable, program the barologger to record barometric pressure. The barologger should be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.
6. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally, the transducer should be 5 to 10 feet lower than the maximum depth of the slug testing equipment, not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom, as silt can clog the pressure transducer. Clamp the pressure transducer cable to the well casing or other static object.
7. View the measured water level in real time on the laptop computer. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer should be permitted to equilibrate to groundwater temperatures to ensure accurate water-level measurements.
8. Determine the volume of the water slug. A general guideline is that initial displacements are generally between 1 and 3 feet, but should depend on the anticipated response (i.e., larger initial displacements should be chosen for formations with high hydraulic conductivity, smaller initial displacements can be used for formations with low hydraulic conductivity).

Slug Volume (gal)	Slug Volume (mL)	Casing Diameter (in)	Theoretical Initial Displacement (ft)
0.25	946	2	1.56
0.5	1893	2	3.13
1	3785	2	6.25
0.5	1893	4	0.77

Slug Volume (gal)	Slug Volume (mL)	Casing Diameter (in)	Theoretical Initial Displacement (ft)
1	3785	4	1.54
2	7570	4	3.08
1	3785	6	0.68
2	7570	6	1.36
3	11355	6	2.04

Notes:
gal = gallons (U.S.)
mL = milliliters
in = inches
ft = feet

9. Measure the slug volume and place in a container that is easy to quickly pour from. Note the measured volume.
10. Insert the wide mouth funnel into the well casing.
11. Quickly pour the slug through the funnel and into the well. Note the approximate time required to insert the slug.
12. Observe the water level response on the laptop computer or use water level meter.
13. Measure depth to water, being careful not to interfere with the pressure transducer cable. Several manual depth to water measurements should be made throughout the test.
14. Allow sufficient time for water level to recover to static level. If completing one test, then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test should be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.
15. Repeat steps 9 through 14.
16. Save all data files to the laptop, backup to flash drive, and finalize any field notes.
17. Review the data collected to determine the reasonableness of the preliminary results. The observation of apparently anomalous results should be discussed with senior project staff prior to additional testing or leaving the field site. The water level record for each test should show static conditions, the insertion or removal of the slug(s), and the water level response. Make notes on the field form and notebook concerning any irregularities.
18. Decontaminate all down-well equipment in accordance with project plans.

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ATTACHMENT C

TGI: Baildown Testing

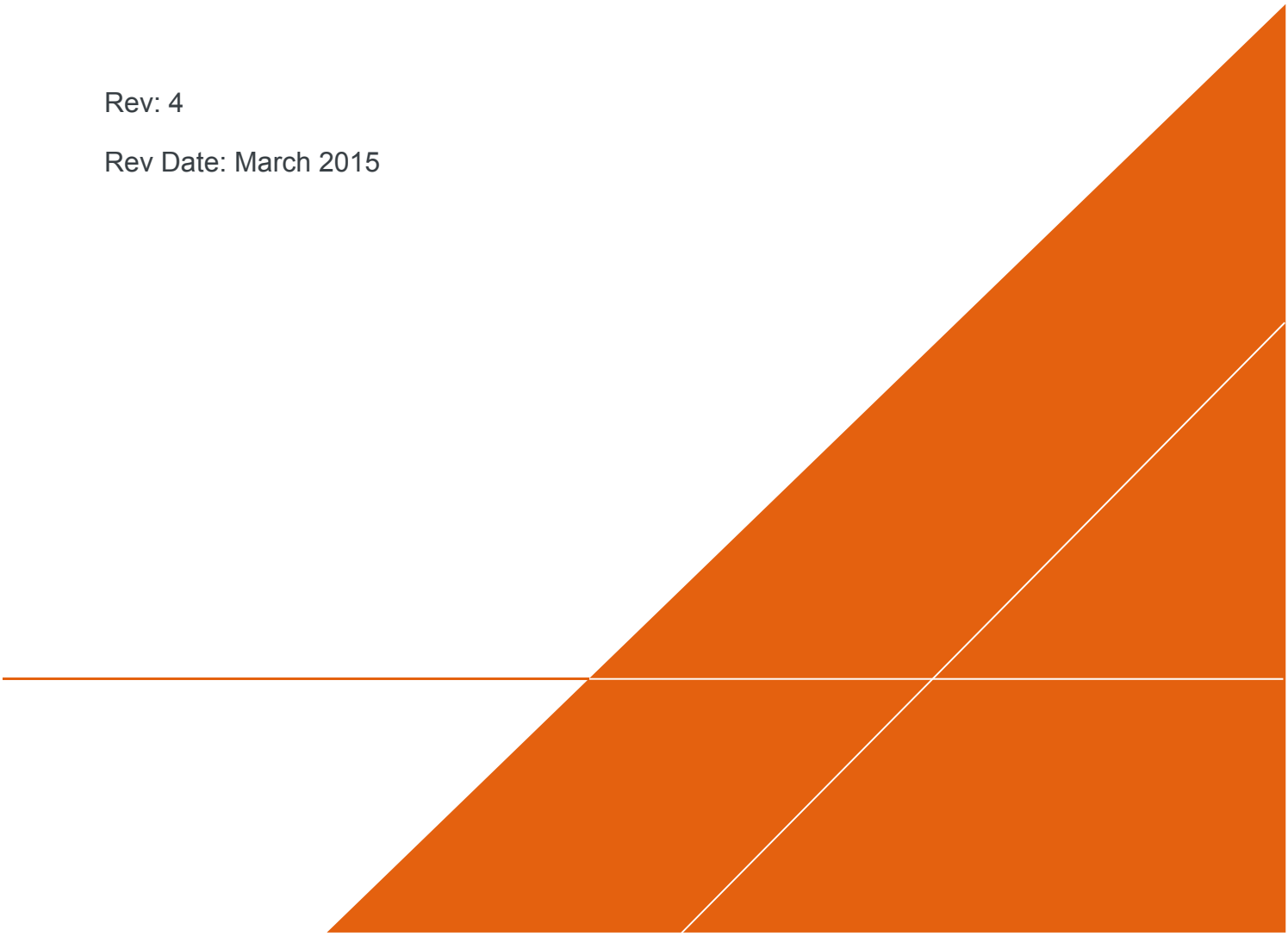
ARCADIS Design & Consultancy for natural and built assets

Slug Testing TGI Attachment C

TECHNICAL GUIDANCE INSTRUCTIONS FOR BAILDOWN TESTING

Rev: 4

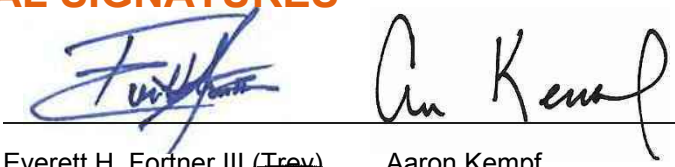
Rev Date: March 2015



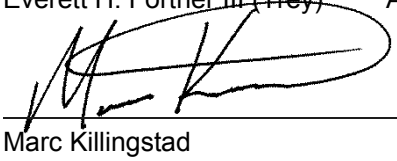
TGI VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by

APPROVAL SIGNATURES

Prepared by:  Date: March 5, 2015

Everett H. Fortner III (Trey) Aaron Kempf

Reviewed by:  Date: March 5, 2015

Marc Killingstad

1 SCOPE AND APPLICATION

A bailer is used to remove a volume of water (slug) to complete rising-head tests. A bailer removes water from a well in a near-instantaneous manner. The water level response is observed using a pressure transducer.

2 EQUIPMENT LIST

The following materials should be available during slug testing using a water slug:

- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Bailers of known size/capacity
- Pressure transducer and cable
- Graduated cylinder or similar measuring device
- Pressure transducer software
- Laptop computer and/or data transfer device
- Rope or cables (for deep wells) (chemical resistant, low stretch is optimal)
- Water level meter
- Measuring tape
- Spring-loaded clamps and zip ties
- Decontamination equipment
- Slug test field forms
- Field notebook
- Waterproof marker.

3 PROCEDURE

1. Select a bailer according to a target initial displacement using the table below. A general guideline is that initial displacements are between 1 and 3 feet, but should depend on the anticipated response (i.e., larger initial displacements should be chosen for formations with high hydraulic conductivity, smaller initial displacements can be used for formations with low hydraulic conductivity).

Bailer Volume (gal)	Bailer Volume (mL)	Casing Diameter (in)	Theoretical Initial Displacement (ft)
0.25	946	2	1.56
0.5	1893	2	3.13
1	3785	2	6.25
0.5	1893	4	0.77
1	3785	4	1.54
2	7570	4	3.08
1	3785	6	0.68
2	7570	6	1.36
3	11355	6	2.04

Notes:
gal = gallons, U.S. liquid
ml = milliliters
in = inches
ft = feet

2. Decontaminate all down-well equipment: pressure transducer and cable, slug(s), rope or cable, water level meter in accordance with project-specific requirements. In general, wells should be tested from least contaminated to more contaminated, if possible or applicable.
3. Measure depth to water and well total depth. Determine the water column length.
    a. Multiple depth to waters should be measured and any trends should be noted.
    b. The "static" depth to water should be representative of the water level after the well equalizes with atmosphere.
4. Measure depth to water and well total depth. Total depth should be taken using a weighted tag line. Determine the water column length.
5. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
6. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory should be used to select the highest measurement frequency possible.
    a. In hydrologic settings where high hydraulic conductivity is expected, water levels should be measured at 0.5-second intervals, or the highest frequency available. This measurement frequency should be selected for gravels and sands.
    b. In hydrologic settings where low hydraulic conductivity is expected, water levels should be measured at 1- to 2-second intervals. This measurement frequency should be selected for silts and clays.
7. If applicable, program the barologger to record barometric pressure. The barologger should be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.

8. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally, the transducer should be 5 to 10 feet lower than the maximum depth of the slug testing equipment not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom, as silt can clog the pressure transducer. Clamp the pressure transducer cable to the well casing or other static object.
9. View the measured water level in real time on the laptop computer. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer should be permitted to equilibrate to groundwater temperatures to ensure accurate water-level measurements.
10. Measure the bailer and rope assembly length and mark the rope at a length as follows:

    Rope Mark #1 = Depth to Potentiometric Surface from TOC

    Rope Mark #2 = Depth to Potentiometric Surface from TOC + Length of Bailer + Safety Factor (Safety Factor = 10% of the Length of Slug)

    When deployed, Rope Mark #2 should ensure that the bailer is fully submerged. If a sufficient water column is not available to obtain a full bailer, measure the volume removed upon removal.

11. Slowly insert the bailer into the well and stop just above the potentiometric surface Rope Mark #1.
12. With slack in the rope and the bailer being suspended above the water column, lower the bailer and place the Rope Mark #2 at the top of casing. Clamp the non-bailer end of the rope to a static object to keep in place.
13. Wait for water level to equilibrate using response from the laptop computer, and/or data transfer device, or from water level meter.
14. Quickly remove the bailer from the water column and carefully pull it to surface. Pour the removed water into an empty bucket.
15. Observe the water level response on the laptop computer and/or measure depth to water, being careful not to interfere with the pressure transducer cable. Several manual depth to water measurements should be made throughout the test.
16. Allow sufficient time for water level to recover to static level. If completing one test, then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test should be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.
17. Measure the volume of water removed by the bailer that was poured into the empty bucket using a graduated cylinder.
18. Repeat rising-head slug tests for data reproducibility. If possible, complete a third test with a bailer or multiple bailers connected in series that equates to twice the volume as the original.
19. Save all data files, backup on flash drive, and finalize any field notes.
20. Review the data collected to determine the reasonableness of the preliminary results. The observation of apparently anomalous results should be discussed with senior project staff prior to additional testing or leaving the field site. The water level record for each test should show static conditions, the insertion or removal of the slug(s), and the water level response. Make notes on the field form and notebook concerning any irregularities.
21. Decontaminate all down-well equipment.

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ATTACHMENT D

TGI: Pneumatic Slug Testing

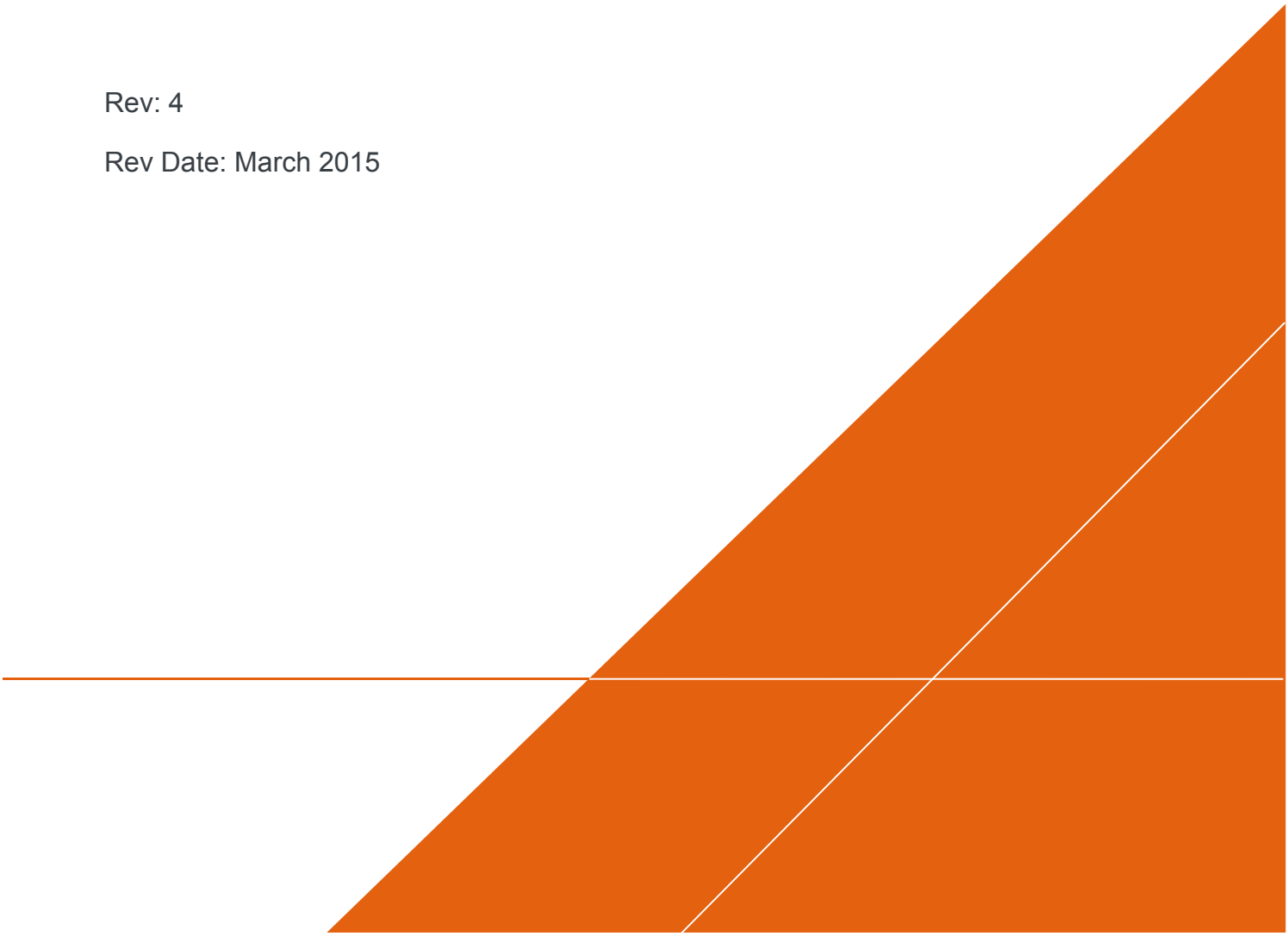
ARCADIS Design & Consultancy for natural and built assets

Slug Testing TGI Attachment D

TECHNICAL GUIDANCE INSTRUCTIONS FOR PNEUMATIC SLUG TESTING

Rev: 4

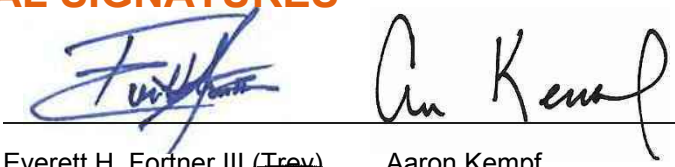
Rev Date: March 2015



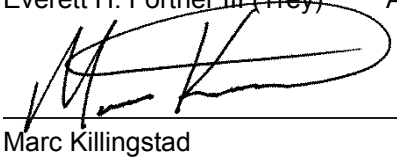
TGI VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by

APPROVAL SIGNATURES

Prepared by:  Date: March 5, 2015

Everett H. Fortner III (Trey) Aaron Kempf

Reviewed by:  Date: March 5, 2015

Marc Killingstad

1 SCOPE AND APPLICATION

Pneumatic slug tests are conducted by sealing the well head and applying air pressure to depress the water level. As air pressure is increased in the well, the water level falls until the water pressure and the air pressure return to equilibrium. After the water level is stable, air is released from the sealed well head by opening an air release valve. The water level recovery (i.e., rising head) typically produces very high quality data with little interference. A pressure transducer is used to monitor and record the change of the water level in the well during the pneumatic slug test.

2 EQUIPMENT LIST

The following materials should be available during slug testing using a water slug:

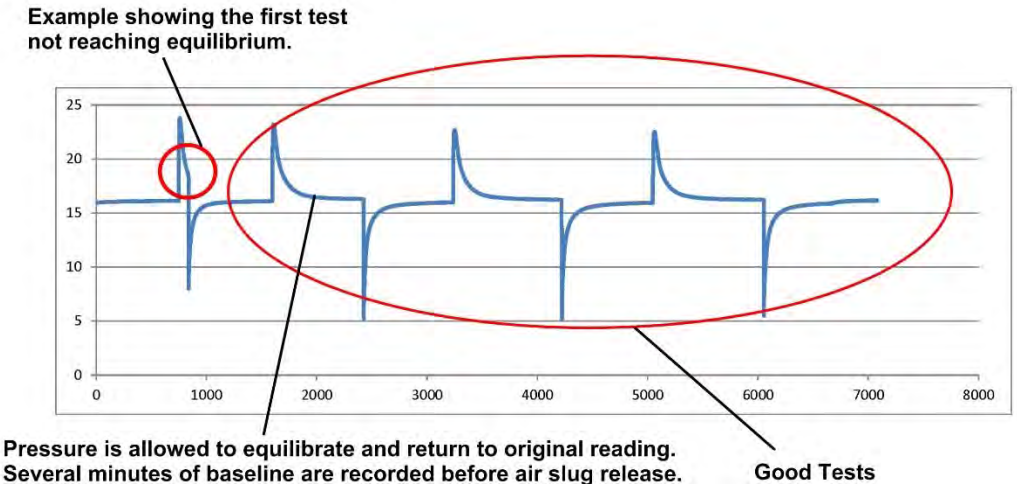
- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Pneumatic slug test manifold (see attached Figure 1 for suggested configuration)
- Pressure transducer and cable
- Pressure transducer software
- Air pressurization source (compressed or pump) and appropriate hoses
- Leak prevention supplies (Teflon pipe sealant, plumbers putty or similar product)
- Laptop computer and/or data transfer device
- Water level meter
- Measuring tape
- Decontamination equipment
- Slug test field forms
- Field notebook
- Waterproof marker.

3 PROCEDURE

1. Decontaminate all down-well equipment: pressure transducer, cable, and water level meter in accordance with project-specific requirements. In general, wells should be tested from least contaminated to more contaminated, if possible or applicable.
2. Measure depth to water and well total depth. Determine the water column length.
    a. Multiple depth to waters should be measured and any trends should be noted.
    b. The "static" depth to water should be representative of the water level after the well equalizes with atmosphere.

3. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
4. Attach the pneumatic slug test manifold onto the top of the well casing. Tighten the rubber connector to ensure an airtight seal.
5. If applicable, program the barologger to record barometric pressure. The barologger should be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.
6. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory should be used to select the highest measurement frequency possible.
    a. In hydrologic settings where high hydraulic conductivity is expected, water levels should be measured at 0.5-second intervals, or the highest frequency available. This measurement frequency should be selected for gravels and sands.
    b. In hydrologic settings where low hydraulic conductivity is expected, water levels should be measured at 1- to 2-second intervals. This measurement frequency should be selected for silts and clays.
7. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally, the transducer should be 5 to 10 feet lower than the maximum depth of the slug testing equipment not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom as silt can clog the pressure transducer. Clamp the pressure transducer cable to the well casing or other static object.
8. View the measured water level in real time on the laptop computer. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer should be permitted to equilibrate to groundwater temperatures to ensure accurate water-level measurements.
9. Re-measure the depth to water
10. Close the air release valve.
11. Close the inlet air valve with the pressure regulator closed.
12. Verify that the incoming pressure is less than safe operating pressure of the manifold pressure regulator (<10 psi is necessary) before attaching air hose (not applicable for hand pump).
13. Attach air hose and open regulator to verify incoming pressure (not applicable for hand pump).
14. Close regulator and open the inlet air valve.
15. Slowly open the pressure regulator to pressurize well head and depress water level a sufficient distance without lowering the head below the top of the well screen (2.31 feet of water is equal to 1 psi). Keep to the rule of thumb of 1 to 3 feet displacement and follow best practices with two duplicate tests and a third test with double the displacement. Larger displacements may be appropriate for high conductivity formations. Begin with a low pressure and gradually increase the pressure in order to obtain the desired displacement. DO NOT OVER PRESSURIZE THE WELL (do not exceed ~2 psi). If using a hand pump, pressurize the well head with pump with regulator open.
16. Close the regulator and leak check the system with leak detection fluid and fix any leaks. If the leak is very slow, or down the well, the regulator may be used to maintain a constant pressure head.
17. Check the pressure transducer response and air pressure to verify system is stable. This may take a period of time, as the pressure transducer is equalizing to both the pressurized atmosphere in the well and the displaced water column (see below figure). Stabilization is

reached once the reported pressure returns to near the original pressure. If it is stable proceed to the next step, if not check the seals.



18. Record a baseline pressure for a minimum of 3 minutes. Record data on the field form.
19. Close inlet valve and quickly open the release valve to initiate the test.
20. Allow sufficient time for water level to recover to static level. If completing one test (just a falling head test or just a rising head test), then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test should be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.
21. Save all data files to the laptop, backup on flash drive or by emailing, and finalize any field notes.
22. Review the data collected to determine the reasonableness of the preliminary results. The observation of apparently anomalous results should be discussed with senior project staff prior to proceeding. The water level record for each test should show static conditions, pressurization of the well column, and the recovery response. Make notes on the field form and notebook concerning any irregularities.
23. Decontaminate all down-well equipment.

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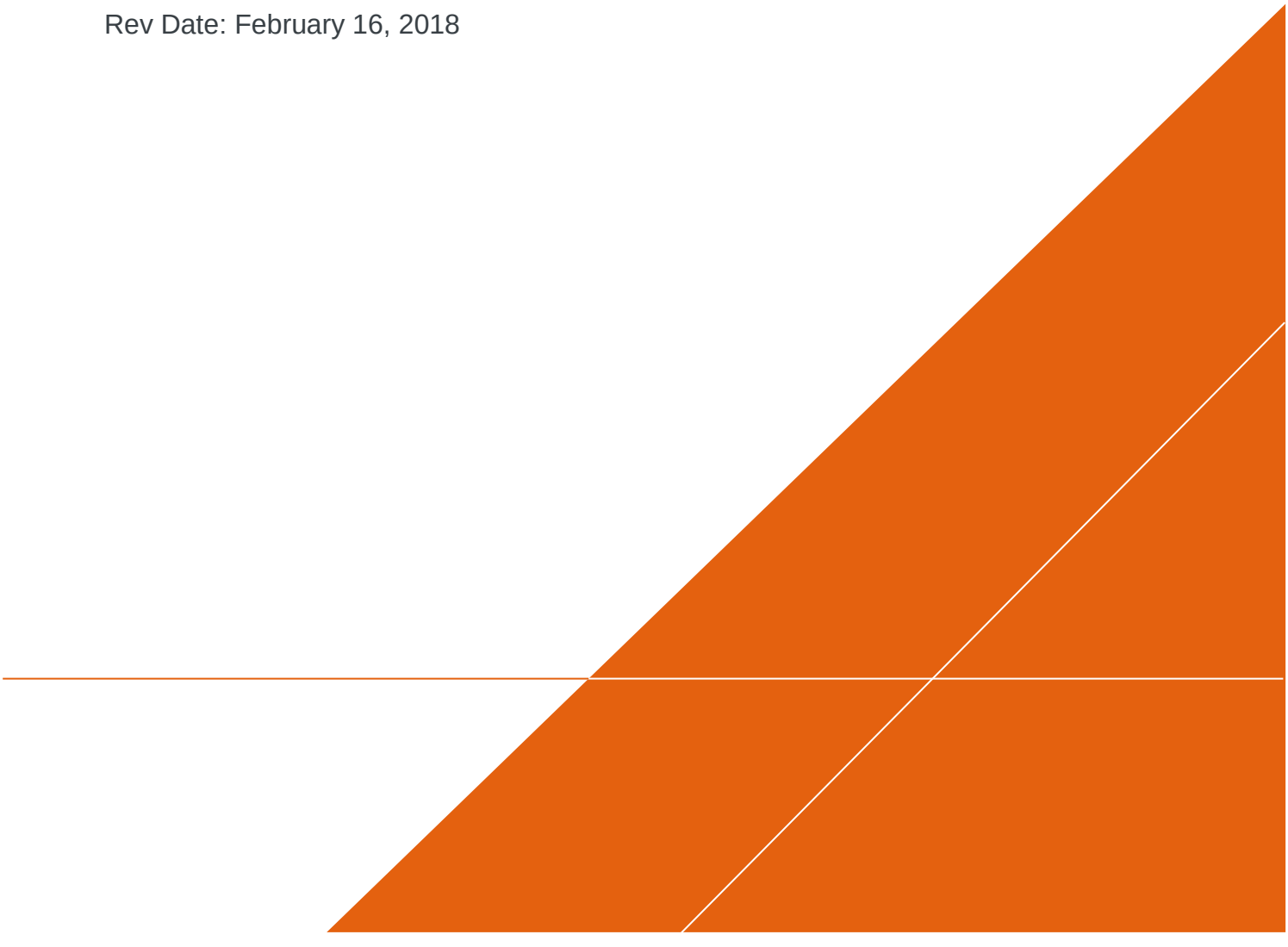
RI-SOP-7

ARCADIS Design & Consultancy for natural and built assets

TGI - SOIL DESCRIPTION

Rev: #2

Rev Date: February 16, 2018



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	May 20, 2008	17	Original SOP	Joe Quinnan Joel Hunt
1	September 2016	15	Updated to TGI	Nick Welty Patrick Curry
2	February 16, 2018	15	Updated descriptions, attachments and references in text	Nick Welty Patrick Curry

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APPROVAL SIGNATURES

Prepared by:		June 30, 2017
	Patrick Curry, PG	Date:
Technical Expert Reviewed by:		June 30, 2017
	Nicklaus Welty, PG	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Arcadis Technical Guidance Instruction (TGI) describes proper soil description procedures. This TGI should be followed for unconsolidated material unless there is an established client-required specific procedure or regulatory-required specific procedure. In cases where there is a required specific procedure, it should be followed and should be referenced and/or provided as an appendix to reports that include soil classifications and/or boring logs. When following a required non-Arcadis procedure, additional information required by this TGI should be included in field notes with client approval.

This TGI has been developed to emphasize field observation and documentation of details required to:

- make hydrostratigraphic interpretations guided by depositional environment/geologic settings;
- provide information needed to understand the distribution of constituents of concern; properly design wells, piezometers, and/or additional field investigations; and develop appropriate remedial strategies.

This TGI incorporates elements from various standard systems such as ASTM D2488-06, Unified Soil Classification System, Burmister and Wentworth. However, none of these standard systems focus specifically on contaminant hydrogeology and remedial design. Therefore, although each of these

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systems contain valuable guidance and information related to correct descriptions, strict application of these systems can omit information critical to our clients and the projects that we perform.

This TGI does not address details of health and safety; drilling method selection; boring log preparation; sample collection; or laboratory analysis. Refer to other Arcadis procedure, guidance, and instructional documents, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan (HASP), as appropriate.

3 PERSONNEL QUALIFICATIONS

Soil descriptions should only be performed by Arcadis personnel or authorized sub-contractors with a degree in geology or a geology-related discipline. Field personnel will complete training on the Arcadis soil description TGI in the office and/or in the field under the guidance of an experienced field geologist with at least 2 years of prior experience applying the Arcadis soil description method.

4 EQUIPMENT LIST

The following equipment should be taken to the field to facilitate soil descriptions:

- field book, field forms or PDA to record soil descriptions;
- field book for supplemental notes;
- this TGI for Soil Descriptions and any project-specific procedure, guidance, and/or instructional documents (if required);
- field card showing Wentworth scale;
- Munsell® soil color chart;
- tape measure divided into tenths of a foot;
- stainless steel knife or spatula;
- hand lens;
- water squirt bottle;
- jar with lid;
- personal protective equipment (PPE), as required by the HASP; and
- digital camera

5 CAUTIONS

Drilling and drilling-related hazards including subsurface utilities are discussed in other procedure documents and site-specific HASPs and are not discussed herein.

Soil samples may contain hazardous substances that can result in exposure to persons describing soils. Routes for exposure may include dermal contact, inhalation and ingestion. Refer to the project specific HASP for guidance in these situations.

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6 HEALTH AND SAFETY CONSIDERATIONS

Field activities associated with soil sampling and description will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Know what hazardous substances may be present in the soil and understand their hazards. Always avoid the temptation to touch soils with bare hands, detect odors by placing soils close to your nose, or tasting soils.

7 PROCEDURE

1. Select the appropriate sampling method to obtain representative samples in accordance with the selected sub-surface exploration method, e.g. split-spoon or Shelby sample for hollow-stem drilling, acetate sleeves for direct push, bagged core for sonic drilling, etc.
2. Proceed with field activities in required sequence. Although completion of soil descriptions is often not the first activity after opening sampler, identification of stratigraphic changes is often necessary to select appropriate intervals for field screening and/or selection of laboratory samples.
3. Set up boring log field sheet.
    - Drillers in both the US and Canada generally work in feet due to equipment specifications. Use the Arcadis standard boring log form (**Attachment A**).
    - The preferred boring log includes a graphic log of the principal soil component to support quick visual evaluation of grain size. The purpose of the graphic log is to quickly assess relative soil permeability. Note, for poorly sorted soils (e.g. glacial till), the principal component may not correlate to permeability of the sample. In this case, the geologist should use best judgement to graph overall soil type consistent with relative soil permeability. For example, for a dense sand/silt/clay till, the graphic log would reflect the silt/clay, rather than sand.
    - Record depths along the left-hand side at a standard scale to aid in the use of this tool. See an example completed boring log (**Attachment B**).
4. Examine each soil core (this is different than examining each sample selected for laboratory analysis), and record the following for each stratum:
    - depth interval;
    - principal component with descriptors, as appropriate;
    - amount and identification of minor component(s) with descriptors as appropriate;
    - moisture;
    - consistency/density;
    - color; and
    - additional description or comments (recorded as notes).
5. At the end of the boring, record the amount of drilling fluid used (if applicable) and the total depth logged.

The above is described more fully below.

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DEPTH

To measure and record the depth below ground surface (bgs) of top and bottom of each stratum, the following information should be recorded.

1. Measured depth to the top and bottom of sampled interval. Use starting depth of sample based upon measured tool length information and the length of sample interval.
2. Length of sample recovered, not including slough (material that has fallen into hole from previous interval), expressed as fraction with length of recovered sample as numerator over length of sampled interval as denominator (e.g. 14/24 for 14 inches recovered from 24-inch sampling interval that had 2 inches of slough discarded).
3. Thickness of each stratum measured sequentially from the top of recovery to the bottom of recovery.
4. Any observations of sample condition or drilling activity that would help identify whether there was loss from the top of the sampling interval, loss from the bottom of the sampling interval, or compression of the sampling interval. Examples: 14/24, gravel in nose of spoon; or 10/18 bottom 6 inches of spoon empty.

DETERMINATION OF COMPONENTS

Obtain a representative sample of soil from a single stratum. If multiple strata are present in a single sample interval, each stratum should be described separately. More specifically, if the sample is from a 2-foot long split-spoon where strata of coarse sand, fine sand and clay are present, then the resultant description should be of the three individual strata unless a combined description can clearly describe the interbedded nature of the three strata. Example: Fine Sand with interbedded lenses of Silt and Clay, ranging between 1 and 3 inches thick.

Identify principal component and express volume estimates for minor components on logs using the following standard modifiers.

Modifier	Percent of Total Sample (by volume)
and	36 - 50
some	21 - 35
little	10 - 20
trace	<10

Determination of components is based on using the Udden-Wentworth particle size classification (see below) and measurement of the average grain size diameter. Each size grade or class differs from the next larger grade or class by a constant ratio of ½. Due to visual limitations, the finer classifications of Wentworth's scale cannot be distinguished in the field and the subgroups are not included. Visual determinations in the field should be made carefully by comparing the sample to the Soil Description Field Guide (**Attachment C**) that shows Udden-Wentworth scale or by measuring with a ruler. Use of field sieves is encouraged to assist in estimating percentage of coarse grain sizes. Settling test or wash method (Appendix X4 of ASTM D2488) is encouraged for determining presence and estimating percentage of clay and silt. Note that "gravel" is not an Udden-Wentworth size class.

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Udden-Wenworth Scale Modified Arcadis, 2008			
Size Class	Millimeters	Inches	Standard Sieve #
Boulder	256 – 4096	10.08+	
Large cobble	128 - 256	5.04 -10.08	
Small cobble	64 - 128	2.52 – 5.04	
Very large pebble	32 – 64	0.16 - 2.52	
Large pebble	16 – 32	0.63 – 1.26	
Medium pebble	8 – 16	0.31 – 0.63	
Small pebble	4 – 8	0.16 – 0.31	No. 5 +
Granule	2 – 4	0.08 – 0.16	No.5 – No.10
Very coarse sand	1 -2	0.04 – 0.08	No.10 – No.18
Coarse sand	½ - 1	0.02 – 0.04	No.18 - No.35
Medium sand	¼ - ½	0.01 – 0.02	No.35 - No.60
Fine sand	1/8 -¼	0.005 – 0.1	No.60 - No.120
Very fine sand	1/16 – 1/8	0.002 – 0.005	No. 120 – No. 230
Silt (subgroups not included)	1/256 – 1/16	0.0002 – 0.002	Not applicable (analyze by pipette or hydrometer)
Clay (subgroups not included	1/2048 – 1/256	.00002 – 0.0002	

Identify components as follows. Remove particles greater than very large pebbles (64-mm diameter) from the soil sample. Record the volume estimate of the greater than very large pebbles. Examine the sample fraction of very large pebbles and smaller particles and estimate the volume percentage of the pebbles, granules, sand, silt and clay. Use the jar method, visual method, and/or wash method (Appendix X4 of ASTM D2488) to estimate the volume percentages of each category.

Determination of actual dry weight of each Udden-Wentworth fraction requires laboratory grain-size analysis using sieve sizes corresponding to Udden-Wentworth fractions and is highly recommended to determine grain-size distributions for each hydrostratigraphic unit.

Lab or field sieve analysis is advisable to characterize the variability and facies trends within each hydrostratigraphic unit. Field sieve-analysis can be performed on selected samples to estimate dry weight fraction of each category using ASTM D2488 Standard Practice for Classification of Soils for Engineering Purposes as guidance, but replace required sieve sizes with the following Udden-Wentworth set: U.S. Standard sieve mesh sizes 6; 12; 20; 40; 70; 140; and 270 to retain pebbles; granules; very coarse sand; coarse sand; medium sand; fine sand; and very fine sand, respectively.

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PRINCIPAL COMPONENT

The principal component is the size fraction or range of size fractions containing the majority of the volume. Examples: the principal component in a sample that contained 55% pebbles would be “Pebbles”; or the principal component in a sample that was 20% fine sand, 30% medium sand and 25% coarse sand would be “Sand, fine to coarse” or for a sample that was 40% silt and 45% clay the principal component would be “Clay and Silt”. Shade the boxes on the graphic log (**Attachment A**) up to and including the box with the principal component. The purpose of the graphical log is to provide a relative estimate of permeability. As noted above, for poorly sorted soils such as glacial till, the principal component may not correlate to permeability of the sample. In this case, the geologist should use best judgement to graph overall soil type consistent with relative soil permeability.

Include appropriate descriptors with the principal component. These descriptors vary for different particle sizes as follows.

Angularity – Describe the angularity for very coarse sand and larger particles in accordance with the table below (ASTM D-2488-06). Figures showing examples of angularity are available in ASTM D-2488-06 and the Arcadis Soil Description Field Guide.

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces.
Sub-angular	Particles are similar to angular description but have rounded edges.
Sub-rounded	Particles have nearly plane sides but have well-rounded corners and edges.
Rounded	Particles have smoothly curved sides and no edges.

Plasticity – Describe the plasticity for silt and clay based on observations made during the following test method (ASTM D-2488-06).

- As in the dilatancy test below, select enough material to mold into a ball about ½ inch (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
- Shape the test specimen into an elongated pat and roll by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation. Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble when the soil is near the plastic limit.

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Description	Criteria
Non-plastic	A 1/8-inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled, and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

Dilatancy – Describe the dilatancy for silt and silt-sand mixtures using the following field test method (ASTM D-2488-06).

- From the specimen select enough material to mold into a ball about ½ inch (12 mm) in diameter. Mold the material adding water if necessary, until it has a soft, but not sticky, consistency.
- Smooth the ball in the palm of one hand with a small spatula.
- Shake horizontally, striking the side of the hand vigorously with the other hand several times.
- Note the reaction of water appearing on the surface of the soil.
- Squeeze the sample by closing the hand or pinching the soil between the fingers, and not the reaction as none, slow, or rapid in accordance with the table below. The reaction is the speed with which water appears while shaking and disappears while squeezing.

Description	Criteria
None	No visible change in the specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

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Note that silt and silt-sand mixtures will be non-plastic and display dilatancy. Clay mixtures will have some degree of plasticity but do not typically react to dilatancy testing. Therefore, the tests outlined above can be used to differentiate between silt dominated and clay dominated soils.

MINOR COMPONENT(S)

The minor component(s) are the size fraction(s) containing less than 50% volume. Example: the identified components are estimated to be 60% medium sand to granules, 25% silt and clay; 15 % pebbles – there are two identified minor components: silt and clay; and pebbles.

Include a standard modifier to indicate percentage of minor components (see Table on Page 6) and the same descriptors that would be used for a principal component. Plasticity should be provided as a descriptor for clay and clay mixtures. Dilatancy should be provided for silt and silt mixtures. Angularity should be provided as a descriptor for pebbles and coarse sand. For the example above, the minor constituents with modifiers could be: some silt and clay, low plasticity; little medium to large pebbles, sub-round.

SORTING

Sorting is the opposite of grading, which is a commonly used term in the USCS or ASTM methods to describe the uniformity of the particle size distribution in a sample. Well-sorted samples are poorly graded and poorly sorted samples are well graded. Arcadis prefers the use of sorting for particle size distributions and grading to describe particle size distribution trends in the vertical profile of a sample or hydrostratigraphic unit because of the relationship between sorting and the energy of the depositional process. For soils with sand-sized or larger particles, sorting should be determined as follows:

Well sorted – the range of particle sizes is limited (e.g. the sample is comprised of predominantly one or two grain sizes).

Poorly sorted – a wide range of particle sizes are present.

You can also use sieve analysis to estimate sorting from a sedimentological perspective; sorting is the statistical equivalent of standard deviation. Smaller standard deviations correspond to higher degree of sorting (see Remediation Hydraulics, 2008).

MOISTURE

Moisture content should be described for every sample since increases or decreases in water content is critical information. Moisture should be described in accordance with the table below (percentages should not be used unless determined in the laboratory).

Description	Criteria
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet (Saturated)	Visible free water, soil is usually below the water table.

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CONSISTENCY or DENSITY

This can be determined by standard penetration test (SPT) blow counts (ASTM D-1586) or field tests in accordance with the tables below. When drilling with hollow-stem augers and split-spoon sampling, the SPT blow counts and N-value is used to estimate density. The N-value is the blows per foot for the 6" to 18" interval. Example: for 24-inch spoon, recorded blows per 6-inch interval are: 4/6/9/22. Since the second interval is 6" to12", the third interval is 12" to 18", the N value is 6+9, or 15. Fifty blow counts for less than 6 inches is considered refusal. In recent years, more common drilling methods include rotary-sonic or direct push. When blow counts are not available, density is determined using a thumb test. Note however, the thumb test only applies to fine-grained soils.

Fine-grained soil – Consistency

Description	Criteria
Very soft	N-value < 2 or easily penetrated several inches by thumb.
Soft	N-value 2-4 or easily penetrated one inch by thumb.
Medium stiff	N-value 9-15 or indented about ¼ inch by thumb with great effort.
Very stiff	N-value 16-30 or readily indented by thumb nail.
Hard	N-value > than 30 or indented by thumbnail with difficulty

Coarse-grained soil – Density

Description	Criteria
Very loose	N-value 1- 4
Loose	N-value 5-10
Medium dense	N-value 11-30
Dense	N-value 31- 50
Very dense	N-value >50

COLOR

Color should be described using simple basic terminology and modifiers based on the Munsell system. Munsell alpha-numeric codes are required for all samples. If the sample contains layers or patches of varying colors this should be noted and all representative colors should be described. The colors should be described for moist samples. If the sample is dry it should be wetted prior to comparing the sample to the Munsell chart.

ADDITIONAL COMMENTS (NOTES)

Additional comments should be made where observed and should be presented as notes with reference to a specific depth interval(s) to which they apply. Some of the significant information that may be observed includes the following.

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- Odor - You should not make an effort to smell samples by placing near your nose since this can result in unnecessary exposure to hazardous materials. However, odors should be noted if they are detected during the normal sampling procedures. Odors should be based upon descriptors such as those used in NIOSH “Pocket Guide to Chemical Hazards”, e.g. “pungent” or “sweet” and should not indicate specific chemicals such as “phenol-like” odor or “BTEX” odor.
- Structure
- Bedding planes (laminated, banded, geologic contacts).
- Presence of roots, root holes, organic material, man-made materials, minerals, etc.
- Mineralogy
- Cementation
- NAPL presence/characteristics, including sheen (based on client-specific guidance).
- Reaction with HCl - typically only used for special soil conditions, such as caliche environments.
- Origin, if known (Lacustrine; Fill; etc.).

EXAMPLE DESCRIPTIONS



51.4 to 54.0’ CLAY, some silt, medium to high plasticity; trace small to large pebbles, sub-round to sub-angular up to 2” diameter; moist, stiff, dark grayish brown (10 YR 4/2) NOTE: Lacustrine; laminated 0.1 to 0.2” thick, laminations brownish yellow (10 YR 4/3).



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32.5 to 38.0’ SAND, medium to very coarse, sub-round to sub-angular; little granule and pebble, trace silt; poorly sorted, wet, grayish brown (10 YR 5/2).

Unlike the first example where a density of cohesive soils could be estimated, this rotary-sonic sand and pebble sample was disturbed during drilling (due to vibrations in a loose sand and pebble matrix) so no density description could be provided. Neither sample had noticeable odor so odor comments were not included.

The standard generic description order is presented below.

- Depth
- Principal Components
  - Angularity for very coarse sand and larger particles
  - Plasticity for silt and clay
  - Dilatancy for silt and silt-sand mixtures
- Minor Components
- Sorting
- Moisture
- Consistency or Density
- Color
- Additional Comments

8 WASTE MANAGEMENT

Project-specific requirements should be identified and followed. The following procedures, or similar waste management procedures are generally required.

Water generated during cleaning procedures will be collected and contained onsite in appropriate containers for future analysis and appropriate disposal. PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures and soil sampling/handling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

Soil materials will be placed in sealed 55-gallon steel drums or covered roll-off boxes and stored in a secured area. Once full, the material will be analyzed to determine the appropriate disposal method.

9 DATA RECORDING AND MANAGEMENT

Upon collection of soil samples, the soil sample should be logged on a standard boring log and/or in the field log book depending on Data Quality Objectives (DQOs) for the task/project. The preferred standard boring log is presented below and is included as **Attachment A**.

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The general scheme for soil logging entries is presented above; however, depending on task/project DQOs, specific logging entries that are not applicable to task/project goals may be omitted at the project manager's discretion. In any case, use of a consistent logging procedure is required.

Completed logs and/or logbook will be maintained in the task/project field records file. Digital photographs of typical soil types observed at the site and any unusual features should be obtained whenever possible. All photographs should include a ruler or common object for scale. Photo location, depth and orientation must be recorded in the daily log or log book and a label showing this information in the photo is useful.

10 QUALITY ASSURANCE

Soil descriptions should be completed only by appropriately trained personnel. Descriptions should be reviewed by an experienced field geologist for content, format and consistency. Edited boring logs should be reviewed by the original author to assure that content has not changed.

11 REFERENCES

Arcadis Soil Description Field Guide, 2008.

Munsell® Color Chart – available from Forestry Suppliers, Inc.- Item 77341 "Munsell® Color Soil Color Charts.

Field Gauge Card that Shows Udden-Wentworth scale – available from Forestry Suppliers, Inc. – Item 77332 "Sand Grain Sizing Folder."

ASTM D-1586, Test Method for Penetration Test and Split-Barrel Sampling of Soils.

ASTM D-2488-00, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)

United States Bureau of Reclamation. Engineering Geology Field Manual. United States Department of Interior, Bureau of Reclamation. http://www.usbr.gov/pmts/geology/fieldmap.htm.

Petrology of Sedimentary Rocks, Robert L. Folk, 1980, p. 1-48.

NIOSH Pocket Guide to Chemical Hazards.

Remediation Hydraulics, Fred C. Payne, Joseph A. Quinnan, and Scott T. Potter, 2008, p 59-63.

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ATTACHMENT A

Arcadis Standard Soil Boring Log Form

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SOIL BORING LOG

Boring/Well _____ Project _____ Page ____ of ____

Site Location _____ Drilling Started _____

Total Depth Drilled _____ Feet Hole Diameter _____ inches Drilling Completed _____

Type of Sample or Coring Device _____ Length and Diameter of Coring Device _____ Sampling Interval _____ feet

Drilling Method _____ Drilling Fluid Used _____

Drilling Contractor _____ Driller _____

Prepared By _____ Helper _____

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD		SAND					GRAVEL				**Udden-Wentworth Description:** principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, additional comments
			clay	silt	very fine	fine	medium	coarse	very coarse	granular	pebble	cobble	boulder	

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SOIL BORING LOG (CONT'D)

Boring/Well _____ Prepared By _____ Page ____ of ____

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD		SAND					GRAVEL				Udden-Wentworth Description: principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, addtl. Comments
			clay	silt	very fine	fine	medium	coarse	very coarse	granular	pebble	cobble	boulder	

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ATTACHMENT B

Example of Completed Arcadis Soil Boring Log

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SOIL BORING LOG

Boring/Well SB-01 Project Example Page 1 of 1

Site Location Anytown, North America Drilling Started 6/26/17

Total Depth Drilled 20 Feet Hole Diameter 4.5 inches Drilling Completed 6/26/17

Type of Sample or Coring Device Continuous Length and Diameter of Coring Device 5', 2.25" macrocore Sampling Interval 5 feet

Drilling Method Geoprobe Drilling Fluid Used NA

Drilling Contractor Fibertec Driller Ryan Brown

Prepared By A. DeGrandis Helper Grant Berger

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD: clay	MUD: silt	SAND: very fine	SAND: fine	SAND: medium	SAND: coarse	SAND: very coarse	GRAVEL: granular	GRAVEL: pebble	GRAVEL: cobble	GRAVEL: boulder	Udden-Wentworth Description: principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, additional comments
	0.3	1	X	X	X	X	X							(0.0-4.0) SAND, fine to medium, sub-rounded; little granules to small pebbles, sub-rounded to sub-angular; trace silt; poorly sorted, dry to moist, dark grayish brown (10YR 4/2).
60" (HA)	0.1	2	X	X	X	X	X							
	0.0	3	X	X	X	X	X							
	0.0	4	X	X	X	X	X							
	1.4	5	X	X										NOTE: Ablation till.
	1.7	6	X	X										
	0.9	7	X	X										(4.0-10.0) SILT, non-plastic, rapid dilatancy; dry to moist, soft to medium stiff, gray (10YR 5/1).
52"	1.2	8	X	X										
	0.6	9	X	X										
	0.2	10	X	X										
	0.0	11	X	X	X	X								(10.0-15.5) SAND, fine, sub-rounded; trace silt; well sorted, moist to wet, pale brown (10YR 6/3).
	0.0	12	X	X	X	X								
60"	0.1	13	X	X	X	X								
	0.0	14	X	X	X	X								
	0.0	15	X	X	X	X								NOTE: Wet at 12.0'
	0.2	16	X	X	X	X								
	0.0	17	X	X										(15.5-20.0) CLAY, high plasticity, no dilatancy; little silt; moist, soft to medium stiff, light gray (10YR 7/1) to dark gray (10YR 4/1).
58"	0.0	18	X	X										
	0.0	19	X	X										
	0.0	20	X	X										NOTE: Lacustrine, minor 0.1-0.25" lamination.
														End of boring - 20.0'

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ATTACHMENT C

Arcadis Soil Description Field Guide

ON THE ORIGINAL DRAWING.
USE TO VERIFY FIGURE
REPRODUCTION SCALE

SOIL DESCRIPTION FIELD GUIDE (JUNE 30, 2017; REV. 2.0)

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FINE-GRAINED SOILS

Description	Criteria
	Descriptor - Plasticity
Nonplastic	A 1/8-inch (3mm) thread cannot be rolled at any moisture content.
Low	Thread can barely be rolled, and lump cannot be formed when drier than plastic limit.
Medium	Takes considerable time and rolling to reach plastic limit. Thread cannot be rolled after reaching plastic limit. Lump crumbles when drier than plastic limit.
High	Thread is easy to roll and quickly reaches plastic limit. Thread can be rerolled several times after reaching plastic limit. Lump can be formed without crumbling when drier than plastic limit.
	Descriptor - Dilatancy
No Dilatancy	No visible change when shaken or squeezed.
Slow	Water appears slowly on the surface of soil during shaking and does not disappear or disappears slowly when squeezed.
Rapid	Water appears quickly on surface of soil during shaking and disappears quickly when squeezed.
Minor Components with Descriptors	
	Moisture
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet	Visible free water; soil is usually below the water table. (Saturated)
	Consistency
Very soft	N-value < 2 or easily penetrated several inches by thumb.
Soft	N-value 2-4 or easily penetrated 1 inch by thumb.
Medium stiff	N-value 5-8 or indented about 1/2 inch by thumb with great effort.
Stiff	N-value 9-15 or indented about 1/4 inch by thumb with great effort.
Very stiff	N-value 16-30 or readily indented by thumb nail.
Hard	N-value > than 30 or indented by thumbnail with difficulty.
Color using Munsell	
Geologic Origin (if known)	
Other	

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

10-15 feet CLAY, medium to high plasticity; trace silt; trace small to very large pebbles, subround to subangular up to 2" diameter; moist, stiff, dark grayish brown (10YR 4/2). NOTE: Lacustrine; laminated 0.1 to 0.2" thick, laminations brownish yellow (10YR 4/3).



DESCRIPTION ORDER

Depth Interval
Principal Components with Descriptors
Minor Components with Descriptors
Sorting
Field Moisture Condition
Density/Consistency
Color using Munsell
Geologic Origin (if known)
Other descriptions as NOTES:
- Odor
- Stratigraphy
- Structure
- Sphericity
- Cementation
- Reaction to acid

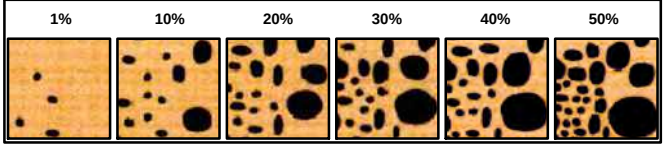
MINOR COMPONENTS % MODIFIERS

Modifier	Percent of Total Sample (by volume)
and	36 - 50
some	21 - 35
little	10 - 20
trace	<10

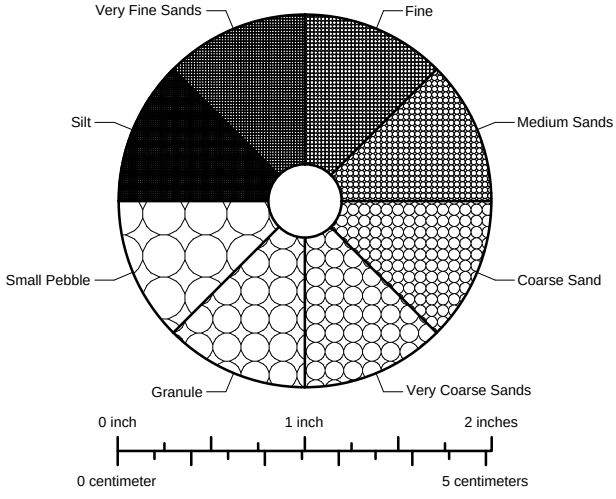
UDDEN-WENTWORTH SCALE

Fraction	Sieve Size	Grain Size	Approximate Scale
Boulder		256 - 4096 mm	Larger than volleyball
Large Cobble		128 - 256 mm	Softball to volleyball
Small Cobble		64 - 128 mm	Pool ball to softball
Very Large Pebble		32 - 64 mm	Pinball to pool ball
Large Pebble		16 - 32 mm	Dime size to pinball
Medium Pebble		8 - 16 mm	Pencil eraser to dime size
Small Pebble	No. 5+	4 - 8 mm	Pea size to pencil eraser
Granule	No. 10 - 5	2 - 4 mm	Rock salt to pea size
Very Coarse Sand	No. 18 - 10	1 - 2 mm	See field gauge card
Coarse Sand	No. 35 -18	0.5 - 1 mm	See field gauge card
Medium Sand	No. 60 - 35	0.25 - 0.5 mm	See field gauge card
Fine Sand	No. 120 - 60	0.125 - 0.25 mm	See field gauge card
Very Fine Sand	No. 230 - 120	0.0625 - 0.125 mm	See field gauge card
Silt and Clay. See SOP for description of fines	Not Applicable	<0.0625 mm	Analyze by pipette or hydrometer

PARTICLE PERCENT COMPOSITION ESTIMATION



GRAPH FOR DETERMINING SIZE OF PARTICLES



FOR COARSE-GRAINED SOILS

Description	Criteria
	Descriptor - Angularity
Angular	Particles have sharp edges and relatively planar sides withunpolished surfaces.
Subangular	Particles are similar to angular but have rounded edges.
Subround	Particles have nearly planar sides but have well-roundedcorners and edges.
Round	Particles have smoothly curved sides and no edges.
Minor Components with Descriptors	
	Sorting Cu= d60/d10
Well Sorted	Near uniform grain-size distribution Cu= 1 to 3.
Poorly Sorted	Wide range of grain size Cu= 4 to 6.
	Moisture
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet	Visible free water; soil is usually below the water table. (Saturated)
	Density
Very loose	N-value 1 - 4
Loose	N-value 5 - 10
Medium Dense	N-value 11 - 30
Dense	N-value 31 - 50
Very dense	N-value >50
Color using Munsell	
Geologic Origin (if known)	
Other	
	Cementation
Weak Cementation	Crumbles or breaks with handling or little finger pressure.
Moderate Cementation	Crumbles or breaks with considerable finger pressure.
Strong Cementation	Will not crumble with finger pressure.
	Reaction with Dilute HCl Solution (10%)
No Reaction	No visible reaction.
Weak Reaction	Some reaction, with bubbles forming slowly.
Strong Reaction	Violent reaction, with bubbles forming immediately.

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

10 -15 feet SAND, medium to very coarse; little granules to medium pebbles, subround to subangular; trace silt; poorly sorted, wet, grayish brown (10YR5/2).



ON THE ORIGINAL DRAWING.
USE TO VERIFY FIGURE
REPRODUCTION SCALE

SOIL DESCRIPTION FIELD GUIDE (JUNE 30, 2017; REV. 2.0)

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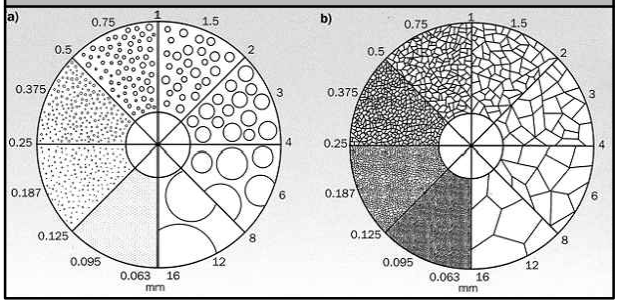
VARIATIONS IN SOIL STRATIGRAPHY

Term	Thickness of Configuration
Parting	0 - to 1/16-inch thickness.
Seam	1/16 - to 1/2-inch thickness.
Layer	1/2 - to 12-inch thickness.
Stratum	> 12-inch thickness.
Pocket	Small erratic deposit, usually less than 1 foot in size.
Varved Clay	Alternating seams or layers of sand, silt, and clay (laminated).
Occasional	≤ 1 foot thick.
Frequent	> 1 foot thick.

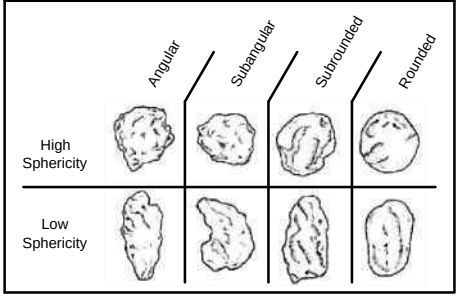
SOIL STRUCTURE DESCRIPTIONS

Term	Description
Homogeneous	Same color and appearance throughout.
Laminated	Alternating layers < 1/4 inch thick.
Stratified	Alternating layers ≥ 1/4 inch thick.
Lensed	Inclusions of small pockets of different materials, such as lenses of sand scattered through a mass of clay; note thickness.
Blocky	Cohesive soil can be broken down into small angular lumps, which resist further breakdown.
Fissured	Breaks along definite planes of fracture with little resistance to fracturing.
Slickensided	Fracture planes appear to be polished or glossy, sometimes striated.

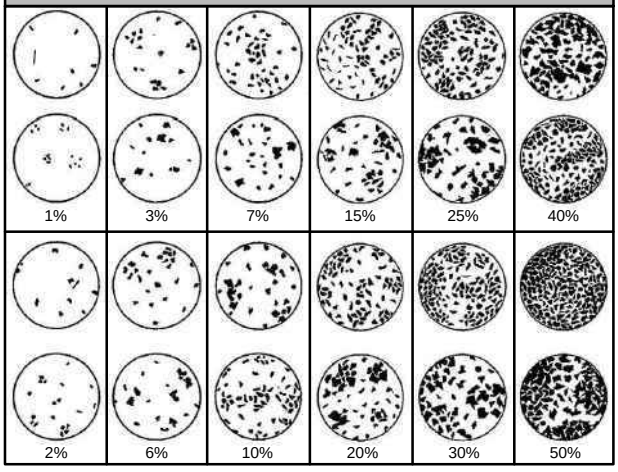
GRAPH FOR DETERMINING SIZE OF PARTICLES



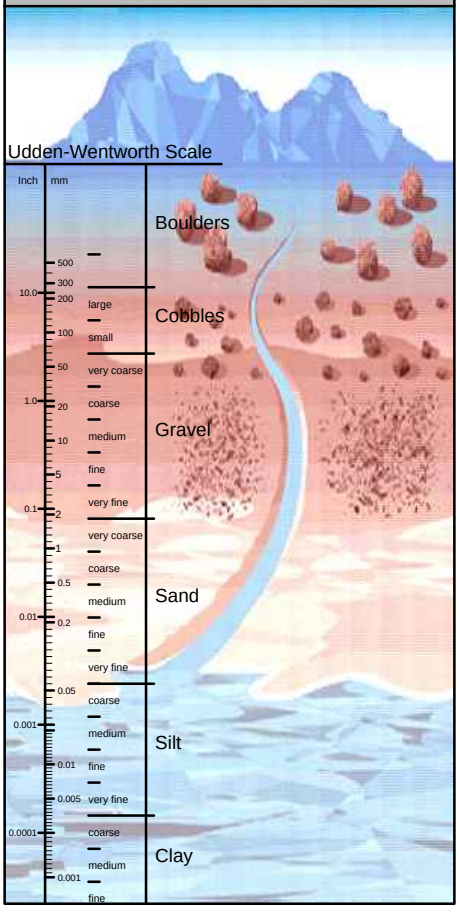
ANGULARITY CHART



PARTICLE PERCENT COMPOSITION ESTIMATION



SORTING



SETTLING TABLE (SILT/CLAY)

Diameter of Particle (mm)	<0.625	<0.031	<0.016	<0.008	<0.004	<0.002	<0.0005
Depth of Withdrawal (cm)	10	10	10	10	5	5	3
Time of Withdrawal	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec
Temperature (Celsius)							
20	00:00:29	00:01:55	00:07:40	00:30:40	00:61:19	04:05:00	37:21:00
21	00:00:28	00:01:52	00:07:29	00:29:58	00:59:50	04:00:00	
22	00:00:27	00:01:50	00:07:18	00:29:13	00:58:22	03:54:00	
23	00:00:27	00:01:47	00:07:08	00:28:34	00:57:05	03:48:00	
24	00:00:26	00:01:45	00:06:58	00:27:52	00:55:41	03:43:00	33:56:00
25	00:00:25	00:01:42	00:06:48	00:27:14	00:54:25	03:38:00	
26	00:00:25	00:01:40	00:06:39	00:26:38	00:53:12	03:33:00	
27	00:00:24	00:01:38	00:06:31	00:26:02	00:52:02	03:28:00	
28	00:00:24	00:01:35	00:06:22	00:25:28	00:50:52	03:24:00	31:00:00
29	00:00:23	00:01:33	00:06:13	00:24:53	00:49:42	03:10:00	
30	00:00:23	00:01:31	00:06:06	00:24:22	00:48:42	03:05:00	

10 inches
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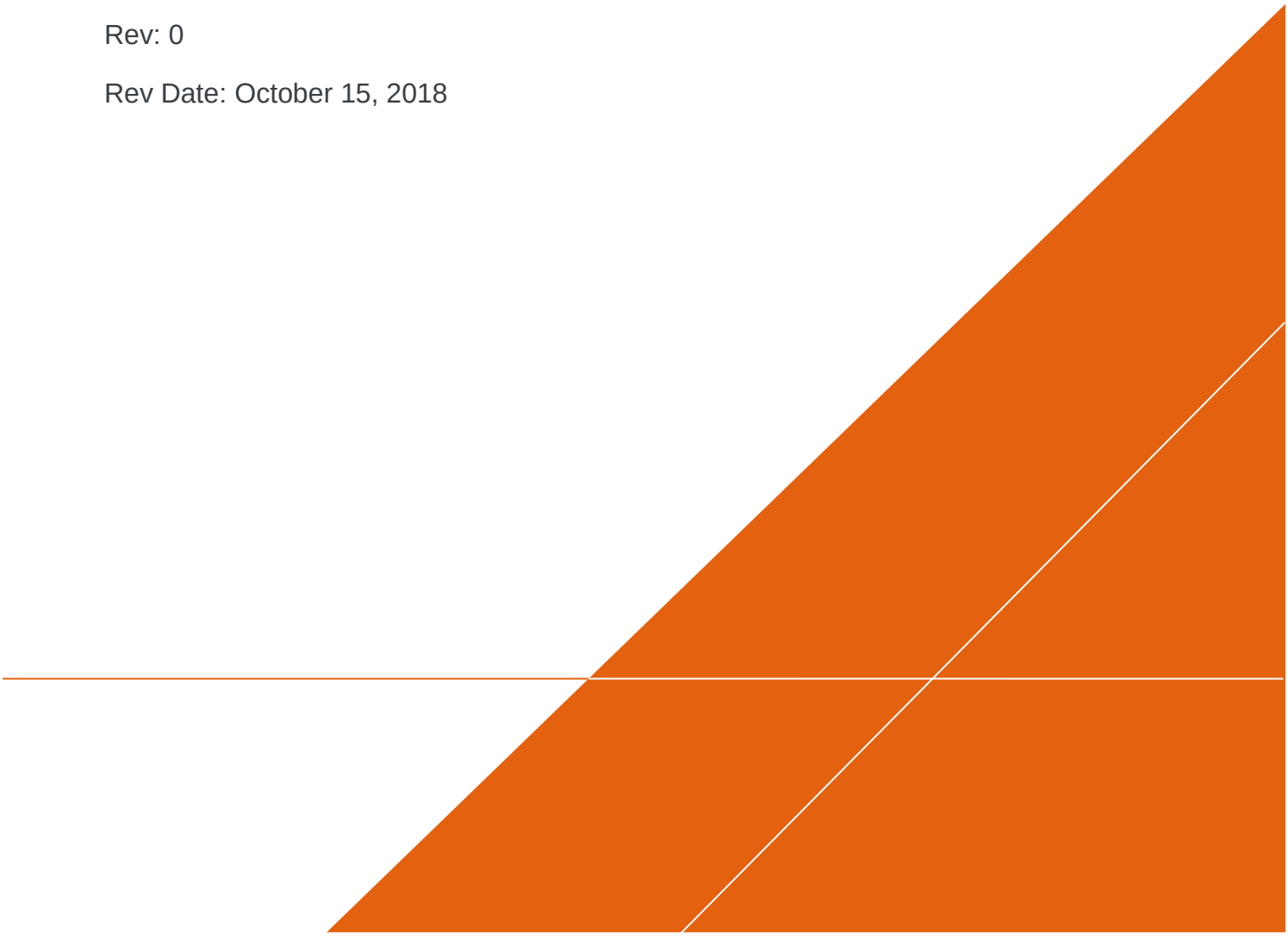
RI-SOP-8

ARCADIS Design & Consultancy for natural and built assets

TGI - BEDROCK CORE COLLECTION AND DESCRIPTION

Rev: 0

Rev Date: October 15, 2018



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 15, 2018	All	Updated and re-written as TGI	Marc Killingstad

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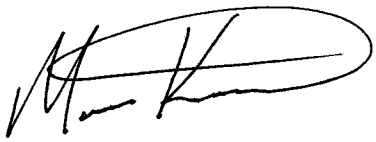
APPROVAL SIGNATURES

Prepared by:		10/15/2018
	Michael Cobb	Date:
Technical Expert Reviewed by:		10/15/2018
	Marc Killingstad (Technical Expert)	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what are described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) describes the procedures to be used to collect and describe bedrock core samples. The approach described here is applicable for subsurface investigations employing a standard wire-line or conventional diamond-bit coring approach, where the project objectives may include:

- Describing bedrock lithology, degree of weathering, fracturing, and other field-observable rock characteristics
- Evaluating relative groundwater yield of fractures or intervals to assist in well design decisions

The methodology described here is in general accordance with *ASTM Method D 2113-99, Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation*. Additional terminology standards are based on the New York Department of Transportation's *Rock Core Evaluation Manual* (NYSDOT, 2015). This approach and level of detail is appropriate for most environmental-site subsurface investigations. Given the diverse nature of bedrock, and variety of potential project objectives, the project team will review site-specific data needs prior to starting work and, if needed, adapt the field procedures.

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The scope of this TGI is specific to core collection and description; it does not encompass the broader suite of tasks associated with bedrock drilling or well construction (see relevant SOP and TGIs, as needed). Note that coring work is often combined with related bedrock characterization techniques, including packer-testing, geophysical logging, FLUTe™ profiling and whole-core rock sampling. These tasks are outside of the scope of this TGI; however, if such additional work is part of the project scope, the planning and sequencing of coring will consider the requirements of those tasks.

3 PERSONNEL QUALIFICATIONS

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed.

In addition, Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements or access control requirements.

Bedrock core logging will only be performed by Arcadis personnel or authorized subcontractors with a bachelor's degree in geology or a geology-related discipline. Field personnel will complete training on this TGI in the office and/or in the field under the guidance of an experienced field geologist with at least 2 years of prior experience with bedrock core description.

Note that this TGI is written specifically for site characterization and remediation projects. When bedrock core samples are to be used for engineering purposes (e.g., foundation design, rock mechanics, design of excavation support), field staff will work under the direction of a geotechnical engineer.

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4 EQUIPMENT LIST

Typically Provided by Geologist	Typically Provided by Driller
• Approved site-specific Health and Safety Plan (HASP) • Approved site-specific FIP/work plan which will include boring location map and drilling plan • Required PPE (see site-specific HASP) • Field logbook and/or rock coring logs • Permanent marking pen for labeling boxes and cores (indelible ink) • 6-foot wooden folding ruler (or similar) graduated in tenths-of-feet (not inches) • Distilled water and spray bottle for wetting and washing core • Camera and/or smart device (phone or tablet) • Pen knife (to test rock hardness) • Munsell rock color chart • Rock hammer • Plastic sheeting (e.g., Weatherall Visqueen) • Stopwatch • Carpenter's protractor • Photoionization detector (PID) or Flame ionization detector (FID) (as appropriate, depending on site-specific constituents of concern) • Air monitoring equipment (as required) • Hand lens (optional) • 10% Hydrochloric acid solution (appropriately labeled eye-dropper for carbonate identification [optional]) • Sturdy saw horses to support core box at working height (optional)	• Core boxes • Wood blocks to separate core runs in core boxes • Rubber hammer (for tapping rock core out of core barrel)

5 CAUTIONS

- **Review relevant guidance**: Utility avoidance, drilling, decontamination, management of investigation derived waste and related tasks will be completed in accordance with a project-specific field implementation plan (FIP)/work plan and/or applicable SOPs or TGIs.
- **Use a trusted, experienced driller:** The quality of bedrock core samples often depends on the skill of the driller (e.g., at selecting the correct tooling, down-pressure and spin-rate for the type of rock and depth). An inexperienced driller will often drill more slowly and cause unnecessary mechanical breaks in the core. It is also important to use a rig equipped with a high-speed coring head. Many rotary or auger rigs are not capable of the speeds required for coring, unless modified for coring.

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- **Choose a clean water supply for drilling fluid:** Water is the preferred drilling fluid when coring. The water used for drilling will be of sufficient quality to meet project objectives. Testing of water supply will be considered. Drilling muds are to be avoided, except in special cases where circulation cannot be maintained.
- **Understand your driller's plans for recirculation of drilling water:** Recirculation is common practice in coring, to limit generation of large quantities of investigation-derived waste (IDW). Water is pumped down the inside of the core barrel to cool the bit and carry rock cuttings back to the surface through the annular space outside the barrel. The return water spills into a mud tub, often designed with several baffles to help cuttings fall out of suspension. This water is then pumped back down the core barrel, or recirculated, until the sediment load is too great, then water must be replaced. Recirculation can increase the risk of cross-contamination, so caution is needed. However, coring without recirculation can quickly generate very large quantities of IDW and is often not practicable.
- **Avoid cross-contamination**: Core drilling often involves creating long open boreholes that may, at least temporarily, penetrate confining beds or create artificial connections between fracture zones at different depths. If cross-contamination is a concern at a site, work will be planned to limit the length of open sections (e.g., by telescoping casing), and limit the duration that a borehole stands open. Field crews will stop-work if dense-non-aqueous phase liquid (DNAPL) is encountered (e.g., if sheens are observed on drilling return water).

6 HEALTH AND SAFETY CONSIDERATIONS

Conduct drilling and related tasks in in accordance with a site-specific Health and Safety Plan (HASP). Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work. Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP.

Note that full core boxes can be heavy and awkward to lift, depending on core-diameter and box size. Be sure to use proper bending and lifting techniques to avoid muscle strain and other potential injuries. Use two people to lift heavy core boxes whenever feasible.

Use appropriate hand protection when conducting carbonate-rock test (using dilute acid) and hardness tests (using a penknife). If site- or client-specific health and safety requirements prohibit use of fixed/folding-blade knives, an alternative steel object (e.g., nail) may be substituted.

7 PLANNING CONSIDERATIONS

Bedrock coring is the primary method available for collecting representative, minimally disturbed field samples from bedrock boreholes. The most common approach involves a cylindrical diamond-impregnated core bit attached to an outer string of drill pipe. The entire pipe is spun at a high velocity, cutting a donut-shaped hole, leaving an intact core of rock that passes through the bit into the core barrel as drilling continues deeper. At the end of a core run (typically 5 or 10-feet long), the core is snapped off by backing the tools slightly.

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What coring method is appropriate for the job? There are two common bedrock coring methods that use different approaches to retrieve cores:

- **Wireline Coring**: Core samples are brought to the surface between runs using a retriever on a wire that connects to the top of the inner core-barrel and lifts it to the surface, leaving the outer barrel in place. This is the most common method and is preferred in most cases.
- **Conventional Coring**: Core is retrieved by removing the entire coring tool string from the borehole. This approach is less common, but it occasionally used for shallow boreholes where only one or two runs are required (e.g., to confirm rock at the base of overburden borehole).

What core size is appropriate? Coring tools exist in several common sizes, generally referred to by a two-letter code. The most-common dual-tube wire-line core-sizes are listed in the table below.

	Common Wireline Core Bit Sizes (inches)		
	NQ	**HQ**	**PQ**
Core Diameter	**1.88**	**2.50**	**3.34**
Hole Diameter	**2.98**	**3.78**	**4.83**

Note: conventional core sizes are denoted NX, HX or similar, and have slightly different sizing.

NQ cores are most often used in shallow geotechnical applications, while HQ core are the most common used for environmental well drilling. HQ's size is suitable for most geophysical logging techniques, and some small-diameter packer assemblies, but subsequent reaming is often (though not always) required to enlarge the borehole before a well can be set. PQ is used less frequently because the larger size adds considerable weight, but a PQ core hole can more often be used to build a monitoring well without additional reaming.

What type of rock is expected? It is critical to have a good idea of what conditions will be encountered before starting.

- Review previously completed logs.
- Check available geologic maps or water-resources reports.
- Consult other knowledgeable geologists who have experience in the area.
- Learn what bedrock units might be present, how they are commonly described, and whether they have useful diagnostic characteristics.
- Learn whether there are any key marker beds, or whether there any key confining beds that should not be penetrated.
- If needed, the field geologist will review lithologic characterization techniques specific to the types of rock expected.

What level of logging detail is required? Core description can be time-consuming; therefore, consider the project data needs and establish priorities for what aspects of the rock will be classified. For most environmental projects, highly detailed logging of lithology and petrology are unnecessary, while fractures

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are of paramount concern. Sometimes identifying a particular contact is critical, but minor lithologic variability is not. Establishing priorities in advance will allow the field geologist to prepare and perform efficiently in the field. *When planning, note that a single geologist often cannot keep up with the requirements of core-collection and description in real-time (e.g., as the hole is being drilled). Additional "catch-up" time is often necessary and will be considered in project planning.*

8 PROCEDURE

Core description is a multi-step process. The general sequence of work can be summarized as follows:

Stage	Activity
Rig set-up	Establish measuring points, measure tooling, establish roles and procedures with driller.
Active coring	Track drilling progress. Track water use. Watch return water for signs of impact. Conduct air monitoring. Setup and label core boxes.
Core extraction	Arrange core in box. Screen core for contaminant impacts. Mark and label core and fractures. Measure recovery. Calculate rock quality designation (RQD).
Core logging	Describe rock lithology, structure, weathering, fracturing, and other characteristics.
End of hole	Photograph core boxes. Store or dispose of core.

8.1 Before Coring Starts

Prepare for accurately tracking depths

1. Discuss with driller what reference point will be used for ground surface (e.g., the base of the mud tub) and mark it, if needed. All depths will be recorded relative to this datum.
2. Measure and mark (if needed), a fixed reference point above the ground surface that will serve as the starting/stopping point for core runs (e.g., the top of an outer casing or a vice)
3. Measure out core tooling lengths, including barrels, bits and subs, so that the depth of the barrel will be known to the nearest tenth of a foot.

Prepare for tracking water usage

1. Confirm whether the driller will be recirculating drilling water or using a continuous clean source. [NOTE: *Recirculation is a common practice in coring; however, it is not permissible for all projects and jurisdictions*].
2. Discuss with driller how water usage will be tracked. Water lost in each run will be estimated, either via a change in level of a mud tub and/or drop in level of a separate water tank.
3. Measure dimensions of mud tub and/or water tank to estimate volume. Mark graduations, if needed, so that volume changes can be estimated.

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8.2 Active Coring

1. Request that the driller tag borehole with a weighted line before start of coring and periodically between runs to confirm depth.
2. Use a stopwatch (or equivalent) to time the length of each run (e.g., 40 min for 10 feet)
3. Note starting and ending water volume in tanks and/or mud tub.
4. Note the starting and ending core-run depth by noting the position of joints in the drilling string relative to the fixed reference point.
5. To the extent feasible, the driller should maintain a consistent bit pressure, water pressure, and rotational rate throughout a run, and avoid stopping or backing the tools, until the run is complete.
6. Request that driller alert you to changes in drilling condition during a run, including:
    a. Significant change in drilling rate that may indicate a change in lithology or weathering.
    b. A change in water recirculation rate that may indicate a major fracture
    c. Bit drop, which may indicate a void or highly-weathered zone.
    d. Any odors or sheens that may occur from the return water. [NOTE: *Under most drilling programs, the appearance of sheens or NAPL in the drilling water is cause to exercise stop-work authority. Drilling through a zone known to contain NAPL must be done only with CPM approval*.]
    e. Change in sediment load in return water, which may indicate a highly-weathered zone.
7. If air monitoring is required at the borehole (based on HASP and nature of contaminants present), periodically screen the driller breathing zone and return water splash zone.

8.3 Core Extraction

8.3.1 Core Handling and Labelling

In most instances, core samples will be placed directly into core-boxes by the driller. Core samples will be placed with increasing depths aligned left to right and top to bottom. If core is covered in sediment or mud, it is helpful to rinse the core with clean water before placing it in the box.

The drillers will take care not to unnecessarily break the core as they are extracting from the barrel; however, they will have to break long sections of core that overlap the box-edges, typically using a hammer.

Core and core boxes will be labeled using a heavy indelible marker (e.g., Sharpie) as follows:

What to Label	How to Label
Start and end of core runs	Label box edges or insert wooden blocks to separate runs. Label run number at top of run; e.g., “Run 2”, at the start of Run 2.

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What to Label	How to Label
Vertical alignment of core	Draw short arrows on each major section of core pointing toward ground surface. (An alternate method using two parallel colored stripes is common but is challenging on wet core.)
Mechanical breaks (also called driller breaks)	Two parallel lines crossing the break at a right angle and labeled with the letter "M" (see Section 8.3.2)
Fractures (natural breaks)	A single line crossing the fracture at a right angle
Intervals with no core recovery	Insert a wooden spacer marked "No recovery" and with corresponding depth for any interval where no rock was recovered (e.g., a weathered zone that washed out, or karst void)
Sections of core removed from box	Insert a wooden spacer marked "Removed" with corresponding depth.
Core box lid (outside)	Site or project name, well or borehole ID, date drilled, box number (e.g., "1 of 5") and start and end depth of core contained in the box. (Additional information such as site address and project number can be included, if needed.)
Core box lid (inside)	Label the same as the box exterior. If core is expected to be archived, it is common practice to also include the depths, recovery and rock quality designation (RQD) for each run contained in the box.
Core box left end	Site or project name, well or borehole ID, box number, and start and end depth of core contained in the box.

8.3.2 Assessing Natural or Mechanical Breaks

When evaluating a core, it is necessary to determine whether the observed fractures are natural or mechanical. The primary indicators to look for include:

Signs of a Natural Fracture	• Weathering on face • Oxidation of minerals adjacent to face • Clay on face (if distinct from sediment in drilling fluid) • Linear striations on face
Signs of Mechanical Break	• Absence of weathering or oxidation • Crisp edges

Other considerations when evaluating the nature of break include:

- Weak, friable rock (such as shale), will often have numerous mechanical breaks that that are indistinguishable from real ones. Judgement is required, but for the sake of RQDs, such fractures are generally considered natural.

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- If the core spins on itself inside the barrel A rounded or “ground” fracture faces can sometimes occur. This can happen to either a natural or mechanical break, so other evidence is required.

Assessing the nature of fractures can be challenging and subjective. In general, where the case is uncertain, assume the fracture is natural.

8.3.3 Core Run Description

Several descriptors are made on the basis of the core-run, typically 5 or 10 feet in nominal length. Lengths and depths are best recorded to the nearest 0.1 foot.

What to Record	How to Determine
Start and end depth	Determine by tracking advancement of the core tooling, referenced to ground surface. Note that cores occasionally snap off above the drilled depth on retrieval, leaving a cored “stub” in the hole, which is typically retrieved in the next run. When this happens, the run depths and retrieved core depths will differ. Both values will be recorded. Periodic soundings are helpful to verify depth.
Recovery length and percentage	Measure the total length of recovered core. The recovery percentage is based on start and end depths of the retrieved core (i.e., do not count a stub of core left in the hole).
Rock Quality Designation (RQD)	Add up the length of unfractured core-pieces greater than 4-inches in length (where fractures are dipping, measure between the points where fractures intersect the center-line of the core). Divide by the total length of the core run (bottom minus top depth of core recovered) and record the result as a percentage. Note that most common practice is to exclude obvious mechanical breaks when assessing RQD sections. However, where it is unclear whether a break is natural or mechanic, assume it is natural for the RQD assessment.
Water Loss	Determine based on changed level in mud tub, water tank, or other method (determined in consultation with the driller before coring starts). If a sudden change in water loss is observed during the run, record the approximate depth where it occurred.
Run-Time (optional)	Stopwatch recording of the total time to core a run. This can be useful in showing transition between rock types, or that the core bit has dulled. In some cases, foot-by-foot times can be recorded by chalk-marking increments on the barrel.

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8.3.4 Contaminant Screening of Core

Methods for screening for contamination while coring depend on the nature of impacts suspected. As noted above, air-monitoring at the ground surface of the borehole, and continuous visual observation of the return water while drilling generally provide the first indication of an impact.

Specific procedures for screening cores will be identified in the project FIP/work plan. Common approaches include the following:

- If NAPL may be present, fracture surfaces will be visually inspected for sheens or NAPL.
- Though field staff will NOT intentionally sniff the core, obvious odors are sometimes useful indicators. Field descriptions of odors will be general, and not attempt to specify what contaminant it smells like.
- If screening core for volatile organic compounds (VOCs) with a photo-ionization detector (PID), focus attention on fractures. With the core lying in the core-box, separate the fracture slightly, cover the opening with a gloved hand, and then insert the PID tip into the fracture aperture.
- If NAPL is suspected (e.g., based on high PID hits, or sheens in the return water), but not visible on the core, one of several commercially available NAPL-detection kits (using hydrophobic dye) may be applied to the core as a supplemental test.

As noted above, when NAPL is observed in a borehole, drilling will almost always stop to avoid dragging the impacts down—drilling deeper will occur ONLY when necessitated by the project objectives, and ONLY after consulting project leadership.

8.4 Procedures for Core Logging

Logging core includes two parts: (1) describing the nature of the rock (e.g., lithology, structure, bedding) and (2) logging observed discontinues (e.g., fractures or weathered zones).

8.4.1 Logging Rock Characteristics

The field geologist will log the following characteristics of the rock core:

What to Record	How to Describe
Depth	Note top of an interval being described, relative to ground surface. Avoid referencing depths relative to the position in the core run.
Rock type	Describe based on observation. Use terminology consistent with local mapping, if available. If the specific type cannot be determined in the field, use a more general descriptor (e.g., metamorphic).
Grain size	See chart in **Attachment 1**. For crystalline rocks, note applicable texture.
Color	Reference Munsell rock color chart. Describe matrix color and major clast color separately, if applicable.

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What to Record	How to Describe
Weathering state	See terminology chart (**Attachment 1**).
Hardness	See terminology chart (**Attachment 1**).
Degree of Fracturing	See terminology chart (**Attachment 1**). Also note general characteristics of fractures (e.g., if oxidized, filled, rough or smooth, dominantly aligned with bedding/foliation, etc.). Call out depths of major fractures. (See also, Section 8.4.2)

Other observations may also be made, if appropriate to the rock type. Common supplemental observations include:

- Diagnostic minerals present
- Presence and abundance of fossils
- Particle angularity/shape, e.g., for conglomerates or breccias
- Effervesce, e.g., if testing for limestone or dolomite using a hydrochloric acid solution
- Presence of healed fractures.
- Observations of porosity, pitting, vugs, or cavities (see terminology chart in **Attachment 1**)

8.4.2 Logging Discontinuities

In addition to characterizing the rock mass (as described above), core descriptions will often identify the depth and characteristics of specific fractures and other discontinuities. In general, the following will be noted:

- Fractures (excluding mechanical breaks), including descriptors for orientation, filling, oxidation or mineralization
- Zones of notable porosity (e.g., pitting, vugs)
- Zones of intense weathering (e.g., greater than surrounding rock mass)

Discontinuities are logged either in list-form, or on a scaled-graphical log, using standard abbreviations to identify important characteristics (see **Attachment 1**).

Note that in moderately or intensely fractured rock, logging every observed fracture may not be practical, or especially useful. Generalizations such as “intensely fractured zone” are often appropriate.

8.5 Procedures to Complete after Completion of Core Hole

8.5.1 Photographing Core

All core boxes will be photographed in a systematic manner. Best practice includes the following steps:

- Place the core box in a well-lit space

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- Lay a tape measure or marking stick for scale along the length of the core box
- Wet the core with a spray bottle
- Take a high-resolution photograph showing the entire core box and labeled lid from directly above or at slight angle.
- If close-ups of particular features are needed, include a visible scale and labeled notecard in the photo.
- If a color is a key diagnostic aspect, a standard color reference chart may be included in the photos.

It is generally easiest to obtain consistent, high quality photographs by taking the photos in batches (e.g., after a borehole is completed), rather than attempting to take photographs immediately after each core run.

8.5.2 Core Storage or Disposal

If core will be stored for potential future use, boxes will be moved to a central location. In general:

- Core boxes will be stored under cover, ideally indoors and somewhere where they will not need to be moved often
- Boxes will be places on pallets (or similar) to keep off the ground
- Boxes will be stacked so that the labeled ends are visible and facing the same direction
- Boxes will be stacked no more than about 3 feet high (to avoid lifting above waist level)

If core is to be discarded, do so only after reviewing notes and confirming that all important details have been recorded. Core will be disposed-of consistent with project IDW requirements. Core boxes will not be removed from a site without appropriate planning and approval.

9 WASTE MANAGEMENT

Coring may generate several types of IDW in addition to the cores themselves:

- Coring typically generates substantial quantities of drilling fluid. It is typically a mixture of water and suspended fine sediment. In most cases, this is drummed. For large jobs, roll-off or “sludge” boxes may be more economical.
- Solid rock cuttings also accumulate in the mud tub. These are typically shoveled into drums.
- Other waste streams include decontamination liquids, and disposable materials (well material packages, personal protective equipment [PPE], etc.).

Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that all IDW will be placed in clearly labeled, appropriate containers and documented in the field log book.

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10 DATA RECORDING AND MANAGEMENT

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements as outlined in the FIP/work plan and/or QAPP.

Core descriptions and related activities will be recorded in a field book and/or on appropriate field forms. In addition to the core description information detailed above, field notes will record personnel present on site, including driller names, drilling equipment used, significant weather conditions, and the timing of all activities.

Field forms, logs/notes (including daily field and relevant calibration logs), and digital records will be maintained by the field team lead.

Records will be transmitted to the Arcadis Project Manager and/or Task Manager, as appropriate, at the end of each day or as specified in the FIP/work plan.

Electronic data files will be sent to the project team and uploaded to the electronic project folder daily or as specified in the FIP/work plan.

Management of the original documents from the field will be completed in accordance with the site-specific QAPP.

11 REFERENCES

ASTM, 1999. Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation, D 2113-99.

New York State Department of Transportation (NYSDOT), 2015. Geotechnical Engineering Manual, Rock Core Evaluation Manual, GEM-23, Rev. 2.

12 ATTACHMENTS

Attachment 1. Bedrock Core Description Terminology

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Attachment 1
Bedrock Core Description Terminology

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Bedding Thickness*

Term	Average Thickness
Massive	No visible bedding
Very Thick-Bedded	Greater than 4 ft. (> 1.2 m)
Thick-Bedded	1 ft. to 4 ft. (0.3 m to 1.2 m)
Medium-Bedded	4 in. to 12 in. (100 mm to 300 mm)
Thin-Bedded	1.2 in. to 4 in. (30 mm to 100 mm)
Very Thin-Bedded	0.5 in. to 1.2 in. (13 mm to 30 mm)
Laminated	< 0.1 in.

*For igneous and metamorphic rocks, planar features such as foliation and banding are described using same thickness designations (e.g., thin-banded).

Fracturing

Term	Length of Most Recovered Core
Unfractured	No observed fractures.
Very slightly fractured	> 3 ft. (1 m).
Slightly fractured	1 to 3 ft. (0.3 to 1 m)
Moderately fractured	0.33 to 1 ft. (0.1 to 0.3 m)
Intensely fractured	0.1 to 0.33 ft. (0.03 to 0.1 m)
Very intensely fractured	Core mostly broken; few intact core segments

Pores or Voids

Term	Pore Size
Porous	Smaller than pinhead. Presence indicated by degree of absorbency
Pitted	Pinhead to ¼ inch across.
Vug	¼ inch across to diameter of core
Cavity	Larger than core diameter

Weathering State

Term	Characteristics
Fresh	No visible signs of decomposition or discoloration
Slightly weathered	Slight discoloration inward from open fractures; otherwise fresh
Moderately weathered	Discoloration throughout. Weaker minerals such as feldspar decomposed. Strength somewhat less than fresh rock, but cores cannot be broken by hand or scraped by knife. Texture preserved.
Highly Weathered	Most minerals somewhat decomposed. Specimens can be broken by hand with effort, or shave with knife. Texture become indistinct, but fabric preserved.
Extremely weathered (saprolite)	Minerals decomposed to soil, but fabric and structure preserved
Decomposed (residual soil)	Rock fully decomposed to plastic soils. Rock fabric and structure completely destroyed.

Hardness

Term	Field Test
Soft	Can be scratched with fingernail.
Medium Hard	Easily scratched by penknife
Hard	Difficult to scratch by penknife.
Very hard	Cannot be scratched by penknife.

Grain-Size

Term	Size
Microcrystalline / Aphanitic*	No visible grains
Fine grained	0.06 - 0.25 mm
Medium grained	0.25 - 0.5 mm
Coarse grained	0.5 - 2.0 mm

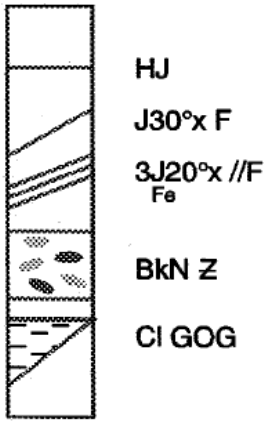
*Aphanitic applies to detrital rocks

Common Abbreviations

Abbr.	Definition	Abbr.	Definition	Abbr.	Definition
BkN	broken	JxF	joint crosses foliation	si	silt
CAL	calcareous or calcite	l	laminae	SZ	sheer zone
cl	clay	//	parallel	U	unfoliated or unstratified
F	foliation	m	mud in opening	v	vuggy
Fe	iron staining	MB	mechanical break	VJ	vertical joint
GOG	gouge	QTZ	quartz	w	weathered
HJ	horizontal joint	s	solution enlargement	WZ	weathered zone
J	joint *	S	stratification	x	crossing
J//F	joint is parallel to foliation	sa	sand	Z	Zone

* The term “joint” may indicates any natural fracture, including bedding plane fractures.

Example Graphic Log



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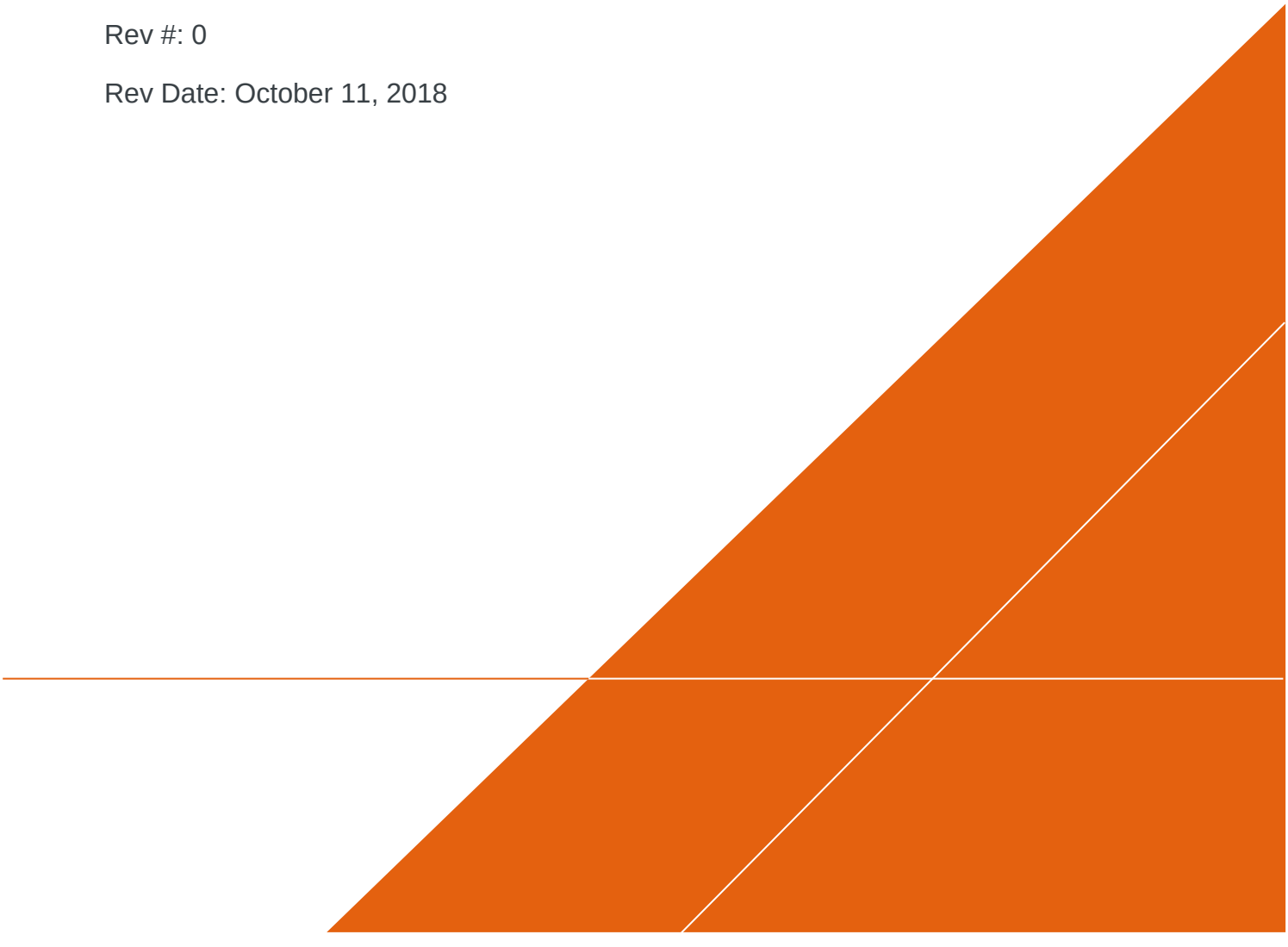
TGI – SOIL DRILLING AND SAMPLE COLLECTION

Rev #: 0

Rev Date: October 11, 2018



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 11, 2018	All	Updated and re-written as a TGI	Marc Killingstad

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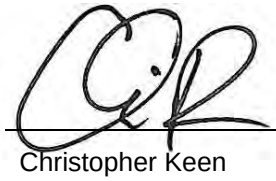
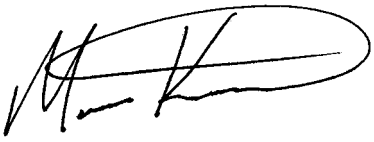
APPROVAL SIGNATURES

Prepared by:		10/11/2018
	Christopher Keen	Date:
Technical Expert Reviewed by:		10/11/2018
	Marc Killingstad (Technical Expert)	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) describes general drilling procedures and the methods to be used to field screen and collect soil samples for laboratory analysis in unconsolidated sediments. For soil description procedures, please refer to the *TGI - Soil Description*. For monitoring well installation in granular aquifers, please refer to the *TGI - Monitoring Well Installation*.

Overburden (unconsolidated sediments) drilling is commonly performed using the hollow-stem auger drilling method. Other drilling methods suitable for overburden drilling, which are sometimes necessary due to site-specific geologic conditions, include: drive-and-wash, spun casing, rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and hand tools may also be used. Drilling within consolidated materials such as fractured bedrock is commonly performed using water-rotary (coring or tri-cone roller bit), air rotary or rotasonic methods. For guidance when drilling in consolidated materials (i.e., bedrock), please refer to the *TGI – Bedrock Core Collection and Description*.

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The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, site or regional geologic knowledge, types of sampling to be conducted, required sample quality and volume, and cost.

Field screening of soil samples is commonly performed using a photoionization detector (PID) and/or a flame ionization detector (FID). These instruments are used to measure relative concentrations of volatile organic compounds (VOCs) for the selection of samples for further laboratory or field analysis. Field screening for dense non-aqueous phase liquids (DNAPL) may be performed using hydrophobic dye (Oil Red O or Sudan IV), which is pertinent at chlorinated solvent sites.

Collection of soil samples for laboratory analysis may be performed using a variety of techniques including grab samples and composite or homogenized samples. Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample. Samples for VOC analysis will not be homogenized or composited and are collected as discrete grab samples.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools).

3 PERSONNEL QUALIFICATIONS

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, Arcadis field personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP), Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Personnel responsible for overseeing drilling operations will have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

Arcadis personnel directing, supervising, or leading soil sampling activities will have a minimum of 1 year of previous environmental soil sampling experience. Field employees with less than 6 months of experience will be accompanied by a supervisor (as described above) to ensure that proper sample collection techniques are employed.

Additionally, the Arcadis field team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

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4 EQUIPMENT LIST

The following materials will be available, as required, during soil boring drilling, field screening, and sampling activities:

- Site-specific HASP and health and safety documents identified in the HASP
- Field Implementation Plan (FIP)/work plan that includes site map with proposed boring locations, field sampling plan (with corresponding depths, sample analyses, sample volume required, and sample holding time), and previous boring logs (as available)
- Appropriate personal protective equipment (PPE), as specified in the HASP
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified in the Traffic Safety Plan (TSP)
- Photoionization detector (PID), flame ionization detector (FID) or other air monitoring equipment, as needed, in accordance with the HASP
- Drilling equipment required by *ASTM D1586*, when performing split-spoon sampling
- Disposable plastic liners, when drilling with direct-push equipment
- Appropriate soil sampling equipment (e.g., stainless steel spatulas/spoons/bowls, knife)
- Stainless steel hand auger and stainless-steel spade if using manual methods
- Indelible ink pens
- Engineer's ruler or survey rod
- Sealable plastic bags (e.g., Ziploc®)
- Air-tight sample containers and 8-oz. glass Mason jars or driller's jars
- Aluminum foil
- Plastic sheeting (e.g., Weatherall Visqueen)
- Decontamination equipment (buckets, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels)
- Appropriate sample blanks (trip blank supplied by the laboratory), as specified in the FSP
- Soil sample containers and labels (supplied by the laboratory) appropriate for the analytical method(s) with preservative, as needed (parameter-specific)
- Appropriate transport containers (coolers) with ice and appropriate labeling, packing, and shipping materials;
- Appropriate soil boring log (**Attachment 1**)
- Chain-of-custody forms
- Field notebook.

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- Digital camera (or smart phone with camera)
- Drums or other containers appropriate for soil and decontamination water, as specified by the site investigation-derived waste (IDW) management plan, and appropriate drum labels

5 CAUTIONS

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service. See appropriate guidance for proper utility clearance protocol. Work will be performed in accordance with the Arcadis *Utility Location and Clearance Health and Safety Standard* and the *Utilities and Structures Checklist* will be completed before beginning any intrusive work.

Prior to beginning field work, the project technical team will ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members. An internal call with the project technical team to review the FIP/work plan scope and objectives is strongly recommended prior to mobilization to ensure that the field work will be effectively and efficiently executed.

Some regulatory agencies have specific requirements regarding borehole abandonment and grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the drilling plan.

If DNAPL is known or expected to exist at the site, refer to the project specific documents (e.g., DNAPL Contingency Plan) for additional details regarding drilling to reduce the potential for inadvertent DNAPL remobilization.

Similarly, if light non-aqueous phase liquid (LNAPL) is known or expected to be present as “perched” layers above the water table, refer to the DNAPL Contingency Plan. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Water used for drilling, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply will be considered.

Specifications of materials used for backfilling the borehole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPL is likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.

Store and/or stage empty and full sample containers and coolers out of direct sunlight. Be careful not to over-tighten lids with Teflon® liners or septa. Over-tightening can impair the integrity of the seal and can cause the glass to shatter and create a risk for hand injuries.

NOTE: *Field logs and some forms are considered to be legal documents. All field logs and forms will therefore be filled out in indelible ink. Do not use permanent marker or felt-tipped pens for labels on*

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sample container or sample coolers. Permanent markers could introduce volatile constituents into the samples.

NOTE: *An Arcadis employee that is appropriately trained at the correct level of internal hazardous materials/DOT (Department of Transportation) shipping must complete an Arcadis shipping determination to address applicable DOT and IATA (International Air Transport Association) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.*

6 HEALTH AND SAFETY CONSIDERATIONS

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Working outside at sites with suspected contamination may expose field personnel to hazardous materials such as contaminated groundwater or non-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include biological hazards (e.g., stinging insects, ticks in long grass/weeds, etc.), and potentially the use of sharp cutting tools (scissors, knife). Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives and use appropriate hand protection.

If thunder or lighting is present, discontinue drilling and sampling until 30 minutes have passed after the last occurrence of thunder or lighting.

7 PROCEDURE

The procedures for drilling and the methods to be used to field screen and collect soil samples for laboratory analysis are presented below:

DRILLING PROCEDURES

Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area.
2. Advance boring to designated depth:
    a. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent)
    b. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent)

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c. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)

d. A common sampling method that produces high-quality soil samples with relatively little soil disturbance is described in *ASTM D1586 – Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils* (ASTM D1586).

    i. Split-spoon samples are obtained during drilling using hollow-stem auger, drive-and-wash, spun casing, and fluid/mud rotary

e. Rotasonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks

f. Dual-rotary removes cuttings by compressed air or water/mud and allow only a general assessment of geology

3. Describe each soil sample as outlined in the appropriate project records (refer to the description procedures outlined in the *TGI - Soil Description*)

    a. Record descriptions on the soil boring log (**Attachment 1**) and/or field notebook.

    b. When possible photo document the samples (e.g., soil cores, split-spoons)

    c. During soil boring advancement, document all drilling events in field notebook, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-barrel sampler in 6-inch increments) and work stoppages

    d. Blow counts will not be available if rotasonic, dual-rotary, or direct-push methods are used; however, if standard penetration testing is required during rotasonic drilling, an automatic drop hammer may be used in conjunction with the method to switch from core barrel advancement to standard penetration testing

4. The drilling contractor will be responsible for obtaining accurate and representative samples, informing the supervising Arcadis geologist of changes in drilling pressure, and keeping a separate general log of soils encountered, including blow counts

    a. The term “samples” means soil materials from particular depth intervals, whether or not portions of these materials are submitted for laboratory analyses

    b. Records will also be kept of occurrences of premature refusal due to boulders or construction materials that may have been used as fill

    c. Where a boring cannot be advanced to the desired depth, the boring will be abandoned, and an additional boring will be advanced at an adjacent location to obtain the required sample

    d. Where it is desirable to avoid leaving vertical connections between depth intervals (e.g., if DNAPL or perched LNAPL are known or expected to exist at the site), the borehole will be sealed using cement and/or bentonite (see **Section 5** above)

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e. Multiple refusals may lead to a decision by the supervising geologist to abandon that sampling location

Direct-Push Method

The direct-push drilling method may also be used to complete soil borings. Examples of this technique include Geoprobe®, Diedrich Environmental Soil Probe (ESP) System, or AMS PowerProbe. Environmental probe systems typically use a hydraulically operated percussion hammer.

Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual tube liner for sampling soil (dual tube sampling system).

The outside diameter (OD) of the outer casing ranges from 2.25 to 6 inches and the OD of the inner sampling tube diameter ranges from 1.4 to 4.5 inches. The outer casing isolates overlying soil and permits the unit to continue to probe at depth. The dual tube sampling system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, a single rod system may be used that does not provide a cased boring and which does not allow for tremie-grouting from the bottom up.

The known or expected site conditions (e.g., presence of NAPL) will be evaluated when selecting the type of direct-push sampling system to be employed.

Direct-push drilling can generally achieve target depths 100 feet or less and the achievable depth is based on the site geology.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
2. Advance soil boring to designated depth.
    a. Collect soil samples at appropriate interval as specified in in the FIP/work plan (or equivalent)
    b. Collect, document, and store samples for laboratory analysis as specified in in the FIP/work plan (or equivalent)
    c. Decontaminate equipment between samples in accordance with in the FIP/work plan (or equivalent)
    d. Samples will be collected using dedicated, disposable, plastic liners
3. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

Manual Methods

Manual methods may also be used to complete shallow soil borings. Examples of this technique include using a spade, spoon, scoop, hand auger, or slide hammer. Manual methods are typically used to collect surface soil samples (0 to 6 inches) or to complete soil borings/collect soil samples from a depth of 5 feet or less.

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1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris
3. Use a spade, spoon, scoop, hand auger, or slide hammer to collect a sample of the required depth interval
4. Use an engineer's ruler or survey rod to verify that the sample is collected to the correct depth and record the top and bottom depths from the ground surface
5. To collect samples below the surface interval, remove the surface interval first; then collect the deeper interval
    a. To prevent the hole from collapsing, it may be necessary to remove a wider section from the surface or use cut polyvinyl chloride (PVC) pipe to maintain the opening
    b. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent)
    c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent)
    d. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
6. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

FIELD SCREENING PROCEDURES

PID and FID Screening

Soils are typically field screened with a PID or FID for a relative measure of the total VOCs at sites where VOCs are known or suspected to exist. The PID employs a UV lamp to measure VOCs and the ionization energy (IE) of the site constituents need to be considered when selecting the type of lamp (e.g., 10.6 eV, 11.7 eV) that will be used. In general, any compound with an IE lower than that of the lamp photons can be measured. The FID has a wide linear range and responds to almost all VOCs. Field screening is performed using one (or both) of the following two methods:

1. Upon opening the sampler, the soil is split open and the PID or FID probe is placed in the opening and covered with a gloved hand. Such readings will be obtained at several locations along the length of the sample.
2. A portion of the collected soil is placed in a jar, which is covered with aluminum foil, sealed, and allowed to warm to room temperature. After warming, the cover is removed, the foil is pierced with the PID or FID probe, and a reading is obtained.

Initial PID readings will be recorded on the soil boring log (**Attachment 1**) and/or in the field notebook. The soil sample will be separated from the slough material (if any) by using disposable gloves and a pre-cleaned stainless-steel spoon.

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For the second method, a representative portion of the sample will be placed in a pre-cleaned air-tight 8-ounce container (as quickly as possible to avoid loss of VOCs), filling the container half full to allow for the accumulation of vapors above the soil. An aluminum foil seal will be placed between the glass and metal cap and the cap will be screwed on tightly. Unless the screening will be performed immediately after the sample is placed in the container, the sample containers will be stored in a cooler chilled to approximately 4ºC until screening can be performed.

The headspace of the 8-ounce container will be measured using a PID or FID as follows:

1. Samples will be taken to a warm work space and allowed to equilibrate to room temperature for at least one hour.
2. Prior to measuring the soil vapor headspace concentration, the 8-ounce container will be shaken.
3. The headspace of the sample will then be measured directly from the 8-ounce container by piercing the aluminum foil seal with the probe of the PID or FID and measuring the relative concentration of VOCs in the headspace of the soil sample. The initial (peak) reading must be recorded.

The PID or FID must be calibrated according to the manufacturer's specifications at a minimum frequency of once per day prior to collecting PID or FID readings. The PID will be calibrated to a benzene-related compound (isobutylene) while the FID will be calibrated to methane.

The time, date, and calibration procedure must be clearly documented in the field notebook and/or the calibration log book.

If at any time the PID or FID results appear erratic or inconsistent with field observations, then the instrument will be recalibrated.

If calibration is difficult to achieve, then the PID's lamp will be checked for dirt or moisture and cleaned, or technical assistance will be required. Maintenance and calibration records will be kept as part of the field quality assurance program.

NAPL Screening

To screen for the potential presence of non-aqueous phase liquid (NAPL) in soil, drilling procedures must allow for high-quality porous media samples to be taken. Split-spoon samplers or direct-push samplers will be collected continuously ahead of the auger, drill casing/rods, or probe rods.

Upon opening each split-spoon sampler or direct-push plastic liner sleeve, the soil will immediately be evaluated for the presence of visible NAPL. If NAPL is immediately visible in the sample, its depth will be noted.

Additionally, the soil will be screened for the presence of organic vapors using a PID or FID. During screening, the soil will be split open using a clean spatula or knife and the PID or FID probe will be placed in the opening and covered with a gloved hand (**Method 1** above). Such readings will be obtained along the entire length of the sample. Alternatively, **Method 2** for PID/FID screening (outlined above) may also be performed. If the PID or FID examination reveals the presence of organic vapors above 100 parts per million (ppm), the sample will undergo further detailed evaluation for visible NAPL.

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The assessment for NAPL will include the following tests/observations:

- Evaluation for Visible NAPL Sheen or Free-Phase NAPL in Soil Sampler
    - NAPL sheen will be a colorful iridescent appearance on the soil sample
    - NAPL may also appear as droplets or continuous accumulations of liquid with a color typically ranging from yellow to brown to black, depending on the type of NAPL
    - Creosote DNAPL (associated with wood-treating sites) and coal tar DNAPL (associated with manufactured gas plant [MGP] sites) are typically black and have a characteristic, pungent odor
    - Pure chlorinated solvents may be colorless in the absence of hydrophobic dye. Solvents mixed with oils may appear brown
    - Particular care will be taken to fully describe any sheens observed, staining, discoloration, droplets (blebs), or NAPL saturation
- Soil-Water Pan Test
    - A portion of the selected soil interval with the highest PID or FID reading above 100 ppm will be placed in a disposable polyethylene dish along with a small volume of potable or distilled water
    - The dish will be gently tilted back and forth to mix the soil and water, and the surface of the water will be viewed in natural light to observe the development of a sheen, if any
    - A small quantity of Oil Red O or Sudan IV hydrophobic dye powder will be added, and the soil and dye will be manually mixed for approximately 30 to 60 seconds and smeared in the dish to create a paste-like consistency
    - A positive test result will be indicated by a sheen on the surface of the water and/or a bright red color imparted to the soil following mixing with dye
- Soil-Water Shake Test
    - A small quantity of soil (up to 15 cc) will be placed in a clear, colorless, jar containing an equal volume of potable or distilled water (40-mL vials are well suited to this purpose, but not required)
    - After the soil settles into the water, the surface of the water will be evaluated for a visible sheen under natural light
    - The jar will be closed and gently shaken for approximately 10 to 20 seconds
    - Again, the surface of the water will be evaluated for a visible sheen or a temporary layer of foam
    - A small quantity (approximately 0.5 to 1 cc) of Oil Red O or Sudan IV powder will be placed in the jar
    - The sheen layer, if present, will be evaluated for a reaction to the dye (change to bright red color)
    - The jar will be closed and gently shaken for approximately 10 to 20 seconds
    - The contents in the closed jar will be examined under natural light for visible bright red dyed liquid inside the jar

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- A positive test result will be indicated by the presence of a visible sheen or foam on the surface of water, a reaction between the dye and the sheen layer upon first addition of the dye powder, a bright red coating on the inside of the vial (particularly above the water line), or red-dyed droplets within the soil

NOTE: *If NAPL is obviously present upon opening the soil sampler or evaluating the soil sample within the split-spoon sampler or direct-push liner sleeve, it is not necessary to perform a soil-water pan test or soil-water shake test. In addition, it is not necessary to perform both a soil-water pan test and a soil-water shake test; either test method is acceptable. The pan test may be preferred in some circumstances because the presence of a sheen may be easier to see on a wider surface.*

NOTE: *When using hydrophobic dye in the tests above, color will be assessed outdoors under natural light during the period between sunrise and sunset, regardless of the degree of cloud cover. The hydrophobic dye Safety Data Sheets (SDS) will be incorporated into the HASP and reviewed prior to use and the dyes will be carefully handled and disposed in accordance with regulations*.

SOIL SAMPLE COLLECTION FOR LABORATORY PROCEDURES

If not specifically identified in the FIP, soil samples will be selected for laboratory analysis based on:

1. Their position in relation to identified source areas
2. The visual presence of source residues (e.g., NAPL)
3. The relative levels of total VOCs based on field screening measurements
4. The judgment of the field coordinator

Samples designated for laboratory analysis will be placed in the appropriate containers.

Sample containers for VOC analysis will be filled first immediately following soil core retrieval to reduce loss of VOCs.

If samples will be collected for other analytical parameters, a sufficient amount of the remaining soil will then be homogenized as described below and sample containers will be filled for other parameters.

VOC samples will be collected as discrete samples using a small diameter core sampler (e.g., En Core® Sampler, Terra Core™ Sampler).

The En Core® Sampler is a disposable volumetric sampling device that collects, stores and delivers soil samples without in-field chemical preservation. The En Core® Sampler requires the use of a reusable T-handle.

The Terra Core™ Sampler is a one-time use transfer tool, designed to collect soil samples and transfer them to the appropriate containers for in-field chemical preservation (e.g., methanol).

The small diameter core sampler will be used according to the manufacturer's instructions (e.g., En Novative Technologies). Some regulatory agencies have specific requirements regarding VOC sample collection. Determine whether the oversight agency has specific requirements prior to commencing sampling and collect samples at appropriate interval as specified in the FIP/work plan (or equivalent). Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample.

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NOTE: *Samples for VOC analysis will NOT be homogenized or composited and will be collected as discrete samples as described above*.

The procedure for mixing samples is provided below.

1. Mix the materials in a stainless steel (or appropriate non-reactive material) bowl using a stainless-steel spoon (or disposable equivalents)
    a. When dealing with large sample quantities, use disposable plastic sheeting and a shovel or trowel
    b. NOTE: *When preparing samples for metals analyses, do not use disposable aluminum (or metal tools or trays other than stainless steel), as it may influence the analytical results*
2. Flatten the pile by pressing the top without further mixing
3. Divide the circular pile by into equal quarters by dividing out two diameters at right angles
4. Mix each quarter individually using appropriate non-reactive bowls, spoons and/or sheeting
5. Mix two quarters (as described above) to form halves, then mix the two halves to form a composite or homogenized sample
6. Place composite or homogenized sample into specified containers
7. Remaining material will be disposed of in accordance with project requirements and applicable regulations
8. Sample containers will be labeled with sample identification number, date, and time of collection and placed on ice in a cooler (target 4° Celsius)
9. Samples selected for laboratory analysis will be documented (chain-of-custody forms), handled, packed, and shipped in accordance with the procedures outlined in the FIP/work plan (or equivalent).

8 WASTE MANAGEMENT

Investigative-Derived Waste (IDW) generated during drilling activities, including soil and excess drilling fluids (if used), decontamination liquids, and disposable materials (plastic sheeting, PPE, etc.) will be stored on site in appropriately labeled containers (disposable materials will be contained separately) and disposed of properly. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log book.

9 DATA RECORDING AND MANAGEMENT

Management of the original documents from the field will be completed in accordance with the site-specific QAPP.

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In general, drilling activities will be documented on appropriate field/log forms as well as in a proper field notebook. All field data will be recorded in indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead.

Initial field logs and chain-of-custody records will be transmitted to the Arcadis Certified Project Manager (CPM) and Technical Lead at the end of each day unless otherwise directed by the CPM. The field team leader retains copies of the field documentation.

Additionally, all documents (and photographs) will be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of drilling activities, soil descriptions, soil boring information, and quantities of materials used.

In addition, the locations of soil borings will be documented photographically and in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

10 QUALITY ASSURANCE

Quality assurance procedures shall be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate TGI.

Field-derived quality assurance blanks will be collected as specified in the FIP/work plan and/or site-specific QAPP, depending on the project quality objectives. Typically, field rinse blanks (equipment blanks) will be collected when non-dedicated equipment (e.g., split-spoon sampler, stainless steel spoon) is used during soil sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminants from other media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities and during transport to the laboratory.

Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.

11 REFERENCES

ASTM D1586 - *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils*. ASTM International. West Conshohocken, Pennsylvania.

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12 ATTACHMENTS

Attachment 1. Soil Boring Log Form

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ATTACHMENT 1

Soil Boring Log Form

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SOIL BORING LOG

Boring/Well _____ Project _____ Page ____ of ____

Site Location _____ Drilling Started _____

Total Depth Drilled _____ Feet Hole Diameter _____ inches Drilling Completed _____

Type of Sample or Coring Device _____ Length and Diameter of Coring Device _____ Sampling Interval _____ feet

Drilling Method _____ Drilling Fluid Used _____

Drilling Contractor _____ Driller _____

Prepared By _____ Helper _____

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD		SAND					GRAVEL				**Udden-Wentworth Description:** principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, additional comments
			clay	silt	very fine	fine	medium	coarse	very coarse	granular	pebble	cobble	boulder	

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SOIL BORING LOG (CONT'D)

Boring/Well _____ Prepared By _____ Page ____ of ____

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD		SAND					GRAVEL				Udden-Wentworth Description: principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, addtl. Comments
			clay	silt	very fine	fine	medium	coarse	very coarse	granular	pebble	cobble	boulder	

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RI-SOP-10

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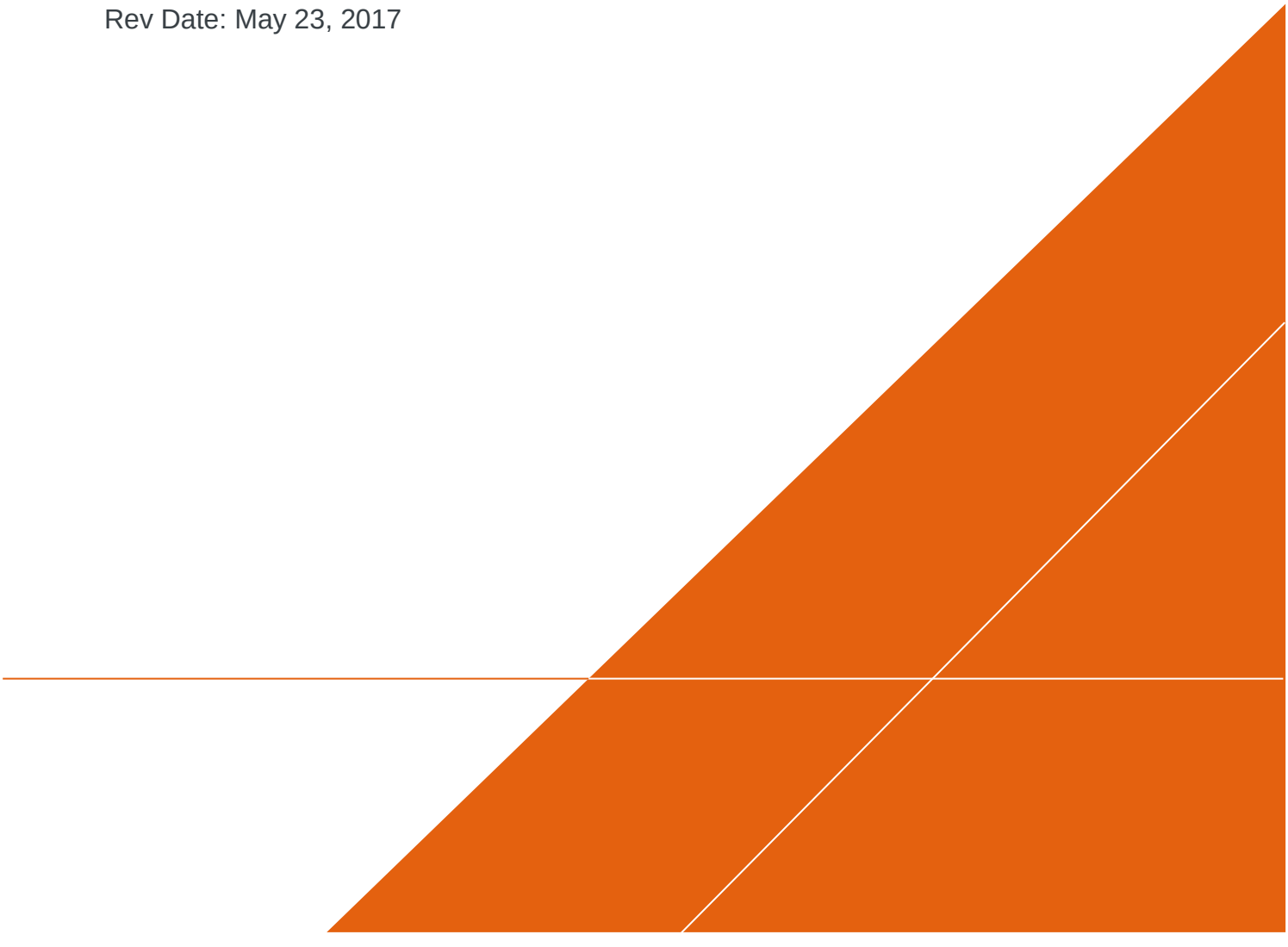
SOP - SAMPLE CHAIN OF CUSTODY

Rev: #1

Rev Date: May 23, 2017



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	April 19, 2017	All	Re-write to COC only	Richard Murphy
1	May 23, 2017	4	Add: Guidance on use of previous version of SOP.	Peter Frederick
		9	Add: Info on COCs for multiple shipping containers	
		7	Modify: Move letter i. to letter m. and change to “when appropriate”	

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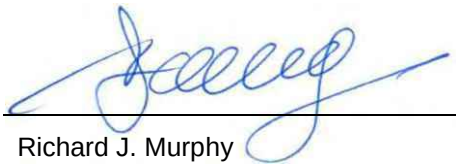
APPROVAL SIGNATURES

Prepared by:		05/23/2017
	Peter C. Frederick	Date:
Technical Expert Reviewed by:		05/23/2017
	Richard J. Murphy	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the general Chain of Custody (COC) procedures and guidance instructions for samples collected from project sites that are relinquished from Arcadis' possession.

COC is defined as the maintenance of an unbroken record of possession of an item from the time of its collection through some analytical or testing procedure. COC is typically documented by a written record of the collection, possession, and handling of samples collected from a project location. Each sample will be tracked by a documented record that efficiently documents the individuals who were responsible for the sample during each successive transfer of that sample to various recipients beyond Arcadis' possession. This information can be used to legally establish the integrity of the samples and therefore the analytical results derived from the samples. This information can be used in addition to other records and documentation regarding the samples, such as field forms, field logs, and photographs.

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A sample is considered under custody if:

- It is in your possession; or
- It is in your view, after being in your possession; or
- It was in your possession and then you then locked it up to prevent tampering; or
- It is in a designated secure area.

Continued use of previous version of SOP:

Although not recommended, Arcadis program-, project-, and client-teams may be able to use the previous version of this SOP provided that it meets all of the quality expectations of Arcadis and client, and meets applicable regulatory requirements. It is up to the program, project, and/or client-team leader to determine whether it is appropriate to adopt the current SOP or to continue using the previous version.

However, all new work not associated with the previous version of this SOP must be performed with the current version of the SOP.

When adopting this new SOP, users of the previous versions must be aware that specific handling, packing, and shipping procedures and guidance has been removed and that those should be addressed within program or project plans (e.g. QAPPs, Work Plans, SAPs, etc.) or in a more detailed SOP or TGI specific to that sampling activity, whether related to media, constituent/analyte, client, state, etc.

In addition, adopting this new SOP will require users to refer to the Arcadis DOT Safety Program for procedures and guidance on the determination and handling, packing, and shipping of samples that are or may be considered hazardous materials.

3 PERSONNEL QUALIFICATIONS

Arcadis personnel performing work under the purview of this SOP will have received appropriate training and have field experience regarding the collection of samples from project locations. Arcadis personnel will have all other applicable and appropriate training relevant to the sampling work and project site.

4 EQUIPMENT LIST

The following list provides materials that may be required for each COC. Project reporting and documentation requirements must be reviewed with the CPM prior to execution of work. Additional materials, tools, equipment, etc. may be required, and project staff are required to verify with the CPM and/or Technical Expert what specific equipment is required to complete the COC.

- Indelible ink pen (preferably either black or blue ink);
- COC form https://thesourceus.arcadissource.com/TKI/Documents/COC%20Form.pdf (**Appendix A**) from either Arcadis, laboratory receiving and analyzing the samples, or other applicable and appropriate entity for the work performed;
- When appropriate, such as for litigation or expert testimony work, custody seals or tape.

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5 CAUTIONS

One way in which the law tries to ensure the integrity of evidence is by requiring proof of the chain of custody by the party who is seeking to introduce a particular piece of evidence.

A proper chain of custody requires three types of affirmations: (1) affirmation that a sample is what it purports to be (for example, soil collected from a specified location and depth); (2) affirmation of continuous possession by each individual who has had possession of the sample from the time it is collected until the time it is analyzed or held by a laboratory; and (3) affirmation by each person who has had possession that sample remained in substantially the same condition and not contaminated or affected by outside influences from the moment one person took possession until the moment that person released the evidence into the custody of another (for example, affirmation that the sample was stored in a secure location where no one but the person in custody had access to it).

Proving chain of custody is necessary to "lay a foundation" for the samples in question, by showing the absence of alteration, substitution, or change of condition.

Ensure that appropriate sample containers with applicable preservatives, coolers, and packing material are planned for and provided at the site at the time of sample collection.

Understand the offsite transfer requirements of the samples for the facility at which samples are collected.

If overnight courier service is required schedule pick-up or know where the drop-off service center is located and the hours of operation.

An Arcadis employee appropriately trained at the correct level of internal hazardous materials/DOT (Department of Transportation) shipping must complete an Arcadis shipping determination to address applicable DOT and IATA (International Air Transport Association) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging, and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.

The person relinquishing possession of the samples or other member of the project team should contact the final recipient of the samples to confirm receipt and review any special provisions on the COC or questions that they may have.

6 HEALTH AND SAFETY CONSIDERATIONS

Follow the health and safety procedures outlined in the project/site Health and Safety Plan (HASP) as well as other applicable H&S requirements, such as:

- Arcadis Hazardous Material/DOT handling, packaging, and shipping training
- Project site-specific H&S training
- Client-specific H&S training
- Constituent-specific H&S training
- Media-specific H&S training

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7 PROCEDURE

Collected samples must be uniquely identified, and properly documented, containerized, labeled with unique identifier, possessed in a secure manner during remainder of sampling event, packaged, and shipped to recipient laboratory.

Sample Identification

The method of sample identification depends on the type of measurement or analyses performed. In some cases, in-situ measurements of existing conditions and/or sample location must be made during sample collection. These data will be recorded directly on field forms, logbooks, or other project record data sheets used to permanently retain this information for the project file. Examples of location identification information includes: latitude/longitudinal measurements, compass directions, well number, building number, floor number, room name, or proximity to a site feature unique to the site. Examples of in-situ measurements are pH, temperature, conductivity, flow measurement, or physical condition of the media being sampled. Physical samples collected are identified by a unique identifying number or code on a sample tag or label. These physical samples are removed from the sample location and transported to a laboratory for analyses.

In some cases, before samples are placed into individual containers and labeled as individual samples, samples may be separated into portions depending upon the analytical methods and required duplicate or triplicate analyses to be performed.

When completing a COC for samples, personnel must complete the following:

1. Written COCs must be completed with indelible ink (preferably either black or blue colored ink).
2. Written COCs must be completed using legible printed writing, and not cursive writing.
3. All entry fields on the COC form must be completed. If information is not applicable for a specific entry field, personnel will either put “N/A” or use a strike-out line or dash like “----------“ to indicate no applicable information is needed for that field.
4. Use of quotation marks or lines/down arrows to represent repetitive/duplicative text in similar fields.
5. Regardless of the type or specific COC form, the following pertinent information must be provided on the COC form:
    a. Arcadis project number
    b. Arcadis project name
    c. Project location, including street address, city, state, building number, providing as much detail as appropriate
    d. Recipient laboratory contact and sample receiving shipping location information
    e. Entities’/persons’ contact information for who will be receiving analytical results
    f. Name of sampler, i.e. person collecting sample and relinquishing possession of samples to the next entity in the chain of custody
    g. Date of sample collection

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h. If appropriate for the sample media, contaminant/constituent of concern, or analytical method, document time of sample collection using standard military time

i. Sample analytical method(s)

j. Turnaround time required for analyses and/or reporting

k. Instructions to laboratory regarding handling, timing, analyses, etc. as applicable and appropriate

l. Printed name and signature of the individual person who collected the samples and relinquishing possession of the samples

m. If appropriate or when documentation of the specific sample collection method will influence how the laboratory handles, prepares, or analyzes the samples, document the sample collection methodology used for collecting the samples (e.g. ASTM D5755)

6. The following additional specific information will be entered on the COC form, regardless of what type of COC is being used:

   a. Unique Sample Identifier – The sample identifier (ID) must be unique to the individual sample it is applied to. The information in which the sample ID conveys is determined by the CPM, Technical Expert, and/or other project team members in advance of sample collection so that sample identification is consistently applied for the project. The sample nomenclature may be dictated by a specific client, program, or project database and require unique identification for each sample collected for the project. Consult with the CPM and/or Technical Expert for additional information regarding sample identification.

      The sample ID could convey specific information regarding the sample to aid personnel in recognizing what the sample represents, or they may be arbitrary so as to facilitate the anonymity of the sample location, media, constituent of concern, project site, etc.

      Examples of unique identifiers include:

      1. Well locations, grid points, or soil boring identification numbers (e.g., MW-3, X-20, SB-30). When the depth interval is included, the complete sample ID would be “SB-30 (0.5-1.0) where the depth interval is in feet. Please note it is very important that the use of hyphens in sample names and depth units (i.e., feet or inches) remain consistent for all samples entered on the chain of custody form. DO NOT use the apostrophe or quotes in the sample ID.

      2. Sample names may also use the abbreviations “FB,” “TB,” and “DUP” as prefixes or suffixes to indicate that the sample is a field blank, trip blank, or field duplicate, respectively.

   b. List the date of sample collection. All indicated dates must be formatted using either mm/dd/yy (e.g., 03/07/09) or mm/dd/yyyy (e.g. 03/07/2009).

   c. When appropriate for the analytical procedure used, list the local time that the sample was collected. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15.

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d. Samples should be indicated to be either “Grab” or “Composite”. Grab samples are collected from only one unique location at one specific point in time.

e. Composite samples are a group of individual samples that are combined for analysis in their totality. Composite samples need to be documented if they are either collected from a number of different locations over a broader area to be representative of the entire area being sampled, or if they are representative of a single location over an extended period of time.

f. If used, preservatives for the individual sample will be noted.

g. The requested analytical method(s) that the samples are being analyzed for must be indicated. As much detail, as necessary, should be presented to allow the analytical laboratory to properly analyze the samples. For example, polychlorinated biphenyl (PCB) analyses may be represented by entering “EPA Method 8082 – PCBs” or “EPA PLM 600-R93-116.” In cases where multiple analytical methods and/or analytical parameters are required for an individual sample, each method should be indicated for the sample (e.g., EPA 8082/8260/8270 or EPA PLM/400-point count).

h. If there are project-specific sample analytes to be reported, they should be specifically listed for each individual sample (e.g., 40 CFR 264 Appendix IX).

i. The total number of containers for each analytical method requested should be documented. This information may be included under the parameter or as a total for the sample.

j. When necessary, note which samples should be used for site specific matrix spikes.

k. Indicate special project-specific requirements pertinent to the handling, shipping, or analyses. These requirements may be on a per sample basis such as “extract and hold sample until notified,” or may be used to inform the laboratory of special reporting requirements for the entire sample delivery group (SDG).

l. Indicate turnaround time (TAT) required for samples on COC. If individual samples have differing TATs, the different TATs for each sample or groups of samples must be clearly indicated.

m. Provide contact name and phone number in the event that problems are encountered when samples are received at the laboratory. The person relinquishing possession of the samples or other member of the project team should contact the final recipient of the samples to confirm receipt and review any special provisions on the COC or questions that they may have.

n. If available, attach the Laboratory Task Order or Work Authorization forms.

o. The “Relinquished By” field must contain the signature of the Arcadis person who relinquished custody of the samples to the next entity in the chain of custody, which may be another person, the shipping courier, or the analytical laboratory.

p. Dates and times must be indicated using the following format:

    1) Date: either mm/dd/yy e.g., 01/01/17 OR mm/dd/yyyy e.g., 01/01/2017

    2) Time: use military format, e.g. 9:30 a.m. is 0930 and 9:30 p.m. is 2130

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q. The “Received By” section is signed by sample courier or laboratory representative who received the samples from the sampler or it is signed upon laboratory receipt from the overnight courier service.

4. When more than one page of the COC form is required to complete the total number of samples, use as many sheets as necessary to accurately and clearly document the samples and information. Some COCs may have a standard first page/cover page, and subsequent pages may not contain all the detailed fields as the first page/cover page. Ensure that any subsequent pages convey all of the necessary and pertinent information for each individual sample as required in this procedure document.
5. Pages of the COC must retain a page count of the total number of pages; e.g., Page 1 of 3, Page 2 of 3, Page 3 of 3.
6. Upon completing the COC forms, forward the original signed COC with the sample package. Ensure that the original COC form is secured with the sample package so that it remains with the physical samples for the duration of transport and handling to its final destination and ensure that the COC form will not be become damaged or rendered unreadable due to sample breakage/leakage if stored inside the sample shipping container or outside influences if COC is stored in an outside plastic pouch to the container.
7. If you’ve collected enough samples that would require more than one container to ship them all to the same laboratory or location, then each separate/individual container that contains any number of samples must have a separate COC representing only those samples contained within that specific container. For example, if you have 3 total shipping containers for all of your samples, you must have a total of 3 separate, individual COCs for each of the 3 containers representing only those samples in their representative container. Thus, every container holding samples must have its own, individual COC.
8. If electronic chain of custody (eCOC) forms are utilized, ensure that the requirements of this procedure and guidance instructions are followed to the extent possible. Verify that proper signature and COC procedures are maintained with the CPM and/or Technical Expert when using eCOC.

8 WASTE MANAGEMENT

Not Applicable.

9 DATA RECORDING AND MANAGEMENT

The original signed COC shall be submitted with the samples. Copies of COC records will be transmitted to the CPM or designee at the end of each day unless otherwise directed by the CPM. The sampling team leader retains copies of the chain of custody forms for filing in the project file. Record retention shall be in accordance with client- and project-specific requirements and Arcadis policies, the most stringent will apply.

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10 QUALITY ASSURANCE

COC forms will be legibly completed in accordance with this procedure and guidance instruction document, as well as other applicable and appropriate project documents such as Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPP), Work Plan, or other project guidance documents.

COC records will be reviewed by the CPM or their appropriate designee for completeness and accuracy to the applicable requirements. Non-conformances will be noted and corrected in a timely manner on the copies retained by Arcadis as well as contacting the ultimate receiving entity for correction to the originally signed COC in their possession.

11 REFERENCES

Arcadis Client Document Retention Guide

Arcadis Transportation Safety Program requirements, procedures, and guidance instructions

EPA Samplers' Guide – Contract Laboratory Program Guidance for Field Samplers, EPA document EPA-540-R014-013 October 2014

EPA Region III – *Sample Submission Procedures for the Office of Analytical Services and Quality Assurance (OASQA) Laboratory Branch* revision 13.0 January 29, 2014

EPA Region I Office Environmental Measurement and Evaluation – *Standard Operating Procedures for Chain of Custody of Samples* revision 1 March 25, 2002

EPA Region IV Science and Ecosystem Support Division *Operating Procedure for Sample and Evidence Management* January 29, 2013

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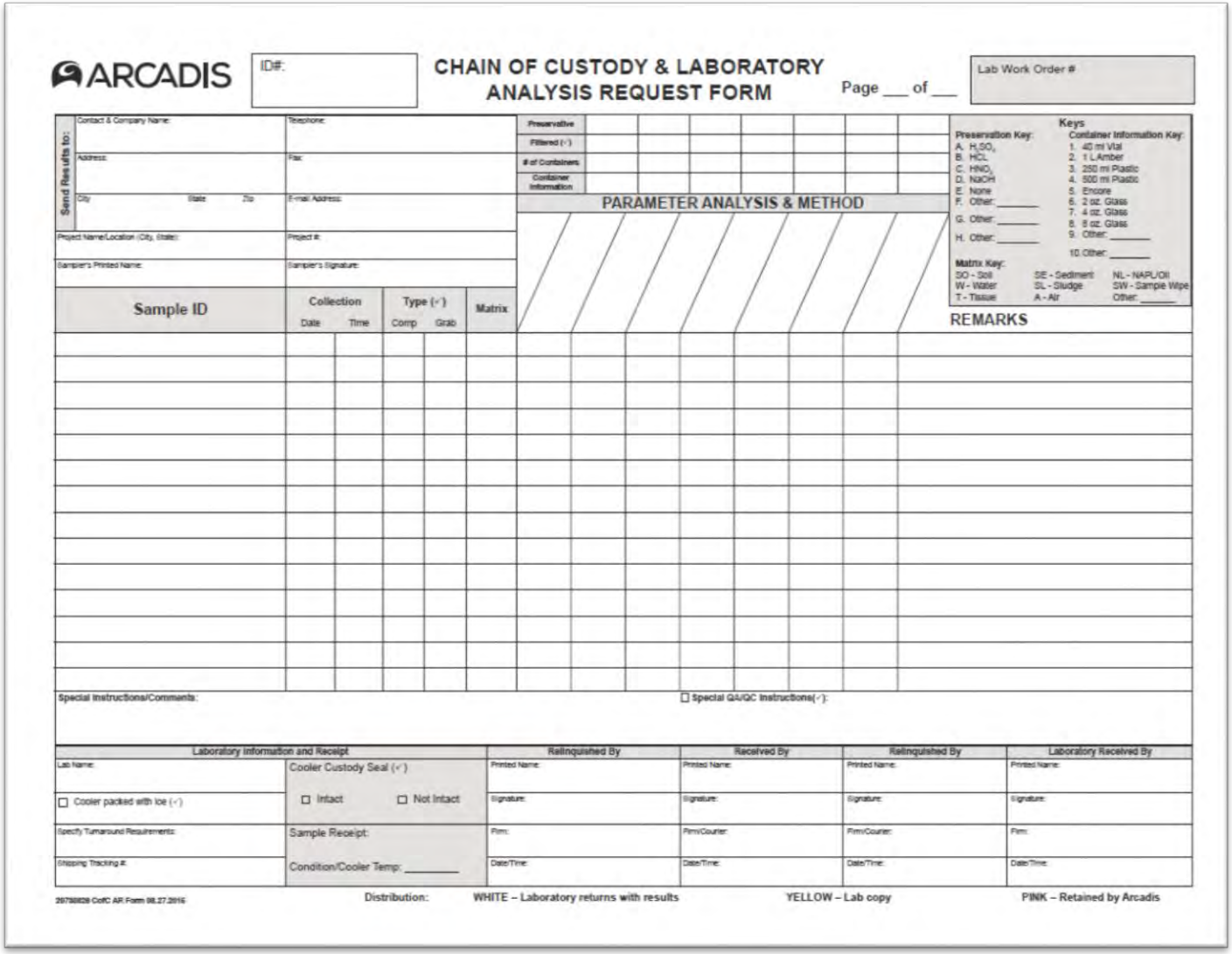
APPENDIX A

Chain of Custody Form

[click image below to access form]



ARCADIS

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:

Telephone:

Address:

Fax:

City State Zip

E-mail Address:

Project Name/Location (City, State):

Project #:

Sampler's Printed Name:

Sampler's Signature:

Preservative

Filtered (✓)

# of Containers

Container Information

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil
W - Water
T - Tissue
SE - Sediment
SL - Sludge
A - Air
NL - NAPL/Oil
SW - Sample Wipe
Other: _____

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix	REMARKS

Special Instructions/Comments:

☐ Special QA/QC Instructions(✓):

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with Ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

29790828 CofC AR Form 08.27.2015

Distribution: WHITE – Laboratory returns with results YELLOW – Lab copy PINK – Retained by Arcadis

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APPENDIX A
Chain of Custody Form
[click image below to access form]

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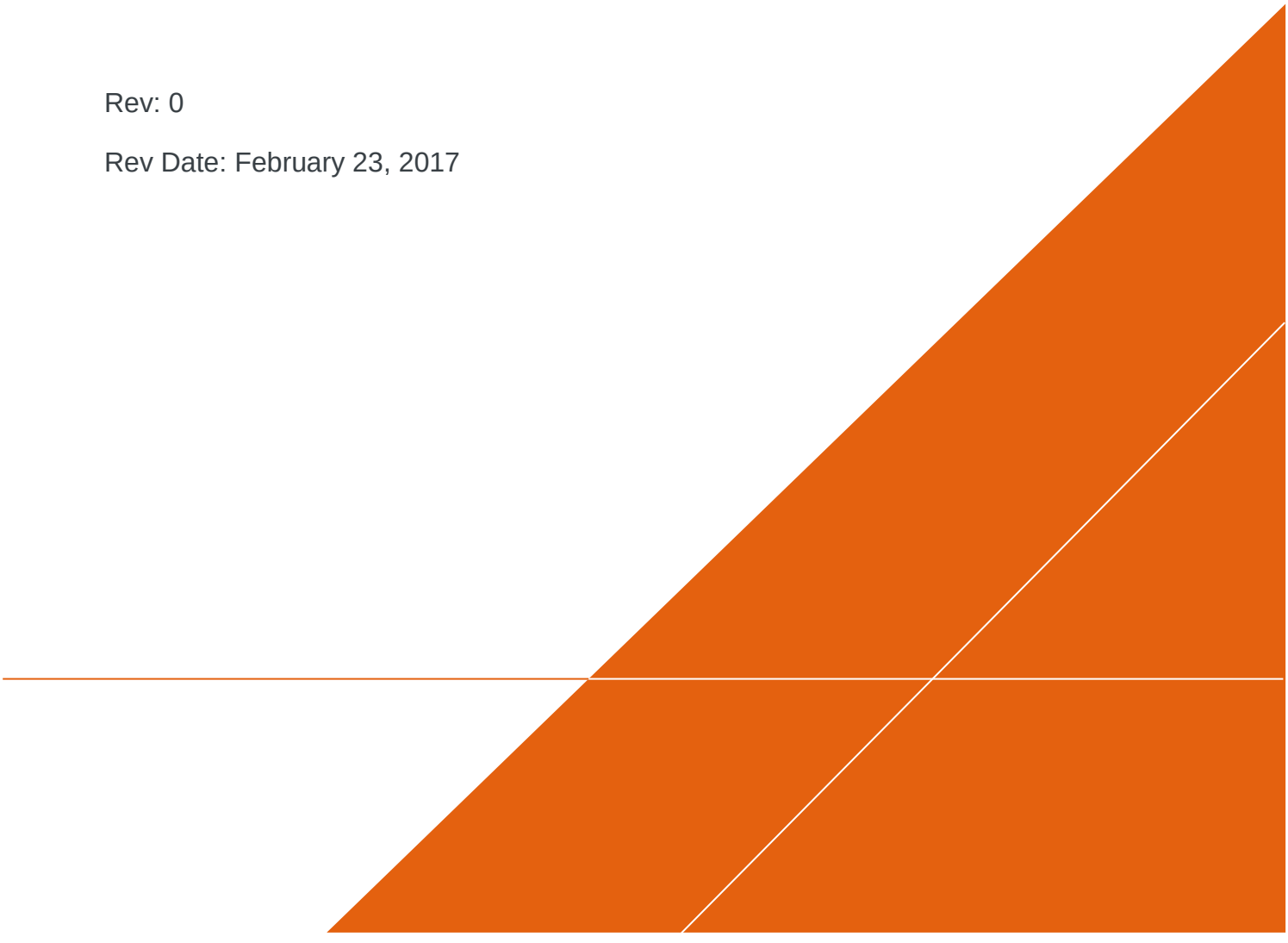
TGI – GROUNDWATER AND SOIL SAMPLING EQUIPMENT DECONTAMINATION

Rev: 0

Rev Date: February 23, 2017



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	February 23, 2017	ALL	Conversion from SOP to TGI	Cassandra McCloud / Pete Frederick

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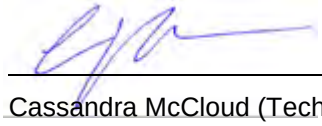
APPROVAL SIGNATURES

Prepared by:  Date: 02/23/2017

Derrick Maurer

Technical Expert Reviewed by:  Date: 02/23/2017

Cassandra McCloud (Technical Expert)

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

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In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

Decontamination is performed on sampling equipment prior to sample collection to ensure that the sampling equipment that contacts a sample, or monitoring equipment that is brought into contact with environmental media to be sampled, is free from analytes of interest and/or constituents that could interfere with laboratory analysis for analytes of interest. Sampling equipment must be appropriately cleaned prior to use for sampling or coming into contact with environmental media to be sampled, and following completion of the sampling event prior to shipment or storage. The effectiveness of the decontamination procedure should be verified by collecting and analyzing equipment blank samples.

The sampling equipment cleaning procedures described herein includes pre-field, in the field, and post-field cleaning of sampling equipment which may be conducted at an established equipment decontamination area (EDA) on site, as appropriate and necessary. Sampling equipment that may require decontamination at a given site includes: soil sampling tools; groundwater, sediment, and surface-water sampling devices; water testing instruments; down-hole instruments; and other activity-specific sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between each

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sample collected, and prior to placing sampling equipment in protective cases, or containers for transport. Cleaning procedures for sampling equipment should be monitored by collecting equipment blank samples as required in project work plans, field sampling plans, quality assurance project plans (QAPP), or other pertinent project documents. Dedicated and/or single-use (i.e., not to be re-used) sampling equipment will not require decontamination.

3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and Technical Guidance Instructions (TGIs) and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project health and safety plan (HASP) and other documents will identify other training requirements or access control requirements.

4 EQUIPMENT LIST

The equipment required for equipment decontamination is presented below:

- Health and safety equipment, including appropriate PPE, as required in the site Health and Safety Plan (HASP)
- Deionized water that meets that analytical criteria for deionized water with no detectable constituents above the reporting limits for the methods to be used and analytes being analyzed for. Deionized water is used for inorganics, and organic-free water for VOCs, SVOCs, pesticides, etc.
- Non-phosphate detergent such as Alconox or, if sampling for phosphorus or phosphorus-containing compounds, Liquinox (or equivalent).
- Tap water
- Rinsate collection plastic containers
- DOT-approved waste shipping container(s), as specified in the work plan, field sampling plan, or regulatory requirements if decontamination waste is to be shipped for disposal
- Brushes
- Large heavy-duty garbage bags
- Spray bottles
- (Optional) – Isopropyl alcohol (free of ketones) or methanol. These can be wipes or diluted with water (usually 1part isopropyl/methanol to 10 parts water) if a spray is needed.
- Airtight, sealable plastic baggies, such as Ziploc-type
- Plastic sheeting

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5 CAUTIONS

Rinse equipment thoroughly and allow the equipment to dry before re-use or storage to prevent introducing solvent into sample medium. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry. Ensure all rinsate materials do not adversely affect sample collection efficiency or analytical results.

Store decontaminated equipment in a clean, dry environment. Do not store near combustion engine exhausts. Properly containerize equipment to ensure cross-contamination doesn't happen from other uncontaminated surfaces or equipment.

If equipment is damaged to the extent that decontamination is uncertain due to cracks, gouges, crevices, or dents, the equipment should not be used and should be discarded or submitted for repair prior to use for sample collection.

A proper shipping determination regarding hazardous materials will be performed by a DOT-trained individual for cleaning materials shipped by Arcadis.

Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock.

6 HEALTH AND SAFETY CONSIDERATIONS

Review the safety data sheets (SDS) for the cleaning agents and materials used in decontamination. If solvent is used during decontamination, use appropriate PPE and work in a well-ventilated area and stand upwind while applying solvent to equipment. Apply solvent in a manner that minimizes potential for exposure to workers and bystanders. Follow health and safety procedures outlined in the HASP.

7 PROCEDURE

A designated area will be established to clean sampling equipment in the field prior to and following sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area, but not at a location that expose equipment to contamination (i.e. exposed to combustion engine exhaust). Detergent solutions will be prepared in clean containers for use in equipment decontamination. Decontaminated equipment should be handled by workers wearing clean gloves, properly changed to prevent cross-contamination.

Cleaning Sampling Equipment

1. Wash the equipment/pump with potable water.
2. Wash with detergent solution (Alconox, Liquinox or equivalent) to remove all visible particulate matter and any residual oils or grease.
3. If equipment is very dirty, precleaning gross debris with a brush and tap water may be necessary.
4. If non-aqueous phase liquids are present, the use of isopropyl alcohol (free of ketones) or methanol is recommended. Cloth wipes or diluted solution can be used to remove the non-aqueous phase liquids that are hard to remove with detergent solution in step 2. Consult with project manager if

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non-aqueous phase liquids are present onsite and design an appropriate decontamination procedure that includes step 4.

5. Rinse with deionized water.

Decontaminating Submersible Pumps

Submersible pumps may be used during well development, groundwater sampling, or other investigative activities. The pumps must be cleaned and flushed before and between uses. This cleaning process will consist of an external detergent solution wash and tap water rinse, a flush of detergent solution through the pump, followed by a flush of potable water through the pump. Flushing will be accomplished by using an appropriate container filled with detergent solution and another container filled with potable water. The pump should be flushed with deionized water as the last step prior to use. The pump will run long enough to effectively flush the pump housing and hose (unless new, disposable hose is used). Disconnect the pump from the power source before handling. The pump and hose should be placed on or in clean polyethylene sheeting to avoid contact with the ground surface.

8 WASTE MANAGEMENT

Equipment decontamination rinsate will be managed in conjunction with all other waste produced during the field sampling effort. Waste management procedures are outlined in the work plan or Waste Management Plan (WMP).

9 DATA RECORDING AND MANAGEMENT

Equipment cleaning and decontamination will be noted in the field notebook for project documentation. Information will include the type of equipment cleaned, the decontamination location, specific procedures utilized, solvents and/or cleaning agents used, source of water, and deviations or omissions from this TGI.

Unusual field conditions should be noted if there is potential to impact the efficacy of the decontamination or subsequent sample collection.

An inventory of the solvents brought on site and used and removed from the site will be maintained in the project documentation. Records will be maintained for solvents used in decontamination, including lot number and expiration date.

Containers with decontamination fluids will be labeled.

10 QUALITY ASSURANCE

Equipment blanks should be collected to verify that the decontamination procedures are effective in minimizing potential for cross contamination. The equipment blank is prepared by pouring deionized water (or organic-free water, for organic analyses) over the clean and dry tools and collecting the water into appropriate sample containers. Equipment blanks should be analyzed for the same set of parameters that are performed on the field samples collected with the equipment that was cleaned as specified in the sampling and analysis plan. Equipment blanks are collected per equipment set, which represents all of the tools needed to collect a specific sample.

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11 REFERENCES

USEPA Region 9 - Field Sampling Guidance #1230, Sampling Equipment Decontamination.

USEPA Region 1 - Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells.

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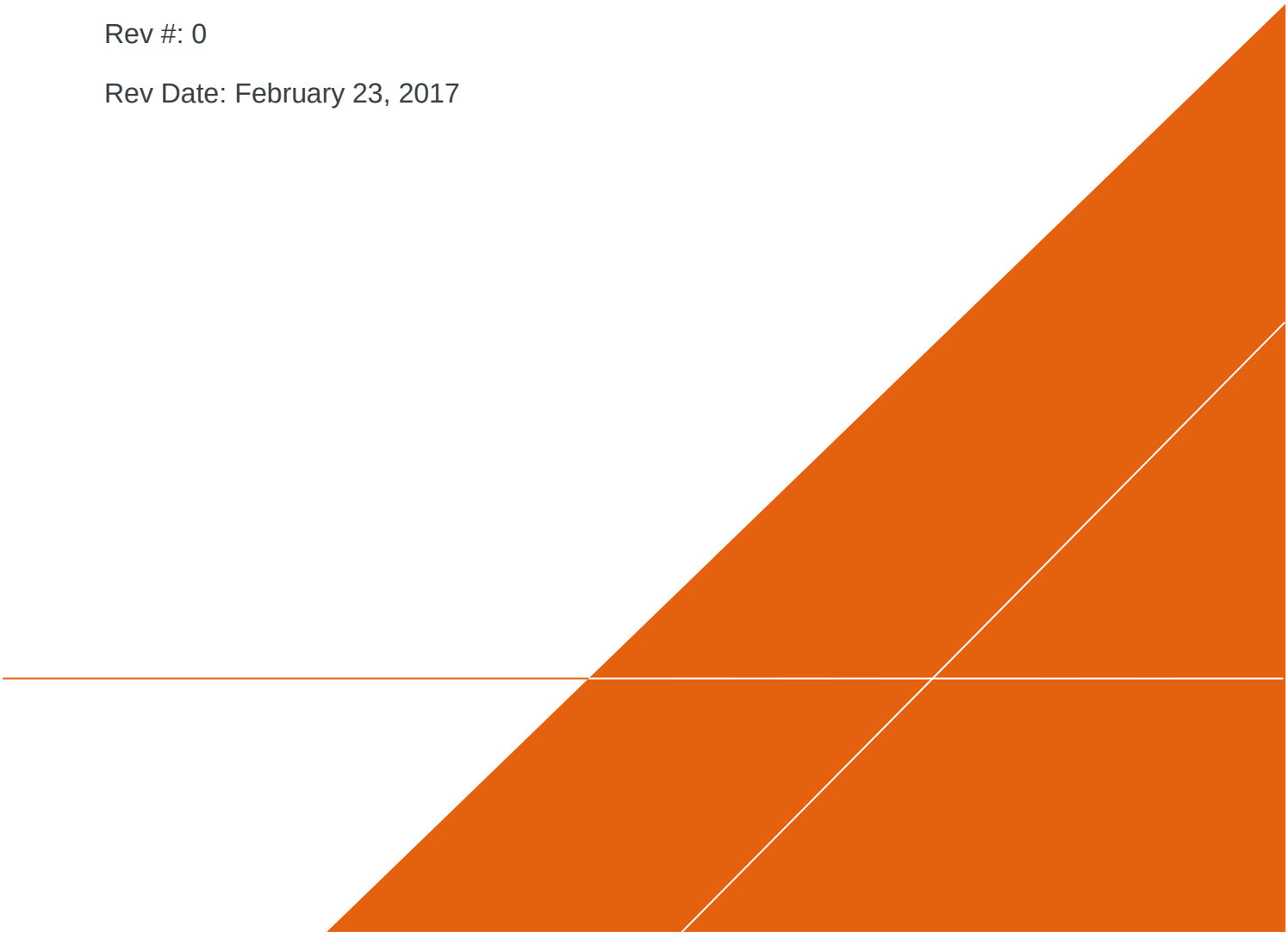
TGI - INVESTIGATION-DERIVED WASTE HANDLING AND STORAGE

Rev #: 0

Rev Date: February 23, 2017



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0	February 23, 2017	ALL	Conversion from SOP to TGI	Ryan Mattson / Peter Frederick

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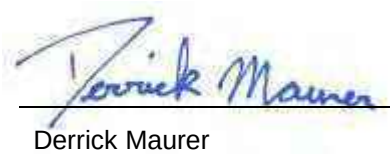
APPROVAL SIGNATURES

Prepared by:		02/23/2017
	Derrick Maurer	Date:
Technical Expert Reviewed by:		02/23/2017
	Ryan Mattson (Technical Expert)	Date:

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2 SCOPE AND APPLICATION

The objective of this Technical Guidance Instruction (TGI) is to describe the procedures to manage investigation-derived wastes (IDW), both hazardous and nonhazardous, generated during site activities, which may include, but are not limited to: drilling, trenching/excavation, construction, demolition, monitoring well sampling, soil sampling, decontamination and remediation. For the purposes of this TGI, IDW is considered to be discarded materials which are defined as solid waste by United States Environmental Protection Agency (EPA) standard 40 CFR § 261.2 (which may include liquids, solids, or sludges). IDW may include soil, groundwater, drilling fluids, decontamination liquids, as well as contaminated personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials. Hazardous or uncharacterized IDW will be collected and staged at the point of generation. Quantities small enough to be containerized in 55-gallon drums will be taken to a designated temporary onsite storage area (discussed in further detail under Drum Storage) pending characterization and disposal. IDW materials will be characterized using process knowledge and appropriate laboratory analyses to determine the waste classification and evaluate proper safe handling and disposal methods.

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This TGI describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures necessary for proper handling and storage of IDW up to the time it is properly transported from the project site and disposed. The procedures included in this TGI for handling and temporary storage of IDW are based on the EPA's guidance document *Guide to Management of Investigation Derived Wastes* (USEPA, 1992). IDW is assumed to be contaminated with the site constituents of concern (COCs) until analytical evidence indicates otherwise. IDW will be managed to ensure the protection of human health and the environment and will comply with all applicable or relevant and appropriate requirements (ARAR). Although not comprehensive, the following laws and regulations on Hazardous Waste Management should be considered as potential ARAR. It is the Arcadis Certified Project Manager (CPM) and/or designated Technical Expert to determine which laws and regulations, at all levels of government, are applicable to each project site and activity falling under this TGI.

Federal Laws and Regulations

- Resource Conservation and Recovery Act (RCRA) 42 USC § 6901-6987.
- Federal Hazardous Waste Regulations 40 CFR § 260-265

Department of Transportation (DOT) Hazardous Materials Transportation 49 CFR

Occupational Safety and Health Administration (OSHA) Regulations 29 CFR

State Laws and Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.

Regional, County, Municipal, and Local Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.

Initial Storage

Pending characterization, IDW will be temporarily stored appropriately within each area of contamination (AOC). Under RCRA, "storage" is defined as the "holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere" (40 CFR § 260.10). The onsite waste staging area will be in a secure and controlled area. Uncharacterized wastes are considered potentially hazardous wastes and must be stored in DOT approved packaging. Liquid wastes must be stored in DOT approved closed head drums or other approved containers (e.g., portable tank containers) that are compatible with the type of material stored therein. Solid materials must be stored in DOT approved open head drums where practicable. Larger quantities of solid IDW can be containerized in bulk containers (such as in a roll-off box). Soil from large excavation projects may be managed in stockpiles with within the AOC and does not need to be containerized until exiting the AOC.

Characterization

Waste characterization can either be based on generator knowledge, such as using historical process knowledge and safety data sheets (SDS), or can be based upon characterization sampling analytical results. IDW typically is not characterized using SDS as it is a mixture of aged chemicals and environmental media. Historical process knowledge should be used to determine if the IDW is a listed hazardous waste (40 CFR § 261.31-33). If the IDW is not a listed hazardous waste, waste

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characterization can be completed by laboratory analysis of representative samples of the IDW. The laboratory used for waste characterization analysis must have the appropriate state and federal accreditations and may be required to be pre-approved by the Client. IDW will be classified as RCRA hazardous or non-regulated under RCRA based on the waste characterization determination.

If IDW is characterized as RCRA hazardous waste, RCRA and DOT requirements must be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR § 262 and 49 CFR § 171-178. Waste material classified as RCRA nonhazardous may be handled and disposed of as nonhazardous waste in accordance with applicable federal, state, and local regulations.

Storage Time Limitations

Containerized hazardous wastes can be temporarily stored for a maximum of 90 calendar days from the accumulation start date for a large quantity generator or a maximum of 180 calendar days from the accumulation start date for a small quantity generator. Wastes classified as nonhazardous may be handled and disposed of as nonhazardous waste and are not subject to storage time limitations.

This is TGI may be modified by the CPM and/or Technical Expert for a specific project or client program, as required, dependent upon client requirements, site conditions, equipment limitations, or limitations imposed by the procedure. The resulting procedure employed to execute the work will be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes will be discussed with the CPM and/or Technical Expert as soon as practicable, and if approved to be performed, be documented.

3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have current regulatory- and Arcadis-required health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and cardiopulmonary resuscitation (CPR), as needed. Personnel handling and packaging hazardous waste and performing hazardous waste characterizations must have RCRA hazardous waste management training per 40 CFR § 264.16.

Although not common practice, in certain situations Arcadis personnel may sign waste profiles and/or waste manifests on a case by case basis for clients, provided the appropriate agreement is in place between Arcadis and the client documenting that Arcadis is not the generator, but is acting as an authorized representative of the generator. Arcadis personnel who sign waste profiles and/or waste manifests will have both current RCRA hazardous waste management training per 40 CFR § 264.16 and current DOT hazardous materials transportation training per 49 CFR § 172.704. Arcadis field personnel will also comply with client-specific training. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and Technical Guidance Instructions (TGIs) and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project health and safety plan (HASP) and other documents will identify other training requirements or access control requirements.

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4 EQUIPMENT LIST

The Following Materials, as required, will be available for IDW handling and Storage:

- Appropriate personal protective equipment as specified in the Site Health and Safety Plan (HASP)
- DOT approved containers
- Hammer
- Leather gloves
- Drum dolly
- Appropriate drum labels (outdoor waterproof self-adhesive)
- Portable tank container
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as determined by the CPM and/or Technical Expert.
- Indelible ink and/or permanent marking pens
- Plastic sheeting
- Appropriate sample containers, labels, and forms
- Stainless-steel bucket auger
- Stainless steel spatula or knife
- Stainless steel hand spade
- Stainless steel scoop
- Digital camera
- Field logbook

5 CAUTIONS

Filled drums can be very heavy, become unbalanced, or spill its contents. Therefore, use appropriate moving techniques and equipment for safe handling. Similar media (e.g. soils with other soils; or liquids with other liquids) will be stored in the same drums to aid in sample analysis and disposal. Drum lids must be secured to prevent rainwater from entering the drums and leakage during movement. Drums containing solid material may not contain any free liquids. Waste containers stored for extended periods of time may be subject to deterioration. Drum Over Packs may be used as secondary containment. All drums must be visually inspected for condition to ensure that they are in good condition without visible evidence of rusting, holes, breakage, etc., to prevent potential leakage and facilitate subsequent disposal. All drum lids must be verified as having a properly functioning secured lid prior to use.

6 HEALTH AND SAFETY CONSIDERATIONS

Click here and enter text] As determined by the site's known and suspected hazards, appropriate PPE must be worn by all field personnel within the designated work area. Exposure air monitoring may be required during certain field activities as required in the Site Health and Safety Plan. If soil excavation in areas with potentially hazardous contaminants is possible, contingency plans will be developed to address the potential for encountering gross contamination or non-aqueous phase liquids. All excavation

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activities shall be in compliance with OSHA standard 29 CFR 1926.651 Excavations, and any other applicable regulations.

Arcadis field personnel and subcontractors will be trained in and perform their work in compliance with all applicable federal, state, and local health and safety regulations as well as Arcadis' HASP and applicable Client health and safety requirements.

7 PROCEDURE

Specific waste temporary storage and handling procedures to be used are dependent upon the type of generated waste, including type of media (e.g. soils or free liquids) and constituents of concern. For this reason, IDW can be stored in a secure location onsite in separate 55-gallon storage drums, where solids can be stockpiled onsite (if nonhazardous) and purge water may be stored in portable tank containers. Waste materials such as broken sample bottles or equipment containers and wrappings will be stored in 55-gallon drums unless they were not in contact with sample media.

Management of IDW

Minimization of IDW should be considered by the project team during all phases of the project. Site managers may want to consider techniques such as replacing solvent based cleaners with aqueous-based cleaners for decontamination of equipment, reuse of equipment (where it can be properly decontaminated), limitation of traffic between exclusion and support zones, and drilling methods and sampling techniques that minimize the generation of waste. Alternative drilling and subsurface sampling methods may include the use of small diameter boreholes, as well as borehole testing methods such as a core penetrometer or direct push technique instead of coring.

Drum Storage

Drums containing hazardous waste will be stored in accordance with the requirements of 40 CFR 265 Subpart I (for containers) and 265 Subpart DD (for containment buildings). All 55-gallon drums will be stored at a secure, centralized onsite location that is readily accessible for vehicular pick-up. Drums confirmed as, or assumed to contain hazardous waste will be stored over an impervious surface provided with secondary spill containment. The storage location will, for drums containing liquid, have a containment system that can contain at least the larger of 10% of the aggregate volume of staged materials or 100% of the volume of the largest container. Drums will be closed during storage and be in good condition in accordance with the Guide to Management of Investigation-Derived Wastes (USEPA, 1992).

Hazardous Waste Determination

Waste material must be characterized to determine if it meets any of the federal definitions of hazardous waste as required by 40 CFR § 262.11. If the waste does not meet any of the federal definitions, it must then be established if any state-specific or local-specific hazardous waste criteria exist/apply.

Generator Status

Once hazardous waste determination has been made, the generator status will be determined. Large quantity generators (LQG) are generators who generate more than 1,000 kilograms of hazardous waste in a calendar month. Small quantity generators (SQG) of hazardous waste are generators who generate

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greater than 100 kilograms but less than 1,000 kilograms of hazardous waste in a calendar month. Conditionally exempt small quantity generators (CESQG) are generators who generate less than 100 kilograms of hazardous waste per month. Please note that a generator status may change from month to month and that a notice of this change is usually required by the generator's state agency.

Accumulation Time for Hazardous Waste

A LQG may accumulate hazardous waste on site for 90 calendar days or less without a permit and without having interim status, provided that such accumulation is in compliance with requirements in 40 CFR § 262.34. A SQG may accumulate hazardous waste on site for 180 calendar days or less without a permit or without having interim status, subject to the requirements of 40 CFR § 262.34(d). CESQG requirements are found in 40 CFR § 261.5. NOTE: The CESQG and SQG provisions of 40 CFR § 261.5, 262.20(e), 262.42(b) and 262.44 may not be recognized by some states (e.g., California and Rhode Island). State-specific and local-specific regulations must be reviewed and understood prior to the generation of hazardous waste.

Satellite Accumulation of Hazardous Waste Satellite accumulation (SAA) will mean the accumulation of as much as fifty-five (55) gallons of hazardous waste, or the accumulation of as much as one quart of acutely hazardous waste, in containers at or near any point of generation where the waste initially accumulates, which is under the control of the operator of the process generating the waste, without a permit or interim status and without complying with the requirements of 40 CFR § 262.34(a) and without any storage time limit, provided that the generator complies with 40 CFR § 262.34(c)(1)(i).

Once more than 55 gallons of hazardous waste accumulates in SAA, the generator has three days to move this waste into storage.

Storage recommendations for hazardous waste include:

- Ignitable Hazardous wastes must be >50 feet from the property line per 40 CFR § 265.176 (LQG generators only).
- Hazardous waste must be stored on a concrete slab (asphalt is acceptable if there are no free liquids in the waste) per 40 CFR § 265.176.
- Drainage must be directed away from the accumulation area.
- Area must be properly vented.
- Area must be secure.

Drum/Container Labeling

Drums will be labeled on both the side and lid of the drum using a permanent marking pen. Old drum labels must be removed to the extent possible, descriptions crossed out should any information remain, and new labels affixed on top of the old labels. Other containers used to store various types of waste (e.g., polyethylene tanks, roll-off boxes, end-dump trailers, etc.) will be labeled with an appropriate "Waste Container" or "Testing in Progress" label pending characterization. Drums and containers will be labeled as follows:

- Appropriate waste characterization label (Pending Analysis, Hazardous, or Nonhazardous)
- Waste generator's name (e.g., client name)
- Project Name
- Name and telephone number of Arcadis project manager

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- Composition of contents (e.g., used oil, acetone 40%, toluene 60%)
- Media (e.g., solid, liquid)
- Accumulation start date
- Drum number of total drums as reconciled with the Drum Inventory maintained in the field log book.

IDW containers will remain closed except when adding or removing waste. Immediately upon beginning to place waste into the drum/container, a “Waste Container” or “Pending Analysis” label will be filled out to include the information specified above, and affixed to the container. Once the contents of the container are identified as either non-hazardous or hazardous, the following additional labels will be applied.

- Containers with waste determined to be non-hazardous will be labeled with a green and white "Nonhazardous Waste" label over the "Waste Container" label.
- Containers with waste determined to be hazardous will be stored in an onsite storage area and will be labeled with the "Hazardous Waste" label and affixed over the "Waste Container" label.

The ACCUMULATION DATE for the hazardous waste is the date the waste is first placed in the container and is the same date as the date on the "Waste Container" label. DOT hazardous class labels must be applied to all hazardous waste containers for shipment offsite to an approved disposal or recycling facility. In addition, a DOT proper shipping name will be included on the hazardous waste label. The transporter should be equipped with the appropriate DOT placards. However, placarding or offering placards to the initial transporter is the responsibility of the generator per 40 CFR § 262.33.

Inspections and Documentation

All IDW will be documented as generated on a Drum Inventory Log maintained in the field log book. The Drum Inventory will record the generation date, type, quantity, matrix and origin (e.g., Boring-1, Test Pit 3, etc.) of materials in every drum, as well as a unique identification number for each drum. The drum inventory will be used during drum pickup to assist with labeling of drums. The drum storage area and any other areas of temporarily staged waste, such as soil/debris piles, will be inspected weekly. The weekly inspections will be recorded in the field notebook or on a Weekly Inspection Log. Digital photographs will be taken upon the initial generation and drumming/staging of waste, and final labeling after characterization to document compliance with labeling and storage protocols, and condition of the container. Evidence of damage, tampering or other discrepancy should be documented photographically.

Emergency Response and Notifications

Specific procedures for responding to site emergencies will be detailed in the HASP. If the generator is designated as a LQG, a Contingency Plan will need to be prepared to include emergency response and notification procedures per 40 CFR § 265 Subpart D. In the event of a fire, explosion, or other release which could threaten human health outside of the site or when Client or ARCADIS has knowledge of a spill that has reached surface water, Client or ARCADIS must immediately notify the National Response Center (800-424-8802) in accordance with 40 CFR § 262.34. Other notifications to state and/or other local regulatory agencies may also be necessary.

Drilling Soil Cuttings and Muds

Soil cuttings are solid to semi-solid soils generated during trenching activities, subsurface soil sampling, or installation of monitoring wells. Depending on the drilling method, drilling fluids known as "muds" may

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be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Soil cuttings will be labeled and stored in 55-gallon drums with bolt-sealed lids.

Excavated Solids

Excavated solids may include, but are not limited to: soil, fill, and construction and demolition debris. Prior to permitted treatment or offsite disposal, potentially hazardous excavated solids may be temporarily stockpiled onsite as long as the stockpile remains in the same AOC from where it was excavated. Potentially hazardous excavated solids removed from the AOC must be immediately containerized in labeled drums or closable top roll-offs lined with 9-mil polyvinyl chloride (PVC) sheeting and are subject to LQG storage time limits. Nonhazardous excavated solids can be stockpiled either inside or outside of the AOC, do not have to be containerized and are not subject to hazardous waste regulations. Potentially hazardous excavated solids must not be mixed with nonhazardous excavated solids. All classes of excavated solid stockpiles should be maintained in a secure area onsite. At a minimum, the floor of the stockpile area will be covered with a 20-mil high density polyethylene liner that is supported by a foundation or at least a 60-mil high density polyethylene liner that is not supported by a foundation. The excavated material will not contain free liquids. The owner/operator will provide controls for windblown dispersion, run-on control, and precipitation runoff. The run-on control system will prevent flow onto the active portion of the pile during peak discharge from at least a 25-year storm and the run-off management system will collect and control at least the water volume resulting from a 24-hour, 25-year storm (USEPA, 1992). Additionally, the stockpile area will be inspected on a weekly basis and after storm events. Individual states may require that the stockpile be inspected/certified by a licensed professional engineer. Stockpiled material will be covered with a 6-mil polyvinyl chloride (PVC) liner or sprayed dust control product. The stockpile cover will be secured in place with appropriate material (concrete blocks, weights, etc.) to prevent the movement of the cover.

Decontamination Solutions

Decontamination solutions are generated during the decontamination of personal protective equipment and sampling equipment. Decontamination solutions may range from detergents, organic solvents and acids used to decontaminate small field sampling equipment to steam cleaning rinsate used to wash heavy field equipment. These solutions are to be labeled and stored in closed head drums compatible with the decontamination solution. Decontamination procedures, including personnel and field sampling equipment, must comply with applicable Arcadis procedural documents.

Disposable Equipment

Disposable equipment includes personal protective equipment (e.g., tyvek coveralls, gloves, booties and APR cartridges) and disposable sampling equipment such as trowels or disposable bailers. If the media sampled exhibits hazardous characteristics per results of waste characterization sampling, contaminated disposable equipment will also be disposed of as a hazardous waste. If compatible with the original IDW waste stream (i.e., the IDW is a solid and the disposal equipment is a solid), the disposable equipment can be combined with the IDW. If these materials are not compatible (i.e., the IDW is a liquid and the disposal equipment is a solid), the disposable equipment will be stored onsite in separate labeled 55-gallon drums. Uncontaminated or decontaminated disposable equipment can be considered nonhazardous waste.

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Purge Water

Purge water includes groundwater generated during well development, groundwater sampling, or aquifer testing. The volume of groundwater generated will dictate the appropriate storage procedure. Monitoring well development and groundwater sampling may generate three well volumes of groundwater or more. This volume will be stored in labeled 55-gallon drums. Aquifer tests may generate significantly greater volumes of groundwater depending on the well yield and the duration of the test. Therefore, large-volume portable polyethylene tanks will be considered for temporary storage pending groundwater-waste characterization.

Purged Water Storage Tank Decontamination and Removal

The following procedures will be used for inspection, cleaning, and offsite removal of storage tanks used for temporary storage of purge water. These procedures are intended to be used for rented portable tanks such as Baker Tanks or Rain for Rent containers. Storage tanks will be made of inert plastic materials. The major steps for preparing a rented tank for return to a vendor include characterizing the purge water, disposing of the purge water, decontaminating the tank, final tank inspection, and mobilization. Decontamination and inspection procedures are described in further detail below.

- Tank Cleaning: Most vendors require that tanks be free of any visible sediment and water before returning, a professional cleaning service may be required. Each specific vendor should be consulted concerning specific requirements for returning tanks.
- Tank Inspection: After emptying the tank, purged water storage tanks should be inspected for debris, chemical staining, and physical damage. The vendors require that tanks be returned in the original condition (i.e., free of sediment, staining and no physical damage).

8 WASTE MANAGEMENT

Soil/Solids Characterization

Waste characterization will be conducted in accordance with waste hauler, waste handling facility, and local/state/federal requirements. In general, RCRA hazardous wastes are those solid wastes determined by a Toxicity Characteristic Leaching Procedure (TCLP) test or to contain levels of certain toxic metals, pesticides, or other organic chemicals above specific applicable regulatory agency thresholds. If the one or more of 40 toxic compounds listed in Table I of 40 CFR § 261.24 are detected in the sample at levels above the maximum unregulated concentrations, the waste must be characterized as a toxic hazardous waste. Wastes can also be considered “listed” hazardous waste depending on site-specific processes.

Composite soil samples will be collected at a frequency of one sample per 10 cubic yard basis for stockpiled soil or one per 55-gallon drum for containerized. A four-point composite sample will be collected per 10 cubic yards of stockpiled material and for each drum. Sample and composite frequencies may be adjusted in accordance with the waste handling facility’s requirements. Waste characterization samples may be analyzed for the TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls (PCBs), as well as reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis. Site-specific constituents of concern including pesticides may require additional

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sampling. Please note that state- or local-specific regulations may require a different or additional sampling approaches.

Wastewater Characterization

Waste characterization will be conducted in accordance with the requirements of the waste hauler, waste handling facility, and local/state/federal governments. In general, purge water should be analyzed by methods appropriate for the known contaminants, if any, that have been historically detected in the monitoring wells. Samples will be collected and analyzed in accordance with the requirements of the waste disposal facility. Wastewater characterization samples may be analyzed for TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis. Site-specific constituents of concern including pesticides may require additional sampling. Please note that state- and/or local-specific regulations may require different or additional sampling approaches.

Sample Handling and Shipping

All samples will be appropriately labeled, packed, and shipped, and the chain-of-custody will be filled out in accordance with current Arcadis sample chain of custody, handling, packing, and shipping procedures and guidance instructions.

It should be noted that additional training is required for packaging and shipping of hazardous and/or dangerous materials. Please refer to the current Arcadis training requirements related to handling and shipping of samples, shipping determinations, and hazardous materials.

Preparing Waste Shipment Documentation (Hazardous and Nonhazardous)

Waste profiles will be prepared by the Arcadis CPM and forwarded, along with laboratory analytical data to the Client for approval/signature. The Client will then return the profile to Arcadis who will then forward to the waste removal contractor for preparation of a manifest. The manifest will be reviewed by Arcadis prior to forwarding to the Client for approval. Upon approval of the manifest, the Client will return the original signed manifest directly to the waste contractor or to the Arcadis CPM for forwarding to the waste contractor. Arcadis personnel may sign waste profiles and/or waste manifests on a case by case basis for clients, provided the appropriate agreement is in place between Arcadis and the client documenting that Arcadis is not the generator, but is acting as an authorized representative of the generator.

Final drum labeling and pickup will be supervised by an Arcadis representative who is trained and experienced with applicable waste labeling procedures. The Arcadis representative will have a copy of the drum inventory maintained in the field book and will reconcile the drum inventory with the profile numbers on the labels and on the manifest. Different profile numbers will be generated for different matrices or materials in the drums. For example, the profile number for drill cuttings will be different than the profile number for purge water. When there are multiple profiles it is critical that the proper label, with the profile number appropriate to a specific material be affixed to the proper drums. A copy of the Arcadis drum inventory will be provided to the waste transporter during drum pickup and to the facility receiving the waste.

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9 DATA RECORDING AND MANAGEMENT

Waste characterization sample handling, packing, and shipping procedures will be documented in accordance with relevant Arcadis procedures and guidance instructions as well as applicable client and/or project requirements, such as a Quality Assurance Project Plan or Sampling and Analysis Plan. Copies of the chain-of-custody forms will be maintained in the project file. Arcadis should photograph or maintain a copy of any hazardous waste manifest signed on behalf of Client in the corresponding office DOT record file.

10 QUALITY ASSURANCE

The CPM or APM will review all field documentation once per week for errors or omissions as compared to applicable project requirements including but not limited to: the proposal/scope of work, QAPP, SAP, HASP, etc. Deficiencies will be noted, tracked, and resolved. Upon correction, they will be noted for project documentation.

11 REFERENCES

United States Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes. Office of Remedial and Emergency Response. Hazardous Site Control Division. January 1992.

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ARCADIS Design & Consultancy for natural and built assets

RI-SOP-13

Arcadis ENV Quality Procedure

TOPIC:
FIELD ACTIVITIES DOCUMENTATION

Revision Date: November 2016 Revision Letter: C	QP#: 3.06

STATEMENT OF POLICY:

It is Arcadis Environment Business Line (ENV) policy that field activities must be documented to facilitate the interpretation of data; show compliance with project plans, work plans, and contract terms; and to serve as evidentiary records. Documentation reflecting activities performed must be legible, organized, and complete. Applicable regulatory and client requirements should be considered when documenting field activities. Project-specific requirements for documentation typically should be described in the Work Plan, Field Sampling Plan (FSP), and/or in the Quality Assurance Project Plan (QAPP).

1. Purpose

The purpose of this Quality Procedure (QP) is to provide a standard procedure for the documentation of fieldwork activities. This documentation pertains to site-related projects, but is not limited to the collection of samples, subsurface information, and oversight of construction activities. Field documentation must include, at a minimum, project title and number, date and times of activities, the identification of the employee performing the work, and the specifics of the work being performed.

2. Responsibilities

Certified Project Manager (CPM) – is responsible for the project-related administration of this QP.

Quality Consultant – is responsible for providing quality assurance and quality control guidance to the CPM in implementing this procedure. Note that for federal projects, there are specific requirements and qualifications for the QA Officer assigned to the project.

Project Team Members – who are assigned to document field activities, are responsible for compliance with this procedure.

3. Terms and Definitions

Field Sampling Plan (FSP) – A document that describes the procedures and protocols necessary to complete field sampling and data collection activities.

Work Plan – A document that describes proposed project activities.

Quality Assurance Project Plan (QAPP) – A document that prescribes the quality assurance/quality control (QA/QC) procedures to be followed. Uniform Federal Policy (UFP) QAPPs are now frequently required for environmental projects by most federal regulatory agencies. A UFP QAPP includes Worksheets used to document the entire project plan developed following the systematic planning process. For more details on the UFP QAPP see http://www.epa.gov/fedfac/documents/qualityassurance.htm. Note that if the project QAPP is written following the UFP format, it will also contain a description of the sampling rationale and sampling locations

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as well as QA/QC requirements. The UFP QAPP format is designed to capture the entire systematic planning process. If a UFP QAPP is written for a project, a separate FSP is generally not required unless specified by the particular client or contract.

Standard Operating Procedure (SOP) and Technical Guidance Instruction (TGI) – Documents that describe a procedure and or protocol necessary to conduct a specific activity.

4. Related Forms

Forms used for documenting field activities may be included as attachments to the FSP or the QAPP and may include the following examples:

- Chain-of-custody (COC) form
- Sample data log
- Field modification form
- Sample receipt form
- Corrective action form
- Field activity log
- Calibration log
- Analysis request and chain-of-custody record
- Daily quality control reports
- Purge log
- Soil boring log.

Examples of SOPs and TGIs with field forms and check-lists can be found in the Arcadis Procedure Library at: https://arcadiso365.sharepoint.com/TEAMS/US_envsoplibrary/SitePages/Home.aspx.

DESCRIPTION OF PROCEDURE:

1. General Requirements

1.1 Documentation Format

Documentation of field activities provides an accurate and comprehensive record of the work performed sufficient for a technical peer to reconstruct the day's activities and confirm that necessary client, regulatory, contract, and work plan requirements were met. General requirements include:

- Use of field books (preferably bound) as the primary source for information collection and recording. Field books should be dedicated to the project and appropriately labeled.
- Use of personal digital assistant (PDA) to document select field sampling and data collection activities using Arcadis' electronic data gathering system (EDGE); examples include subsurface data, well/piezometer

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installation, groundwater sampling, and water-level measurements. Use of PDAs with EDGE is optional but encouraged.

- Use of a Field Activity Log is suggested to formally document activities and events as a supplement to bound field books. The Field Activity Log can be a standard or project-specific form or a bound field book. Preprinted standard forms are available for many activities and should be used whenever possible. These forms will provide prompts and request additional information that may be useful and/or needed. Project-specific field forms may be generated or existing forms may be modified to meet specific project needs. Client-supplied forms may be substituted, as required.
- Appropriate header information is documented on the first page of notes for each day of fieldwork, including project title, project number, date, time, author, and relevant setting information such as weather conditions, topography, surface water conditions, observed site activities/uses, and other persons in field team. In addition, include on every page of notes the page number and date. Project-specific information depends on the nature of work being performed and should be discussed by the project team prior to commencing fieldwork. As appropriate, dedicated field logs/journals or forms should be used. When Field Activity Log Forms are used, information fields that are not applicable should be noted as such with the symbol "N/A" or other appropriate notation.
- Field documentation entries shall be made using indelible ink.
- Data entries shall be legible. A single line should be drawn through incorrect entries and the corrected entry written next to the original strikeout. Strikeouts are to be initialed and dated by the originator.
- Units of measurement shall be specified. The level of accuracy shall be indicated (e.g., observed estimate, quantified census from direct count, and electronic data collection).
- Field records are to be maintained in project files unless otherwise specified by a client or stipulated by a contract.
- Unless addressed specifically by a client or stipulated by a contract, site photographs should be taken to document the general setting and landscape as well as site-specific issues/resources of interest. Photo locations and the compass direction of view should be recorded in the notes with the photo number.

1.2 Documentation Entries

A chronology of field events should be recorded. General entry requirements include:

- Visitors to the site, including owner and regulatory agency representatives
- Summary of pertinent project communications with the client, regulators, or other site visitors during the fieldwork
- Other contractors or entities working on site
- A description of the day's field activities, generally in chronological sequence or in order of significance, using military time notation (e.g., 9:00 a.m. as 0900, and 5:00 p.m. as 1700)
- If applicable, calibration of measuring and test equipment and identification of the calibration standard(s) (use a Calibration Log, if available, with cross-reference entered into the field book)
- Field equipment identification, including information such as the type, manufacturer, model number, or other specific information

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- If applicable sampling activities are being performed, weather information such as temperature, wind speed and direction, precipitation, time of measurement, and units
- Documentation of safety meeting (e.g., tailgates and tailboards) topics and attendees
- Verification of subsurface utility clearance in accordance with ENV ivision policy
- Safety and/or monitoring equipment readings, including time of measurements and units
- If applicable, specific forms used for collection of data are referenced in the field notebook
- Subcontractor progress and/or problems encountered
- Changes in the scope of work
- Other unusual events.

2. Specific Requirements

2.1 Sample Collection

Sample collection data are documented in a bound field book, PDA, and/or on a Field Activity Log. Where both are being used, information contained in one is cross-referenced to the other. Entries such as the following examples should be consistent with the requirements in the project-specific Work Plan, FSP, and QAPP:

- Sample identification number, location taken, depth interval, sample media, sample preservative, collection time, and date
- Sample collection method and protocol
- Physical description of the sample (using a standard classification system for soil)
- If a composite sample, include the number, location(s), and depth(s) of grab samples incorporated in the composite
- Quality-related samples (e.g., field duplicates, trip blanks, equipment rinse, blanks matrix spikes, and matrix spike duplicates)
- Container description and sample volume
- Pertinent technical data, such as pH, conductivity, temperature, and head-space readings
- Pertinent technical comments
- Identification of personnel collecting the sample.

2.2 Sample Labeling

Sample labels must be prepared and attached to sample containers. Labels are either provided by the laboratory performing the analyses or are generated internally. Labels should be indelible and securely attached to the container. The information to be provided may include:

- Sample identification number
- Sample date, initials or name of who collected the sample, and collection time
- Physical description of the sample (e.g., water, solid, gas, or other physical medium)
- Analytical parameters and method(s)

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- Preservatives, if present
- Sample location and depth, if applicable
- Client.

Although this information is typically written out, it can also be recorded in an electronic tracking system if a bar code is used.

2.3 Analysis Request and Chain-of-Custody Record

A critical component of data collection is the documentation that the samples were obtained from specific locations and received by the laboratory or archive without alteration. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal or archive must be properly documented. Documentation will be accomplished through a COC record that documents each sample and identifies the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if at least one of the following criteria is met:

- The sample is in a person's actual possession
- The sample is in unobstructed view, after being in the person's actual possession
- The sample is locked and only accessible by the custodian after having been in the person's actual possession
- The sample is in a secured area, restricted to authorized personnel (e.g., laboratory).

An example COC form to be used by ENV personnel in collecting and shipping samples can be found on the corporate Intranet. A laboratory typically will not accept samples for analysis without a correctly prepared COC form. The COC must be signed by each individual who has the sample in his/her custody. Each sample shipped to a laboratory for analyses must be documented on the COC. Information on this form correlates with other supporting documentation, including the field log book, sample labels, and sample collection logs.

The COC documents the elapsed time and the custodians of the sample from the time of its collection. The individuals who have physically handled the sample(s) or witnessed initial sample collection and packaging (sample team member) must be identified on the form. A sample team member relinquishes the sample by signing the COC. Individuals who either relinquish or receive samples must include their complete names, company affiliation, and the date and time the sample(s) were relinquished. The times that the samples are relinquished and received by the next custodian should coincide, with the exception of transfer by commercial carriers. These carriers will not be required to sign the COC.

If a sample is to be stored for a period of time (e.g., overnight), measures are taken to secure the sample container in a manner that only provides access to the custodian of record. If samples are relinquished to a commercial carrier (i.e., UPS or Federal Express), the carrier waybill number is recorded, and a copy of the waybill is attached to the COC. These documents are maintained with other field documentation. The original COC is sealed inside a zip-top plastic bag and placed inside the shipping container with the samples.

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If corrections are made to the COC, the corrections should be made (single line through the error, initial, and date) by the originator of the change, and, if necessary, an explanation of the change should be provided. The documentation should be of a level of detail that clearly documents the change to a third-party reviewer.

Guidance for choosing a laboratory and completing analyses requests and COC can be found in QP 2.09-Subcontracting Laboratory Services and on the corporate Intranet for the Arcadis Laboratory Program (ALP) and should also be described in the project-specific planning documents (i.e., Work Plan, FSP. or QAPP).

2.4 Subsurface Logs

Test pits, soil borings, monitoring wells or rock coreholes wells, and piezometer installations are to be recorded in bound field books or PDA and may be supplemented with prepared forms. Personnel completing the log are to supply the following information:

- Administrative and technical information included in the header.
- Types of equipment used (e.g., drill rig type, drilling tools used [including diameter and length], or backhoe model).
- Subcontractor/driller used.
- Descriptions of subsurface materials encountered and the number and type of samples collected, if any.
- Subsurface exploration depth and units of measure.
- For drilling, length of recovery.
- Sample type and sample number for geotechnical or analytical samples collected. These data are to be also entered on the sample collection log (if used) and the sample label.
- Classification standard protocol used, if any (e.g., ASTM International Standard Penetration Test).
- Narrative description of the soil, sediment, or bedrock (using standard classification system) and other pertinent information.
- Additional data, such as background and sample vapor/gas readings, observation of sheens, non-aqueous-phase liquid, depth to water (if encountered), presence of (but generally not description of) odors, changes in drilling conditions, and other pertinent information.
- Description of the materials used to seal the boring, unless it is completed as a well or piezometer.

2.5 Monitoring Well/Piezometer Installation

In addition to requirements in Section 2.4, subsequent well or piezometer development activities may involve transcription of field data from the field book onto a computer-based boring log. The field notebook or PDA is to be used to identify the chronology and major events of the installation activity, and the computer-based boring log is to be used to correlate the geologic strata to the major elements of the monitoring well construction. Information to be collected and recorded must meet the regulatory and client requirements and may include the following:

- Location identity
- Screen and riser type, length, diameter, and location

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- Diameter
- Total depth
- Sump location and depth and diameter
- Materials of construction (e.g., stainless steel, polyvinyl chloride, or other material)
- Seal type(s) and or depth(s)
- Sand or gravel pack type, including materials (e.g., silica) and gradation
- Depth to water before and after installation.

2.6 Air Sampling Logs

At a minimum, air sampling documentation should include:

- Start and finish time of sampling
- Sampling location
- Sampling method/media
- Volume sampled.

2.7 Construction, Demolition, Abandonment, and Related Activities

Monitoring and documentation of construction and comparable activities shall be documented in bound field books and/or on appropriate company forms and should include similar information as specified above, including information such as:

- Project name and number
- Owner or client name
- Contractor or subcontractors performing the work
- Contractor or subcontractor superintendent(s) and personnel (as available) on site
- Chronological sequence and description of work activities performed, including workday start and completion times
- Reference to contract sections, work plans, or specifications describing work being performed
- Reference to relevant permit conditions and regulatory requirements and/or reference to regulatory guidance documents controlling work approach
- Listing of all trades performing work by contractor and subcontractor
- Hours worked per trade
- Work hours per day per shift, if applicable
- Equipment on site (e.g., description, model number, size, and type) and hours of use
- Listing of equipment on site being left idle
- Description and quantity of materials used or incorporated, with reference to contract or specification item number, if feasible; include simple sketch of excavation with approximate dimension, if applicable
- Calculations with dimensions for quantities of material used or incorporated

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- Delineation of the work area and access routes (e.g., fencing, flagging, or staking), confirmation that activities occurred within the work area or description of work occurring outside the delineated work area and justification (as needed), and characterization of impacts outside the designated work area
- Documentation of compliance with speed limits, dust control, erosion control best management practices, and other basic elements of construction activities as dictated by project work plans and applicable permits and regulatory criteria.

2.8 Daily Safety Meeting

A Daily Safety Meeting is to be conducted and documented each workday prior to the initiation of field activities, with on-site ENV personnel, contractors, subcontractors, and visitors if possible. Safety topics discussed are entered on the Daily Safety Meeting Form (available on the corporate Intranet). Topics discussed should include site-specific conditions, procedures to be followed that day, and protective equipment. A printed listing of the attendees at the meeting and their signatures should be included. Other required data are:

- Identification of the individual conducting the meeting and his/her signature
- Identification of the project supervisor and project manager.

2.9 Calibration

Documentation of the calibration and calibration results shall be made for field equipment requiring calibration measuring and test equipment calibration data are recorded in the field book or on the Field Activity Log. Calibration data include the following:

- Unique identification of instrument being calibrated, including type, model, and serial number
- Date and time of calibration
- Standards used in the calibration, including standard identity, concentration, lot number, and manufacturer of the standard
- Instrument reading with respect to each calibration standard
- Comments, as necessary, regarding instrument performance.

2.10 Photographs and Videos

When the client allows, photographs and videos may be used to help document pre-, active, and post-field activities. In sensitive areas (e.g., secured or confidential), the client must be contacted to evaluate security procedures concerning use of photographs or videos. Photographic and video documentation should include project title, project number, date, time, and description of conditions. The time should also be documented if time is important to a sequence of photographs.

Photographs are documented by numbering digital photographs and identifying the number and subject on the Field Activity Log. Individual prints may be marked with a stamp or preprinted self-adhesive labels, or by writing

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the project number and sequential number of each photograph and referencing the numbers in the field book, the Field Activity Log, or a dedicated photo log. Videos used for field documentation are to be identified by project title, project number, and description.

2.11 Subcontractor Preparedness Checklist

Prior to starting work, a review is to be made and documented of a subcontractor's preparedness to perform specified activities. This review may be documented on the Field Activities Log or on checklists that may be developed according to requirements for subcontracted work activities. Particular emphasis should be on site-specific issues that may require special consideration such as health and safety, access, and unique settings. These should be discussed in advance with the CPM and the client in developing and implementing the Scope of Work.

— END OF PROCEDURE —

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Implementation Date	Arcadis HS Standard Name	ARCADIS Design & Consultancy for natural and built assets
13 December 2006	Utility Location and Clearance	
Revision Date	Arcadis HS Standard No.	Revision Number
17 March 2017	ARCHSFS019	16

RI-SOP-14

EXECUTIVE SUMMARY

Damaging an underground or aboveground utility can result in serious injury and loss of life, disrupt essential services, and create significant liability to Arcadis, clients, and subcontractors. Therefore, it is Arcadis policy that the following steps be completed prior to beginning any subsurface intrusive work (i.e., any work or activity that breaks the plane of the ground surface):

- The presence of existing or known utilities will be investigated and cleared (to the extent feasible) by locating and marking before the start of any subsurface intrusive work and where appropriate, visually verifying through soft dig methods (referred to as potholing or daylighting) before the start of any subsurface intrusive activity.
- A minimum of **three (3) reliable lines** of evidence are required for an acceptable utility clearance. Each location of subsurface intrusive work must have at least 3 reliable lines of evidence. All lines of evidence used during the utility clearance procedure will be recorded on the Utility and Structures Checklist or equivalent client-provided checklist or permit. If a line of evidence is lost or not apparent, STOP WORK, and re-establish the line of evidence prior to resuming subsurface intrusive work.
- The lines of evidence used will be reasonable and appropriate for the conditions expected to be encountered (soil type, water table, etc.) and the type of utilities expected to be encountered (e.g., gas line versus an irrigation line).
- Contact the State One Call or equivalent service (Nationwide "811") as required by law. The State One Call or equivalent service (Nationwide "811") can only be used as a reliable line of evidence when working within the public right-of-way or easement.
- For point clearance (single intrusive point, used as 1 of the 3 required reliable lines of evidence), the borehole must be cleared to 110% of the diameter of the intrusive device (e.g., auger, drill head, etc.) or an additional 2 inches of overall diameter, whichever is greater.
- Utility clearance information will be documented on the Arcadis Utility and Structures Checklist (USC) or equivalent client-provided checklist or permit. The Utility Structures and Checklist is valid for 15 business days from the date of completion. A copy of the completed Utility and Structures Checklist will remain on-site during all subsurface intrusive work.
- Employees overseeing utility clearance activities will:
  - Be familiar with the contents of this standard and ARC HSFS-019 Supplement 2;
  - Have one year of field experience in the visual identification of utilities; and
  - If operating equipment, have training and six months of experience in the proper operation and results interpretation of any clearance equipment, including without limitation, magnetometers and ground penetrating radar.
- A utility strike is an unplanned contact of a utility during the course of work that results in damage requiring repairs, making a report to the utility owner, or requiring further assessment to evaluate the potential for damage. All utility strikes must be reported within 24 hours using the Utility Line Strike Investigation Form. **Do not enter the incident into 4-Sight until approved to do so by Corporate Legal. Refer to** ARC HSFS-019 Supplement 5**, Utility Strike Emergency Action Plan Guidelines.**

Report Utility Incident Now

1. POLICY

It is the practice of Arcadis and its affiliated companies to implement appropriate, reasonable, and practical standards within acceptable and customary industry practices to promote the health and safety of its employees and avoid and mitigate exposure of risk in the performance of their work. In furtherance of this policy, Arcadis promotes and encourages compliance by all employees with this policy and standards relating to work in the vicinity of subsurface, submerged, or aboveground utilities.

2. PURPOSE AND SCOPE

2.1 Purpose

This standard directs general safety standards and best practices associated with the identification and management of subsurface, submerged, and aboveground utilities on project sites. Utility location standard operating procedures (SOP) for submerged utilities can found in ARC HSFS-019 Supplement 6.

2.2 Scope

This standard assigns responsibilities and expectations for proper utility clearance by both Arcadis employees and Arcadis subcontractors at project sites.

3. DEFINITIONS

Definitions relating to Utility Clearance can be found in Exhibit 1.

4. RESPONSIBILITIES

4.1 Project Manager Responsibilities

For every project site having the potential to come into contact with utilities, Project Managers must ensure that:

- The requirements of this standard are followed.
- Local regulations governing utility clearance are followed. This includes ensuring local and/or state laws defining activities or depth of intrusive work/excavation requiring utility clearance are reviewed as they vary by location. For further information, refer to One Call and State Law Directory.
- Efforts are made to work with the client, project site representatives, public utility companies, and subcontractors to identify the nature of any utilities and to determine control processes that need to be implemented by Arcadis and the subcontractors to prevent damage to these utilities and to properly manage the effects in the event there is utility damage.
- Utility clearance activities are only delegated to a Task Manager or other individual meeting the requirements of Section 4.2 below, as appropriate. However, even if the Project Manager delegates certain responsibilities, the Project Manager maintains primary responsibility for a complete utility clearance.

For additional information on Project Manager responsibilities and best practices, refer to ARC HSFS-019 Supplement 1.

- Project Managers or designee must review the Utility and Structures Checklist with staff and Arcadis subcontractors conducting subsurface intrusive work (including "Sub-of-Subs" when conducting subsurface intrusive work) prior to staff beginning subsurface intrusive work. The Project Manager or designee review must be documented on the Utility and Structures Checklist prior to starting subsurface intrusive work.

4.2 Field Personnel Responsibilities

Arcadis field personnel conducting work on a project site having the potential to come into contact with utilities have the responsibility to:

- Read, understand, and follow this standard and ARC HSFS-019 Supplement 2 and complete the appropriate checklists during the on-site utility and structures locate and clearance process.

- Complete a minimum of one year of utility clearance-related experience before accepting responsibility for any utility clearance tasks. This requires on-site training led by another Arcadis employee with detailed knowledge and experience in identifying utilities and structures.

- Complete training and have 6 months of experience in operating and interpreting the results of remote sensing technologies, including without limitation, magnetometers and ground penetrating radar, before operating such technologies. Field staff should understand the technologies being utilized by a private utility locate contractor and how they are operating in comparison with the site conditions. Refer to ARC HSFS-019 Supplement 3 for more information.

- Prior to beginning subsurface intrusive work, the Utility and Structures Checklist must be completed and signed by the staff member completing or overseeing the clearance. Confirm that the Utility and Structures Checklist was reviewed by the Project Manager or designee as discussed in Section 4.1 above. Review the Utility and Structures Checklist daily prior to starting subsurface intrusive activities to ensure all utilities are identified and markings are present. A copy of the completed Utility and Structures Checklist will remain on-site during all subsurface intrusive work (i.e., any work or activity that breaks the plan of the ground surface).

- Use their STOP WORK Authority to eliminate any reasonable concern if utilities cannot be reasonably located and contact the Project Manager to review the STOP WORK situation and confirm the direction of action before moving forward.

- Ensure that Arcadis subcontractors conduct their own reasonable independent utility clearance efforts as required by Arcadis' standard subcontract and are aware of any Arcadis clearance standards used on-site.

- Be on-site and provide oversight during utility locate activities and any active subsurface intrusive work or activities involving contractor under contract to Arcadis.

- If a utility is damaged and repaired during the course of the field event, field staff must provide oversight and document that the repair appears competent and complete to prevent further damage to the site when the damaged utility is re-activated.

4.3 Arcadis Subcontractor Responsibilities

According to Arcadis' standard subcontract, subcontractors have agreed to take responsibility for any damages resulting from a utility impact caused by their work. Therefore, Arcadis subcontractors are expected to take reasonable time and diligence to conduct their own independent utility clearance using reasonable standards and processes. Subcontractors have the responsibility to stop their work if utility concerns are identified and will report those concerns to the Arcadis employee overseeing their work activities. Arcadis staff should reinforce these responsibilities with subcontractors during job safety briefings.

In jurisdictions where the actual contractor performing the subsurface intrusive work is required to perform utility clearance notifications, the contractor will perform the clearance notification and will provide evidence of the notification to Arcadis (ticket or ticket number, etc.). Refer to ARC HSFS-019 Supplement 4 for Best Practices for State One Call procedures.

- If overhead utilities are present in areas where heavy equipment will be operated, ensure adequate clearance is provided. For heavy equipment that is extendable or telescoping (e.g., excavators, dump trucks, extendable lift trucks), evaluate whether the use of a spotter is necessary prior to operating heavy equipment when in proximity to the overhead utility.

- If a utility is damaged and repaired during the course of the field event, the field subcontractor must verify that the repair is competent and complete to prevent further damage to the site when the damaged utility is re-activated.

5. PROCEDURE

5.1 General

Protocols to be followed during utility and structures location and clearance activities are outlined in:

- Best Practices for Project Managers (or Their Delegates) Concerning Utility Clearance (ARC HSFS-019 Supplement 1).

- Best Practices for Field Personnel Concerning Utility Clearance (ARC HSFS-019 Supplement 2).

- Use and Limitations of Common Underground Locating Technologies and Clearance Methods (ARC HSFS-019 Supplement 3).

- Best Practices for State One Call Procedures (ARC HSFS-019 Supplement 4).
- Emergency Action Plan guidelines for Utility Strikes (HSFS-019 Supplement 5).
- Utility Location Standard Operating Procedure for Aquatic Work Activities (ARC HSFS-019 Supplement 6).

5.2 Lines of Evidence

When locating underground utilities, three (3) reliable "lines of evidence" must be established to help determine where a subsurface utility may be located. A line of evidence may be a site drawing that shows where a utility is located, it could be anecdotal information obtained from owners or employees, it could be established using any number of non-intrusive geophysical methods [e.g., ground penetrating radar (GPR), electromagnetic survey (EM), radio-frequency methods (RF), etc.], or it could involve probing for or exposing the utility by soft dig technologies (i.e., daylighting or potholing). Some lines of evidence will identify utility locations with a high degree of certainty (e.g., direct connect radio-frequency technique, daylighting or potholing, sonde tracing, etc.). Other lines of evidence will identify utilities will less certainty (e.g., anecdotal reports, design drawings, etc.).

Effective utility locate practices must use multiple lines of evidence until there is a high degree of certainty that the underground services have been adequately located. Three (3) reliable lines of evidence are required for an appropriate utility clearance as defined in this standard. All reliable lines of evidence used during the utility clearance procedure will be recorded on the Utility and Structures Checklist or equivalent client-provided checklist or permit. If three (3) reliable lines of evidence have not established certainty in the location of a utility, STOP WORK and do not proceed. Additional reliable lines of evidence must be utilized until the presence or absence of the underground utility can be established. During work activities, if a line of evidence is lost or not apparent (e.g., paint markings have faded), STOP WORK, and re-establish the line of evidence prior to resuming subsurface intrusive work.

Generally, the following lines of evidence may be used to meet this minimum utility clearance requirement:

1. Contacting the State One Call or equivalent service (Nationwide "811") is REQUIRED BY LAW regardless if it will be used as a line of evidence. Contacting the State One Call or equivalent service (Nationwide "811") is an acceptable reliable line of evidence when working within the public right of way or easement. Note that the State One Call can provide valuable information regarding locations and types of utilities entering the private property.

   Note: For work on private property or in areas not served by State One Call or equivalent service, consider using a reputable private utility locating company to locate and mark the utilities. **Use of a reputable private utility locator is encouraged for all projects with subsurface or submerged utilities.** When working with a private locater, it is best practice to pre-plan clearance areas, review required clearance equipment and the re-clearing/confirmation of any public utility mark outs (State One Call or equivalent service Nationwide "811").

2. Use detailed, scaled site utility plans, preferably in the form of an “as-built” or “record” drawing, to identify and/or confirm utility locations. Document request and/or receipt of utility drawings from the property owner/client on the Utilities and Structures Checklist.

3. Interview(s) with knowledgeable site or client personnel. The following questions should be asked during the interview and answers documented on the Utility and Structures Checklist:

   o Employees(s) Name and Affiliation(s) with the site;

   o Types of utilities, including utility composition and location of utilities on-site;

   o Depths of known utilities; and

   o Any other pertinent information regarding utilities on the site.

View the Utilities and Structures Checklist

4. Conduct a detailed visual site inspection of areas around all planned subsurface intrusive work points or areas to identify and/or confirm utility locations. For underground utilities, conduct an inspection for structures that tend to indicate the presence and general location of such utilities, including, but not limited to manholes, vaults, valve covers, valve markers, telephone pedestals, transformer housings, fire hydrants, spigots, sprinkler heads, air relief valves, backflow preventers, meters, downspouts going into the subsurface, power poles with wiring going into the subsurface and line markers. Saw cut lines and concrete/asphalt repairs often yield valuable information regarding utility locations.

   Always discuss the presence of utilities with the site owner, operator, and/or occupant to identify any potential utilities that might not be readily identified by non-intrusive clearing methods or may be:

   o At depths > 5 feet below ground surface; or

   o At very shallow depths (< 2 feet below ground surface), such as communication lines, electrical conduits/wiring, irrigation lines, etc.

If one of the above lines of evidence cannot be utilized or if using the above lines of evidence does not adequately identify utilities with reasonable certainty, one or more additional lines of evidence must be utilized. Commonly used lines of evidence are listed on the Utility and Structures Checklist.

A discussion of use and limitations associated with common utility location and clearance methods is provided in ARC HSFS-019 Supplement 3.

Standard operating procedures for utility location in submerged settings are presented in ARC HSFS-019 Supplement 6.

The lines of evidence will be recorded on the Utility and Structures Checklist or equivalent client-provided checklist or permit.

Note: If a line of evidence is lost, utility markings are removed/worn, or area of previous clearance is not confirmed, STOP WORK and re-establish the

line(s) of evidence prior to resuming subsurface intrusive work. Each location of subsurface intrusive work must have 3 reliable lines of evidence. All lines of evidence used during the utility clearance procedure will be recorded on the Utility and Structures Checklist of equivalent client-provided checklist or permit. If a line of evidence is lost or not apparent, STOP WORK, and re-establish the line of evidence prior to resuming subsurface intrusive work. The Utility Structures and Checklist is valid for 15 business days from the date of completion.

If and when any line of evidence reveals that planned subsurface work will be located inside the 30-inch Tolerance Zone of known/marked/located/observed utilities, the project team must Stop Work and contact Corporate H&S as early as possible for pre-approval.

5.3 Color Codes used for Utility Markings

The following colors are used for marking utilities. Some government agencies or large industrial facilities may use additional colors not provided below. Arcadis policy is to assume any paint marking or pin flag color not provided below is a subsurface utility marking until proven otherwise.

If utilities or subsurface anomalies are identified but the utility type or anomalies are not classified, it is recommend that a pink (Temporary Survey Marking) marking be used. Once the type of utility is established, the pink marks should be repainted/remarked to represent the correct type of utility.

COLOR	Utility Line
WHITE	Proposed Excavation
PINK	Temporary Survey Markings
RED	Electrical Power Lines, Cables, Conduit and Lighting Cables
YELLOW	Gas, Oil, Steam, Petroleum or Gaseous Materials
ORANGE	Communication, Alarm or Signal Lines, Cables or Conduit
BLUE	Potable Water
PURPLE	Reclaimed Water, Irrigation and Slurry Lines
GREEN	Sewer and Drain Lines

APWA and ANSI standard Z-53.1

5.4 Locating Technologies

There are several types of locating technologies that can be used to identify and locate utilities in the subsurface. Project teams need to work closely with private utility locators (PUL) in order to best match locating technology with site conditions. To provide the best results, all possible locating technologies should be available for use and implementation at the project location. Any potential interferences should also be discussed up front and then at the project site during utility location activities. Potential interferences could be soil moisture, soil type, standing water on concrete/asphalt, rebar, fencing, and metal

structures that are in the subsurface. Employees overseeing locating technology activities should have an understanding of device operation and limitations. For further information, refer to ARC HSFS-019 Supplement 3, Use and Limitations of Common Utility Location Technologies and Clearance Methods.

5.5 Clearance Methods

In some cases, proposed subsurface intrusive locations may be pre-cleared using other intrusive methods. Determine the clearance or soft dig method based on-site conditions and utilize the least invasive method possible. The number of subsurface intrusive locations and soil type should be taken into consideration. The following clearance methods are listed from least invasive to most:

1. Vacuum Extraction/Potholing (air or water-based),
2. Air knifing,
3. Hydroknifing,
4. Probing,
5. Hand augering,
6. Hand digging, and
7. Posthole digging.

Single-Point clearance must be 110% of the proposed subsurface intrusive area or the diameter plus 2 inches, whichever is greater. Three-Point clearance must be installed in a triangular pattern around the proposed borehole and in a configuration not to allow for utilities to enter the borehole. Three-Point clearance must be 110% of the proposed intrusive area or the diameter of the intrusive area plus 2 inches, whichever is greater. Each method of clearance should be documented on the Utility and Structure Checklist.

Manual clearing methods, such as shoveling, using pick axes, digging bars and other hand tools, should be avoided completely or only used when absolutely necessary and used with caution. Excessive downward force, prying or use in poor/obstructed visibility conditions is prohibited as these tools can damage utilities.

Surface cover (e.g., asphalt) removal methods within the 30-inch Tolerance Zone that pose excessive downward force, such as jackhammering, should be used with extreme caution. Methods that only cut the surface cover (coring or saw cutting) present less risk due to the absence of the downward force, which could cause collateral damage to shallow subsurface utilities. Note that utilities are often present at the concrete or pavement/soil interface or encased within the concrete or pavement and are easily damaged during concrete coring or pavement removal. Always work slowly, methodically and frequently STOP WORK to evaluate conditions during these work activities.

For borings and excavations, if the utility is known to be at depths where hand clearing is not feasible or creates additional safety concerns, no work will be performed within the 30-inch Tolerance Zone vertically or horizontally of the utility unless manual clearing is performed under the oversight of an Excavation Competent Person as defined in ARC HSCS005 Arcadis Excavation and Trenching.

5.5.1 Temporary Backfilling of Pre-Cleared Boreholes

In some cases, it may be necessary to temporarily backfill a pre-cleared location until the remaining subsurface activities are performed. At these locations where subsurface intrusive work does not immediately follow pre-clearance, it is important to properly backfill and mark the pre-cleared location in order to relocate the pre-cleared location. In general, wooden stakes, survey markers, whiskers, paint marking or other surficial markings alone are inadequate because these markings can be easily removed, damaged or otherwise lost leading to uncertainty regarding the pre-cleared location. Although the specific steps for backfilling a pre-cleared location will depend on site-specific conditions, use the following additional steps to prevent loss of the pre-cleared location:

- Backfill a pre-cleared location with clean sand or other granular material that is significantly different than the surrounding subsurface native material. Native soil should not be used to backfill a pre-cleared location that may require further subsurface work.
- Backfill the top 2 feet of a pre-cleared location with dyed sand or gravel to facilitate re-location.
- Use hammered wooden stakes or delineators to mark locations as an additional measure, if practical.
- In the event that the pre-cleared borehole is located on asphalt or concrete and an asphalt cold patch is required, use white paint to mark the intrusive location with a circle over the asphalt cold patch.
- In some instances, such as projects potentially affected by unexploded ordinance (UXO), the pre-cleared borehole may require that a PVC of matching diameter pipe be inserted into the pre-cleared borehole, filled with clean sand and affixed with a matching cap. Contact the project manager to identify any client-specific requirements.
- Always use a physical subsurface marker such as described above to identify the pre-cleared borehole location. Never rely solely on field measurements or GPS coordinates.
- If a utility or anomaly/obstruction is encountered during the pre-clearing process, backfill the hole with the native soil and mark the location with a pink-painted X and/or NO.

In the event that a previously pre-cleared location cannot be located, the location must be re-cleared prior to performing subsurface intrusive work.

5.6 Clearance for Working in Vicinity of Subsurface Utilities

Prior to the start of subsurface intrusive activities (i.e., excavations, vertical drilling, installing grounding rod, and soil sampling), all utilities must be located and measures must be instituted to avoid subsurface utility hazards. See exemptions for subsurface intrusive work in Exhibit 1 (Definitions). Do not conduct subsurface work within 30 inches of a line marking in all directions. If the centerline of the utility is marked, the diameter of

the utility or utility bank (Exhibit 1) must be incorporated into the 30-inch Tolerance Zone, see Figure 1 located in Exhibit 2 for further instructions.

If and when any line of evidence reveals that planned subsurface work will be located inside the 30-inch Tolerance Zone of known/marked/located/observed utilities, the project team must Stop Work and contact Corporate H&S as early as possible for pre-approval.

If subsurface work must take place within the 30-inch Tolerance Zone of the line marking, the utility must be exposed (potholed) by soft dig/clearance methods prior to starting subsurface intrusive activities (see Section 5.5 for options); **no mechanized equipment is permitted for the exposing of the utility**.

Once the utility has been exposed, if mechanized equipment is planned for use within the 30-inch Tolerance Zone of the utility, such activity must receive pre-approval by Corporate H&S, as necessary, to mitigate or accept the risk associated with the planned work. Additional excavation safety procedures may have to be developed as part of the approval to proceed. It should be noted that any disturbance within the 30 inches or disruption of the bedding materials could affect the integrity of the utility.

For horizontal borings, to avoid striking a utility, damage from vibration, damage by pressure of the advancing boring, do not drill within 30 inches in all directions (3-Dimensional cylinder) of a line marking. Make sure to factor the diameter of the line or utility bank when computing 30-inch Tolerance Zone. When crossing a utility during horizontal drilling, it is recommend that the utility be exposed 30 inches in a 360°-direction. When exposing utilities for horizontal borings, the utility must be exposed (potholed) by soft dig/clearance methods. This recommendation applies even if the operating contractor has technology that places the location to within a few inches. Make sure to factor the diameter of the utility when determining the 30-inch Tolerance Zone. If subsurface work must take place within the 30-inch Tolerance Zone of the line marking, the utility must be exposed (potholed) by soft dig/clearance methods prior to starting subsurface intrusive work (see Section 5.5 for options); **no mechanized equipment is permitted for the exposing of the utility**. Once the utility has been exposed, if mechanized equipment is planned for use within the 30-inch Tolerance Zone of the utility, such activity must receive pre-approval by Corporate H&S, as necessary, to mitigate or accept the risk associated with the planned work. Additional excavation safety procedures may have to be developed as part of the approval to proceed. It should be noted that any disturbance within the 30 inches or disruption of the bedding materials could affect the integrity of the utility.

Additional cautions for horizontal borings include gravity utilities, such as sewers and storm drains, as the depth of these utilities will change (sometimes significantly) as they run across the project site. Always obtain the utility depth at the location where the boring will actually cross the line by collecting sewer depth inverts from identified manholes and interpolating those depths to the area of the subsurface intrusive work.

During well installations and well abandonment via mechanical equipment, the 30-inch Tolerance Zone rule applies outward from the outside edge of the largest diameter auger or tool to be used for installation and abandonment (over drilling). In cases where wells have been previously installed and the 30-inch rule has not been followed, work proposed using mechanized equipment to work within the 30-inch Tolerance Zone will

require approval from Corporate H&S. For more information, see Exhibit 2 for further instructions.

5.6.1 Aboveground Activities causing Subsurface Disturbance in the Vicinity of Underground Utilities

Aboveground activities can cause damage to shallow underground utilities or structures. Plan the intended path/mobilization/operation of Heavy Equipment is cleared to ensure that shallow utilities are not damaged. If Heavy Equipment must cross over shallow utilities, the utilities will be protected. Other subsurface disturbances may lead to damage such as clearing trees/shrubs/vegetation as roots may be entangled with underground piping or structures. For more information, see Best Practices for Field Personnel Concerning Utility Clearance (ARC HSFS-019 Supplement 2).

5.7 Acceptable Clearance for Working in Vicinity of Overhead Power Lines and Other Overhead Lines and Structures

No work will be performed by Arcadis or our subcontractor near overhead power lines where any Unqualified Person or equipment is within the limits specified below unless the power line has been properly covered or de-energized by the owner or operator of the power line. Qualified Person approach distances are defined in Exhibit 5A and 5B of ARC HSFS0006 Electrical Safety Standard.

Power Line Voltage Phase to phase (kV)	Minimum Safe Clearance (feet)
50 or below	10
Above 50 to 200	15
Above 200 to 350	20
Above 350 to 500	25
Above 500 to 750	35
Above 750 to 1,000	45

ANSI standard B30.5-1994, 5-3.4.5

5.7.1 Reducing Vehicle and Mechanical Equipment Clearance Requirements

Any vehicle or mechanical equipment capable of having parts of its structure elevated near energized overhead lines shall be operated so that a clearance of 10 feet (305 centimeters (cm)) is maintained. If the voltage is higher than 50 kilovolts (kV), the clearance shall be increased 4 inches (10 cm) for every 10 kV over that voltage. However, under any of the following conditions, the clearance may be reduced:

- If the vehicle is in transit with its structure lowered, the clearance may be reduced to 4 feet (122 cm). If the voltage is higher than 50 kV, the clearance shall be increased 4 inches (10 cm) for every 10 kV over that voltage.

- If insulating barriers are installed to prevent contact with the lines and if the barriers are rated for the voltage of the line being guarded and are not a part of or an attachment to the vehicle or its raised structure, the clearance may be reduced to a distance within the designed working dimensions of the insulating barrier.

- If the equipment is an aerial lift that is insulated for the voltage involved and if the work is performed by a qualified person, the clearance (between the uninsulated portion of the aerial lift and the power line) may be reduced to the distance given in OSHA 1910.333(c)(3)(ii)(C) Table S-5. Reference information from OSHA 1910.333 Table S-5 and NFPA 70E Table 130.4(C)(a) for alternating-current systems and 130.4(C)(b) for the distances associated with direct-current voltage systems is included as Exhibit 5 of ARC HSFS0006 Electrical Safety Standard.

Employees standing on the ground may not contact the vehicle or mechanical equipment or any of its attachments unless:

- The employee is using protective equipment rated for the voltage; or

- The equipment is located so that no uninsulated part of its structure (that portion of the structure that provides a conductive path to employees on the ground) can come closer to the line than permitted in this section of this standard.

If any vehicle or mechanical equipment capable of having parts of its structure elevated near energized overhead lines is intentionally grounded, employees working on the ground near the point of grounding may not stand at the grounding location whenever there is a possibility of overhead line contact. Additional precautions, such as the use of barricades or insulation, shall be taken to protect employees from hazardous ground potentials, depending on earth resistivity and fault currents, which can develop within the first few feet or more outward from the grounding point.

When a machine is in contact with an overhead power line, do not allow anyone to come near or touch the machine. Stay away from the machine and summon outside assistance.

5.7.2 Acceptable Clearance for Working in Vicinity of Non-Electrical Overhead Utilities and Structures

Arcadis field personnel will identify non-electrical overhead utilities and structures and where possible, work is not be conducted within the 30-inch Tolerance Zone of these overhead utilities and structures. It is recommended that if work will be completed in the vicinity of non-electric overhead utilities, the overhead utilities should be labeled with warning signs, protective barricades, and/or flags. Non-electrical overhead utilities and structures may include, but is not limited to, pipe chases, water lines, ceilings in buildings, etc. Arcadis field personnel will notify its site workers

(employees, subcontractors, vendors, etc.) of known overhead utilities and structures during the tailgate safety meeting. See Exhibit 2 for additional details.

5.8 Reporting Utility Incidents

Arcadis field personnel involved with any subsurface, submerged, and aboveground utility strikes should immediately STOP WORK and contact the Project Manager to discuss the incident unless there are injuries, then call 911 or the available emergency services number for the area and then the Project Manager. The utility strike must be reported to Corporate Health and Safety and Legal Departments immediately and no later than 24 hours. Use the Utility Line Strike Investigation Form as part of the notification process.

Selected utility strike incidents may also utilize a conference call with operations management to review findings and lessons learned. The Business Line Health and Safety Director will make the determination concerning the need to have the incident review call and will arrange the call, if deemed necessary.

5.9 Relationship of this standard to the Project Specific HASP

With the exception of the Utility and Structures Checklist, this standard, including most supplements, are not designed to be printed off and attached to project HASPs. During project health and safety planning, this standard will be reviewed and applicable clearance technologies and methods will be documented on the Utility and Structures Checklist.

Additionally, emergency action standards specific to utility strikes should be addressed. ARC HSFS-019 Supplement 5 provides general guidelines for emergency response to utility strikes. Applicable information may be attached to the Utility and Structures Checklist to facilitate communication of response expectations.

5.10 Required Contract Terms and Conditions

Arcadis' standard client and subcontractor contracts contain required terms and conditions defining responsibility for utility clearance and the allocation of risk associated with an impacted utility. These terms and conditions have prescribed language concerning subsurface work that is presented in Arcadis client contracts and Arcadis' subcontractor contracts, which can be found on the Legal Source site. If such provisions cannot be agreed upon, the reasons are documented and other risk-management actions should be identified, such as limits of liability, add additional physical investigations, additional lines of evidence or utility location, assignment of risk to subcontractors, etc. In addition, any changes to these terms and conditions require approval by Legal Services.

6. TRAINING

Employees responsible for coordinating or conducting utility clearance activities will be familiar with the requirements of this standard. Arcadis in-house 8-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) refresher may provide awareness-level training regarding this utility location and clearance standard.

7. REFERENCES (regulation citation, technical links, publications, etc.)

- Utility and Structures Checklist
- Utility Line Strike Investigation Form
- ARC HSFS-019 Supplement 1, Best Practices for Project Managers (or Their Delegates) Concerning Utility Clearance
- ARC HSFS-019 Supplement 2, Best Practices for Field Personnel Concerning Utility Clearance
- ARC HSFS-019 Supplement 3, Use and Limitations Associated with Location Technologies and Common Utility Clearance Methods
- ARC HSFS-019 Supplement 4, Best Practices for State One Call Procedures and Notifications
- ARC HSFS-019 Supplement 5, Emergency Action Plan guidelines for Utility Strikes
- ARC HSFS-019 Supplement 6, Utility Location SOP for Aquatic Work Activities
- Figure 1 – 30-Inch Tolerance Zone
- ARC HSCS005 Excavation and Trenching
- ARC HSFS0006 Electrical Safety Standard
- One Call and State Law Directory

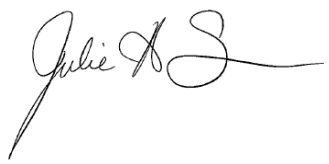
8. RECORDS - DATA RECORDING AND MANAGEMENT

8.1 Utility Clearance Records

All records (maps, checklists and documentation of communications) used to determine the location of utilities should be retained and kept in the project file.

9. APPROVALS AND HISTORY OF CHANGE

Approved By: Julie Santaniello, CSP – Corporate H&S, Manager of Technical Programs



History of Change

Revision Date	Revision Number	Standard Developed/Reviewed By or Revised By	Reason for change
13 December 2006	01	Mike Thomas/Pat Vollertsen	Original document
26 March 2007	02	Mike Thomas/Pat Vollertsen	Put in new company format
15 May 2007	03	Mike Thomas/Pat Vollertsen	Added nation-wide 811 number
6 September 2007	04	Mike Thomas/Pat Vollertsen	Changing over to new template format
22 February 2008	05	Mija Coppola	Changing over to new template format
13 January 2009	06	Mija Coppola	Define lines of evidence
4 October 2010	07	Sam Moyers/Mija Coppola	Reformatting and addition of utility clearance information
13 February 2012	08	Sam Moyers/Mija Coppola	Modified link information for utility strike reporting, clarified local/state requirements in section 4.1 and 4.3
28 January 2013	09	Tony Tremblay	Utility and Structures Checklist revised; hyperlink updated
12 February 2013	10	Amanda Tine/Tony Tremblay	Clarified clearance boundaries for Unqualified staff in Section 5.7 and added information about vehicles and equipment being used near power lines in Section 5.7.1

Implementation Date 13 December 2006	Arcadis HS Standard Name Utility Location and Clearance	ARCADIS Design & Consultancy for natural and built assets
Revision Date 17 March 2017	Arcadis HS Standard No. ARCHSFS019	Revision Number 16

Revision Date	Revision Number	Standard Developed/Reviewed By or Revised By	Reason for change
15 March 2013	11	Kurt Merkle, Rebecca Lindeman / Tony Tremblay	Added additional text to standard for recent lessons learned, added section 5.4 (Locating Technologies) and 5.5 (Clearance Methodologies), added additional details to section 5.6 when working in close proximity to subsurface utilities, and added Supplement 6 - Utility Location SOP for Aquatic Work Activities.
07 July 2013	12	Andrew McDonald/ Tony Tremblay	Removed HSFS-019 Supplement 1, Utility Definitions. Added hyperlink for One Call and State Law Directory. Segregated evidence of sewer or storm drains in USC list. Removed Sam Moyers and added Andrew McDonald as author.
26 September 2014	13	Andrew McDonald/Tony Tremblay	Added Exhibit 1. Definitions and 30 inch tolerance zone. Clarified use of 811 or state one call as a reliable line of evidence. Added best practice to cover backfilling of pre-cleared boreholes. Updated USC list to cover soft dig termination depths and PM review.
23 February 2015	14	Tony Tremblay	Page number correction

Revision Date	Revision Number	Standard Developed/Reviewed By or Revised By	Reason for change
10 May 2016	15	Denis Balcer/Sharon Lingle/Alec MacAdam/Andrew McDonald/Tony Tremblay/Julie Santaniello	ES and Section 4.2 - define subsurface intrusive work; clarify employees providing oversight of utility contractors, Arcadis requirements of operating and interpreting results of utility clearance equipment, and utility clearance before all subsurface intrusive work. Sections 1 and 5.8- changed submarine to submerged. Section 4.1 – added contacting public utility companies to help clear utilities. Section 4.2 – Clarified requirement to complete one year of utility clearance-related experience. Section 4.2 and 4.3 - Added discussion on aboveground activities causing subsurface disturbances. Added responsibility to clear overhead utilities when heavy equipment will be used and to evaluate use of a spotter. Added that repairs to damaged utilities need to be verified as competent and complete. Section 5.2 – Clarified reliable lines of evidence for each subsurface intrusive work point and degrees of certainty. Added all work within 30-inch Tolerance Zone needs Corp H&S preapproval. Section 5.6 and Exhibit 1- Clarify subsurface intrusive work and activity and exemptions for subsurface intrusive work. Section 5.6.1 – Add requirement to evaluate aboveground activities that may lead to subsurface disturbances that may cause damage to shallow underground utilities or structures.

Revision Date	Revision Number	Standard Developed/Reviewed By or Revised By	Reason for change
10 May 2016	15	Denis Balcer/Sharon Lingle/Alec MacAdam/Andrew McDonald/Tony Tremblay/Julie Santaniello	Section 5.7.2 – added non-electric overhead utilities and structures other than power lines need to be identified and marked if working in that area. Section 9 – Changed reviewer from Tony Tremblay to Julie Santaniello. Exhibit 1 – added definitions of Utility Strike, Daylighting, Potholing, Subsurface Intrusive Work, Subsurface Intrusive Activities, and Utility Bank. Standard and Supplements placed on new Arcadis headers. Updated Supplement revision numbers to be consistent with standard. Supplement 2 revised. Utility Clearance and Structures Checklist and Utility Strike Investigation Form revised.
17 March 2017	16	Alec MacAdam/Julie Santaniello	Hyperlink updates; minor formatting; Utility Clearance and Structures Checklist revised.

EXHIBIT 1 – DEFINITIONS

Aboveground Utilities - For the purpose of this procedure, aboveground utilities include, but are not limited to: any aboveground line, pipe, conduit, system, or facility used for producing, storing, conveying, transmitting or distributing communication or telecommunications signals, electricity, gas, liquid, petroleum and petroleum products, coal slurry, hazardous liquids or gases, water under pressure, steam, sanitary sewage, storm water, or other materials, liquids, or gases.

Daylighting – exposing underground utilities or structures through soft dig technology/clearance prior to completing subsurface intrusive activities.

Excavation - Any man-made cut, cavity, trench, or depression, in an earth surface formed by earth removal into which a person can bodily enter.

Overhead Utilities and Structures – Overhead water lines, overhead pipe chases, ceilings in buildings.

Potholing – exposing underground utilities or structures through soft dig technology/clearance prior to completing subsurface intrusive activities.

Subsurface Intrusive Activities – For the purposes of this procedure, subsurface intrusive activities include, but are not limited to: excavations, vertical drilling, installing grounding rod, soil sampling, etc,

Subsurface Intrusive Work – Is any work or activity that breaks the plane of the ground surface. Exemptions include soil sampling using a non-conductive sampling tool to a depth of 6 inches below ground surface (bgs), placement of survey flagging to a depth of 6 inches bgs, and placement of non-conductive survey stake(s) to a depth of 6 inches bgs).

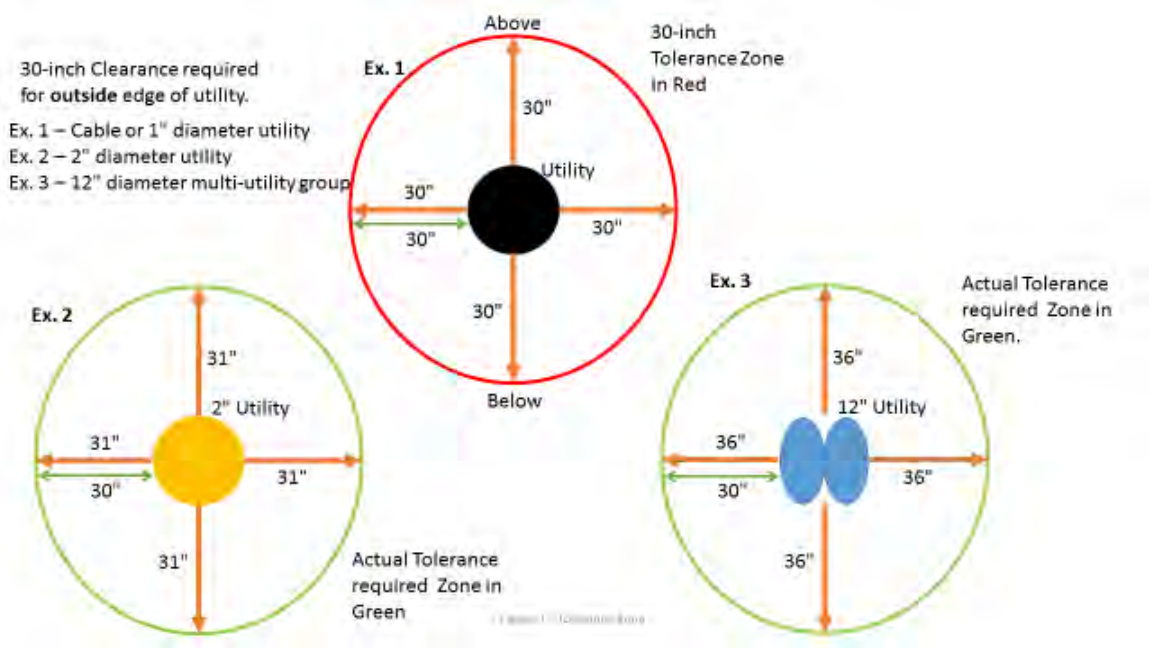
Subsurface Utilities - For the purposes of this procedure, subsurface utilities include, but are not limited to: any underground line, pipe, conduit, system, or facility used for producing, storing, conveying, transmitting or distributing communication or telecommunications signals, electricity, gas, liquid, petroleum and petroleum products, coal slurry, hazardous liquids or gases, water under pressure, steam, storm water, or sanitary sewage; underground storage tanks; tunnels and cisterns; and septic tanks and lines.

Tolerance Zone – The area within 30 inches in all directions from the outside diameter of a located/marked utility in which special care is to be taken. If the centerline of the utility is marked, the diameter of the utility or utility bank/trench must be incorporated into the 30 inches. This area must be hand cleared with non-mechanized equipment. Once the utility has been exposed, if mechanized equipment is planned for use within the 30-inch Tolerance Zone of the utility, such activity must receive pre-approval by Corporate H&S, to mitigate or accept the risk associated with the planned work. See Figure 1 – 30-inch Tolerance Zone.

Utility Bank – a structure containing two or more conduits. A conduit is a single enclosure containing one or more facilities.

Utility Strike – An unplanned contact of a utility (i.e., overhead and structures, aboveground, underground or submerged) during the course of work that results in damage requiring repairs, making a report to the utility owner or requiring further assessment to evaluate the potential for damage

EXHIBIT 2 – FIGURE 1 – TOLERANCE ZONE



APPENDIX F
Field Forms

ARCADIS

Daily Log

Project _____ Project No. _____ Page __ of _____

Site Location _____

Prepared By _____

Date/Time	Description of Activities

ARCADIS UTILITY AND STRUCTURES CHECKLIST

Project: _____

Project Number: _____

Date: _____

Work locations applicable to this clearance checklist **(Photo Document Work Locations)**:

THIS FORM MUST BE COMPLETED IN ENTIRETY PRIOR TO BEGINNING ANY INTRUSIVE WORK

Pre-Field Work

One Call or "811" notified 48-72 hours in advance of work? ☐ Yes ☐ No

Utility companies notified during the One Call process ☐ See attached ticket

_____ _____ _____ _____

_____ _____ _____ _____

List any other utilities requiring notification: ☐ None

_____ _____ _____ _____

Private Locator Contacted ☐ Yes ☐ No

Plan private utility clearance subcontractor assignments, areas, required clearance equipment, depth of clearance needed, types of utilities

Client provided utility maps or "as built" drawings showing utilities? Yes ☐ No

Field Work - This must be completed on site, by staff who have a minimum of one year of field experience in identifying utilities

Lines of Evidence - Must have **3 Reliable Lines of Evidence** Prior to Starting any Intrusive Work

☐ One Call/"811"

Utility Markings Present: ☐ Paint ☐ Pin flags/stakes ☐ Other ☐ None

☐ Client Provided Maps/Drawings **OR** ☐ Maps/Drawings requested but not provided

☐ Client Clearance Name(s)/Affiliation(s) _____

☐ Interview(s): Name(s)/Affiliation(s) _____

_____

Did person(s) interviewed indicate depths of any utilities in the subsurface?

☐ Yes, depths provided:

☐ Did not know or refused to answer

Additional Comments:

☐ Site Inspection & Complete Site Sketch **(Photo Document Marked Utilities & Utility Structures)**

☐ GPR

☐ Air-Knife

☐ Hydro-Knife

☐ Public Records/Maps

☐ Radiofrequency

☐ Metal Detector

☐ Handauger

☐ Potholing

☐ Probing

☐ Private Locator: Name and Company: _____

☐ Marine Locator: Name and Company: _____

☐ Other: _____

_____

Tips for Successful Utility Location:

1. Don't forget to look up
2. Be on site when utilizing private utility locators
3. Select alternate/backup locations during clearance process
4. Mark out all known utilities. Leave nothing to question
5. No hammering- no pickaxes-no digging bars-no hurrying or shortcutting
6. No excessive turning or downward force of handaugers/shovels, etc.
7. Utilities may run directly under asphalt/concrete or be > 5 ft. in depth

Site Inspection

During the site inspection look for the following **("YES" requires additional investigation and must be marked properly prior to performing intrusive work)**:

		Utility Color Codes		
a)	Natural gas line present (evidence of a gas meter)?	Yellow	☐ Yes	☐ No
b)	Evidence of electric lines:	Red		
	i) Conduits to ground from electric meter or along wall?		☐ Yes	☐ No
	ii) Light poles, electric devices with no overhead lines?		☐ Yes	☐ No
	iii) Overhead electric lines present?		☐ Yes	☐ No
c)	Evidence of sewer drains:	Green		
	i) Restrooms or kitchen on site?		☐ Yes	☐ No
	ii) Sewer cleanouts present?		☐ Yes	☐ No
	iii) Combined sewer /storm lines or multiple sewer lines?		☐ Yes	☐ No
d)	Evidence of water lines:	Blue		
	i) Water meter on site or multiple water lines?		☐ Yes	☐ No
	ii) Fire hydrants in vicinity of work?		☐ Yes	☐ No
	iii) Irrigation systems? (Sprinkler heads, valve boxes, controls in building)		☐ Yes	☐ No
e)	Evidence of storm drains:	Green		
	i) Open curbside or slotted grate storm drains		☐ Yes	☐ No
	ii) Gutter down spouts going into ground		☐ Yes	☐ No
f)	Evidence of telecommunication lines:	Orange		
	i) Fiber optic warning signs in areas?		☐ Yes	☐ No
	ii) Lines from cable boxes running into ground?		☐ Yes	☐ No
	iii) Conduits from power poles running into ground?		☐ Yes	☐ No
	iv) Aboveground boxes or housings or wires in work area?		☐ Yes	☐ No
g)	Underground storage tanks:			
	i) Tank pit present?		☐ Yes	☐ No
	ii) Product lines running to dispensers/buildings?		☐ Yes	☐ No
	iii) Vent present away from tank pit?		☐ Yes	☐ No
h)	Do utilities enter or exit existing structures/buildings?		☐ Yes	☐ No
	If Yes, confirm the utility markings outside of structure/building match up.			
i)	Proposed excavation marked in white?	White	☐ Yes	☐ No
j)	Overhead Utilities/Communication Lines **Look Up**:			
	i) Overhead electrical conduit, pipe chases, cable trays ?		☐ Yes	☐ No
	ii) Overhead fire sprinkler system?		☐ Yes	☐ No
	iii) Other overhead lines/utilities, product lines, AC condenser lines?		☐ Yes	☐ No
k)	Aboveground Power lines in or near the work area:			
	i) < 50 kV within 10 ft. of work area?		☐ Yes	☐ No
	ii) >50 - 200 kV within 15 ft. of work area?		☐ Yes	☐ No
	iii) >200-350 kV within 20 ft. of work area?		☐ Yes	☐ No
	iv) >350-500 kV within 25 ft. of work area?		☐ Yes	☐ No
	v) >500-750 kV within 35 ft. or work area?		☐ Yes	☐ No
	vi) >750-1000 kV within 45 ft. of work area?		☐ Yes	☐ No
l)	Other:			
	i) Evidence of linear asphalt or concrete repair?		☐ Yes	☐ No
	ii) Evidence of linear ground subsidence or change in vegetation?		☐ Yes	☐ No
	iii) Unmarked manholes or valve covers in work area?		☐ Yes	☐ No
	iv) Warning signs ("Call Before you Dig", etc.) on or adjacent to site?		☐ Yes	☐ No
	v) Utility color markings not illustrated in this checklist?		☐ Yes	☐ No

Do not initiate intrusive work if utilities are suspected to be present in area and are not located, if markings are over 14 days old, or if clearance methods provide incomplete or conflicting information. Do not perform intrusive work within 30 inches of a utility marking without receiving pre-approval by Corporate H&S .

Name and Signature of person completing the checklist: _____

Date: _____ _____

ARCADIS

Well Abandonment Log

Site Name/Location				Recorded by	
Project/No.		Well ID		Date	
Total Depth (ft)		Construction Material		Casing Diameter (in)	
Start Time		End Time		Well Volume (gallon)	
Method: Overdrill (ft)		Grout/Chips		Bags of Material	
Contractor		Driller		Helper	

	Well Casing Volume Conversion Factor				
gallon/foot	1" = 0.04	1-¼" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65
		1-½" = 0.09	2-½" = 0.26	3-½" = 0.50	6" = 1.47

Time	Notes

NR = Not Recorded

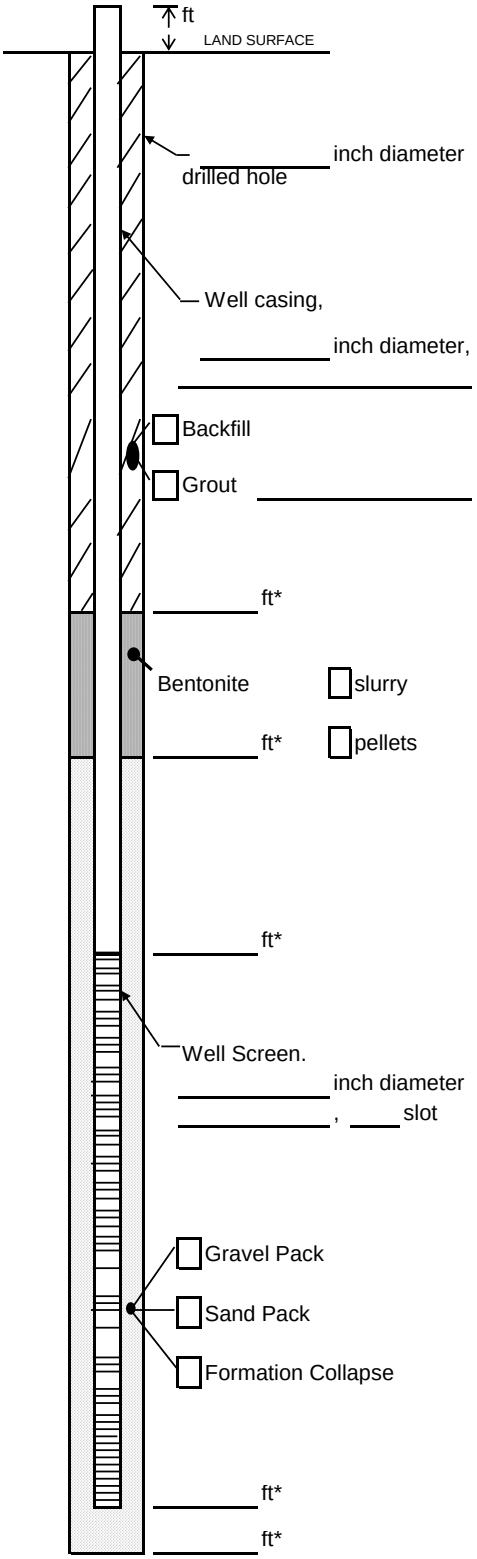
TSD = Total Sound Depth

bgs = below ground surface
PID = Photo Ionization Detector
ppm = part per million

ARCADIS

Well Construction Log

(Unconsolidated)



Measuring Point is
Top of Well Casing
Unless Otherwise Noted.

* Depth Below Land Surface

Project _____ Well _____

Town/City _____

County _____ State _____

Permit No. _____

Land-Surface Elevation and Datum:

_____ feet ☐ Surveyed ☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

Development Technique(s) and Date(s)

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose _____

Remarks _____

Prepared by _____

ARCADIS

Instrument Calibration Log

Project Name: _____ Date: _____

Project Number: _____

Calibrating Personnel: _____

Time of Calibration: _____

Weather Conditions: _____

Barometric Pressure: _____ mm Hg

CALIBRANT	INSTRUMENT	INITIAL READING	VALUE ENTERED	FINAL READING	TIME	TEMP
pH 7.00						
pH 4.01						
Conductivity (_____)						
Turbidity (1.0 NTU)						
Turbidity (10.0 NTU)						
DO (mg/L)						
DO%						
ORP (mV)						
_____						
_____						
_____						

Notes: _____

ARCADIS

Water Level Measurement Form

Page _____ of _____

Project Name: _____ Date: _____

Project Number: _____ Weather: _____

Field Personnel: _____

Well Casing Volumes

gallon/foot			
1-¼" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65
1-½" = 0.09	2-½" = 0.26	3-½" = 0.50	6" = 1.47

Well ID	Time	Measuring Point	TSD (ft)	DTP (ft)	DTW (ft)	Notes

Notes:

_____

_____

_____

ARCADIS

Monitoring Well Development Log

Page _____ of _____

Project/No. _____ Well _____ Date _____

Total Depth _____ Casing Diameter (inches) _____ Purge Method

Water Level _____ Well Volume (gal) _____ Centrifugal _____

Water Column _____ Total Volume Purged _____ Submersible _____

Pump On _____ Pump Off _____ Other _____

Developed By _____

	Well Casing Volumes			
gallon/foot	1-¼" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65
	1-½" = 0.09	2-½" = 0.26	3-½" = 0.50	6" = 1.47

Time	Minutes Elapsed	Rate (gpm) (mL/min)	DTW (ft)	Gallons Purged	pH	Specific Conductance (mS/cm)	Temp. (C) (F)	Turbidity (NTU)	REMARKS (PID readings, color, odor, etc.)

ARCADIS

Groundwater Sampling Form

Page ____ of ____

Project No. _____ Well ID _____ Date _____

Project Name/Location _____ Weather _____

Measuring Pt. Description _____ Screen Setting (ft-bmp) _____ Casing Diameter (in.) _____ Well Material ____ PVC ____ SS ____ Other

Total Depth (ft-bmp) _____ Static Water Level (ft-bmp) _____ Water Column in Well _____ Gallons in Well _____

Calc.Gallons Purged _____ Pump Intake (ft-bmp) _____ Purge Method: Centrifugal _____ Sample Method _____

Gallons Purged _____ MP Elevation _____ Submersible _____

Disp. Bailer _____ Pump On/Off _____

Sample Time: Label _____ Replicate/ Code No. _____ Other _____ Sampled by _____

Time	Minutes Elapsed	Rate (gpm) (mL/min)	Depth to Water (ft) TOC	Gallons Purged	pH	Cond. (µmhos) (mS/cm)	Turbidity (NTU)	Dissolved Oxygen (mg/L)	Temp. (°C) (°F)	Redox (mV)	Appearance	
											Color	Odor

Constituents Sampled	Container	Number	Preservative

Well Information

Well Location: _____ Well Locked at Arrival: Yes / No

Condition of Well: _____ Well Locked at Departure: Yes / No

Well Completion: Flush Mount / Stick Up Key Number To Well:

NOTES: _____

Well Casing Volumes

Gallons/Foot					
	1" = 0.04	1.5" = 0.09	2.5" = 0.26	3.5" = 0.50	6" = 1.47
	1.25" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65	

ARCADIS

Soil/Sediment Sample Log

Sample ID _____ Project/No. _____

Date _____ Sampling Personnel _____

Time _____

Weather _____

DESCRIPTION OF SAMPLE LOCATION:

_____ Soil _____ Sediment

Location _____ Name of Water Body _____

Sample depth (ft) _____ Location _____

Soil type _____ Depth of water _____

_____ Velocity of water _____

Substrate description _____

Description of vegetation _____

_____

FIELD PARAMETERS:

Sample Method _____

Sample Description _____

_____

Color _____

Odor _____

Salinity _____

CONTAINER DESCRIPTION: From _____ Lab _____

Number	Container	Analysis
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
	_____	_____

TOTAL: ☐

ARCADIS

Sample/Core Log

Boring/Well _____ Project/No. _____ Page _____ of ___

Site Location _____ Drilling Started _____ Drilling Completed _____

Total Depth Drilled _____ Feet Hole Diameter _____ inches Type of Sample/ Coring Device _____

Length and Diameter of Coring Device _____ Sampling Interval _____ feet

Land-Surface Elev. _____ feet ☐ Surveyed ☐ Estimated Datum _____

Drilling Fluid Used _____ Drilling Method _____

Drilling Contractor _____ Driller _____ Helper _____

Prepared By _____ Hammer Weight _____ Hammer Drop _____ ins.

Sample/Core Depth (feet below land surface)		Core Recovery (feet)	PID Reading (ppm)	Time/Hydraulic Pressure or Blows per 6 Inches	Sample/Core Description
From	To				

ARCADIS

Sample/Core Log (Cont.d)

Boring/Well _____ Page ____ of ____

Prepared by _____

Sample/Core Depth (feet below land surface) From	To	Core Recovery (feet)	PID Reading (ppm)	Time/Hydraulic Pressure or Blows per 6 Inches	Sample/Core Description

ARCADIS

Investigation Derived Waste Log

Page ____ of ____

Project Name/Location _____ Date _____

Project/No. _____ Weather _____

Technician _____ Project Manager _____

Date	Container Number	Container Type (drum, rolloff, tank)	Container Capacity	Container Description (OT, CT, etc.)	Media (SO, GW)	Content Description	Source	Volume from Source	Storage Location

NOTES:

APPENDIX G
Lab SOPs

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-202
Revision History
Cover Page
Page 1

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument, other minor changes throughout.	DN	1.23.01	1.23.01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 & Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5.23.01	5.23.01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5.21.02	5.21.02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	Minor changes rewording of sec 1.7.6.3 preservation of calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260 B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and Trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practices and correct typos.	LAD	04/06	04/06
09 8260 B	Sect. 11 - added list of waste streams generated and location of satellites. Clarified RT window studies. Added reference to MI Sop. Removed Grand Mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying Calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM Version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSD Batch requirement can not be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10
LAD 05/11 12	Removed Tekmar 2000 and 2016 throughout. Sect. 7.3.1 - Removed ~~5890~~ 5970 GC/MS instrument type. Sect. 7.4.7 - Added RRT information. Sect. 8.1 - Added S.C. marginal exceedence criteria Sect. 9 - Added MDL, LOD and LOQ Criteria. Updated figures.	LAD	05/11	05/11
13	Sect. 5 - Changed Cal Mix and ICV Std. Exp. from 7 to 14 days. Sect. 6 - Add Sample preservation info. Sect. 7.4.4.1 - Added S.C. exemption from 2nd order Cal. Sect. 7.5.1 - Added Extras mix to LCS. Sect. 7.6.12 - clarified nothing why samples need to be reanalyzed. Sect. 8.1 - Added 10% rule for LCS, ICV and MS/D. Sect. 9 - Added LOD/LOQ definitions. Table 1 - Reworded CA for ICV.	LAD	03/12	03/12
14	Sect. 1 and 7 - Removed Quikform references and added reporting from KIMS. Sect. 7 - Removed Soil 200 ug/kg L level and added 80.3% level. Sect. 8 - Added additional marginal exceedance information. Throughout - Fixed typos and made minor edits.	LAD	04/13	04/13
15	Sect. 4 - Removed 5890, 5972 and Tekmar references. (5970 too). Sect. 10 - Updated and added references. Table 3 - Added DoD QSM ver. 5.0 QC requirements. Renumbered Tables 3, 4, 5.	LAD	04/14	04/14

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
16	Sect. 1 – Added sand to m. blk def. Sect. 5 – Separated cal std into VOA gases and non-gases, clarified VOA gases exp. date, combined Surr & IS std, added sand. Sect. 6 – Added Acrolein and Acrylonitrile preservation. Sect. 7 – Added poor purgers % RSD calibration info, added CCV criteria, added use of sand in soil Blank and LCS's, updated cal spike prep. Sect 8 – Added additional LCS criteria. Updated Figures 1 – 3.	LAD	07/16	07/16
17	Sect. 7 - Update BFB Method naming convention, Update Soil calibration levels, Added % Error calculation. Sect. 9 - Added LLOQ reference to LOQ verification.	LAD	03/17	03/17
18	Sect 4, 8, 9 and/or Table 1 - Added LLOQ definition, and LLOQ verification criteria, clarified PQL, LOQ and LLOQ. Added % Error criteria Sect. 7 - Corrected RSE requirement	LAD	06/17	06/17
19	Sect. 5 - Changed IS and Surrogate Standard expiration date from 14 to 30 days. Removed references for 1-Chlorohexane. Removed Table 2 - 4,4 DDT in 4,2 QC criteria. Updated logbook example. Updated references	LAD	10/18	10/18

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-202-19**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-202-19**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each

analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Demonstration of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin

hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire concentrator must be backflushed with warm methanol and water.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph (GC): Hewlett Packard 6890.

4.2 Mass Spectrometer (MS): HP5973

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Column: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.

4.5 Purge and Trap: Archon or Centurion auto samplers, and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS AND STANDARDS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”. After ampulated standards are cranked open, the standard is transferred to a screw top vial and stored in a freezer.

5.3.1 The expiration date for all standards except volatile gases is six months from date of opening the ampule.

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix (without gases) – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

1,2-Dibromo-3-chloropropane	2,2-Dichloropropane	Dibromomethane
1,1,1,2-Tetrachloroethane	2-Butanone	Ethylbenzene
1,1,1-Trichloroethane	2-Chloroethylvinyl ether	Hexachlorobutadiene
1,1,2,2-Tetrachloroethane	2-Chlorotoluene	Idomethane
1,1,2-Trichloroethane	2-Hexanone	Isopropylbenzene
1,1-Dichloroethane	4-Chlorotoluene	Methyl tert-butyl ether
1,1-Dichloroethene	4-Methyl-2-pentanone	Methylene chloride
1,1-Dichloropropene	Acetone	Naphthalene
1,2,3-Trichlorobenzene	Benzene	n-Butylbenzene
1,2,3-Trichloropropane	Bromobenzene	n-Propylbenzene
1,2,4-Trichlorobenzene	Bromochloromethane	p-Isopropyltoluene
1,2,4-Trimethylbenzene	Bromodichloromethane	sec-Butylbenzene
1,2-Dibromoethane	Bromoform	Styrene
1,2-Dichlorobenzene	Carbon disulfide	tert-Butylbenzene
1,2-Dichloroethane	Carbon Tetrachloride	Tetrachloroethene
1,2-Dichloropropane	Chlorobenzene	Tetrahydrofuran
1,3,5-Trimethylbenzene	Chloroform	Toluene
1,3-Dichlorobenzene	cis-1,2-Dichloroethene	trans-1,2-Dichloroethene
1,3-Dichloropropane	cis-1,3-Dichloropropene	trans-1,3-Dichloropropene
1,4-Dichlorobenzene	Cyclohexane	Trichloroethene
	Dibromochloromethane	Vinyl Acetate

5.3.2.2 Gases Calibration Mix - Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Bromomethane
Chloromethane
Dichlorodifluoromethane
Trichlorofluoromethane
Vinyl Chloride
Chloroethane

5.3.2.3 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl chloride	Methyl acetate
Chloroprene	Methyl methacrylate
Diethyl ether	Methyl tert-butyl ether
cis-1,4-Dichloro-2-butene	Pentachloroethane
trans-1,4-Dichloro-2-butene	Propionitrile
1,4-Dioxane	Tertiary-amyl methyl ether
di-Isopropyl Ether	Tertiary-butyl alcohol
Ethyl methacrylate	1,3,5-Trichlorobenzene
Ethyl tertiary-butyl ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.4 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

5.3.2.5 Surrogate/Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 30 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Internal Standards	Surrogate Standards
Pentafluorobenzene	4-Bromofluorobenzene
1,4-Difluorobenzene	1,2-Dichloroethane-D_4
Chlorobenzene-D_5	Toluene-D_8
1,4-Dichlorobenzene-D_4	Dibromofluoromethane

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

5.4 Organic Free Sand – Ottawa Sand or equivalent baked at 110 ºC overnight

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples

All aqueous samples are collected in 40 mL VOA bottles with no headspace, preserved with 1:1 HCl to a pH of <2 and stored at <6°C until analysis. Aqueous samples must be analyzed within 14 days from sample collection if preserved and within 7 days from sample collection if unpreserved.

Samples requiring Acrolein and Acrylonitrile analysis, require preservation of pH of 4-5 and cool to 0-6ºC.

6.2 Soil Samples

Soil samples arriving at the laboratory in Terra-core or Encores Soil samplers must be extruded into water or sodium bisulfate within 48 hours of sampling. Soils samples extruded into water must be frozen at -15°C ± 5°C until analysis. Soil sample extruded into sodium bisulfate must be stored at <6 °C until analysis.

Medium level soil (methanol preserved) samples are sampled into pre-weighed vials containing 5 mLs methanol. Methanol preserved soil samples must be stored at <6°C from the time of receipt at the lab until analysis.

Bulk soil samples are stored at <6 °C until analysis.

All soil/sediments must be analyzed within 14 days from sample collection.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS - Conventions for all instruments are as follows:

- Sub-Directory for data acquisition: C:\HPCHEM\1\DATA
- Tune file: BFB.U

- Method files:

  For BFB Tune: VOABFBAQ.M (waters) or VOABFBSL.M (soils)
  For all samples and standards: I8A05(xx)D.M
  where: I = instrument ID (Each instrument is given a unique identifier).
  A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

  XX = the calibration number in chronological order

- Data files:
  For BFB: IB_ _ _.D

  where: I is the instrument ID
  _ _ _ is a number in chronological order from 000 to 999.

  For all other data files: I_ _ _ _.D
  Where: I is the instrument ID
  _ _ _ _ is a number in chronological order from 0000 to 9999.

  This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

Column:		RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures:	Injection port:	200°
	Transfer line:	150°
	Detector:	240°
Isothermal temperature:		150°
Run time:		8 minutes
Scan start time:		3 minutes

Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

Purge and Trap conditions:

Parameter	Aqueous	Soil
Standby:	35°	35°
Prepurge:	0 min	0 min
Preheat Temp:	Ambient	40°
Sample Temp:	Ambient	40°
Purge:	11 min	11 min
Purge Flow Rate	~24-40 mL/min	~24-40 mL/min
Dry purge:	2-4 min	2-4 min
Desorb preheat:	245°	245°
Desorb Temp:	250°	250°
Desorb Flow Rate:	~15 mL/min	~15 mL/min
Desorb time:	2-5 min	2-5 min
Dry purge:	2-4 min	2-4 min
Bake Time:	10 min	10 min
Bake Temp:	260°	260°
Auto drain:	On	On
Bake gas by pass:	Off	Off
Valve Temp:	120°	120°
Line Temp:	120°	120°
Runs per sample:	1	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

Please refer to the Encon, Archon and Centurion Opperating manuals for more specifics on programming features.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.
To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 200 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

Notes	STD. ID	CAL. Mix 100 ug/mL	Extras Mix 100 ug/mL
AQ curve only	VSTD001	1 uL	1 uL
	VSTD005	5 uL	5 uL
SL curve only	VSTD010	10 uL	10 uL
	VSTD020	20 uL	20 uL
CCV	VSTD050	50 uL	50 uL
	VSTD100	100 uL	100 uL
	VSTD200	200 uL	200 uL

The Surrogate & Internal Standard is spiked by the autosampler. The Archon Surrogate/IS Mix is at 250 ug/ml and the instrument spikes 1 ul. The Centurion Surrogate/IS Mix is prepared at 50 ug/ml and the instrument spikes 5 ul.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Note 1: For poor purging compounds like acetone, the %RSD value may exceed the method acceptance limit of 15% but meet the acceptance criteria for the linear and quadratic calibration models. The average calibration model should still be used because this calibration model is more accurate at concentrations near the LOQ than either the linear or quadratic calibration models.

This is common for acetone but also may apply to other poor purging ketones.

In any instance the % RSD must be below 30%.
Note 2: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used.

7.4.4.2 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\ Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in 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, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤15% for good performing compounds and ≤30% for poor performing compounds.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

For projects or clients requiring DoD QSM, current revision, all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or

equal to those listed in Section 7.4.4, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM, current version, all project analytes must have ± 20%D.

For all other projects, all project analytes should have ± 30%D (± 40%D for poor performers).

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS and Extras standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution plus 5 g baked Ottawa sand, in a VOA vial. The Archon unit adds an additional 10 mL

of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

NOTE: In the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water plus 5 g baked Ottawa sand, spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM, current version, no analytes may be detected >1/2 the PQL and > than the $1/10^{th}$ the measured amount in any sample or $1/10^{th}$ the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be

inspected and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

For projects or clients requiring DoD QSM, current version, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

NOTE: In the event that sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, “Basic Laboratory Technique”, current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Archon Autosamplers

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge

vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

7.6.1.2 Centurion Autosamplers

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, it must be noted in the comments section of the injection logbook. Additional information may be needed to assure that any questions that arise during the review process can be answered.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor samples analyzed after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note

that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the autosampler or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. In this case, the Archon units may be used for the preparative step.

7.6.2 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.
Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and

corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An “M” qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into Kims. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the

standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.2 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. For non-DOD clients, the exceedances from the laboratory established limits or nominal limits must be less than ten percent of the client compound list. For DOD clients, all of the compounds of interest must fall within either Katahdin's statistically derived limits or the DOD QSM, current version, limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state. Additionally, the laboratory statistically derived LCS limits should fall within 70-130%.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

For projects or clients requiring DoD QSM, current version, all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.3 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.4 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory

is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018

The 2009 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

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TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all a nalytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs ≤ 30% for CCCs. Refer to section 7.4.3 also. % Error ≤ 30%	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances.	If the surrogate recoveries in the ICV are low but the target analytes are acceptable, narrate. If the ICV recovery is high but the sample results are <PQL, narrate. If the ICV is out but the batch LCS is in criteria, narrate.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes ≤ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL/LLOQ with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
MDL Studies, LOD and LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	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 performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	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: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time 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.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (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. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	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 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 analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	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. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	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 with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-19	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl ether	45	43, 87

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Ethylbezene	91	106
Ethyl methacrylate	69	41, 99
Ethyl tertiary-butyl ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl alcohol	43	41, 42
Isopropylbezene	105	120
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene chloride	84	86, 49
Methyl acetate	43	74
Methyl methacrylate	69	41, 100
4-Methyl-2-pentanone	43	58, 85, 100
Methyl tert-butyl ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
n-Propylbenzene	91	120
Styrene	104	78
Tertiary-amyl methyl ether	73	55, 87, 71
Tertiary-butyl alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
Xylenes (Total)	106	91

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 5

ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenzene	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butyl ether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl ether			n-Butylbenzene
Carbon disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES

GCMS-T INSTRUMENT RUNLOG **DATE/TIME OF BFB INJECTION:**

Method - Check One	8260			8260 - 10 mL			8260 SIM				624			524
STD IDs: *	BFB STD			CAL STD			GAS STD			XTRAs STD				LCS/MS STD
SURR STD				IS STD			Prep Method			Criteria			pH Paper Lot #:			KI Paper Lot #
SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	5030	5035	1311	KAS	DoD	QAPP	Y/N	ANALYST	pH	TRC	COMMENTS

* Refer to page 1 for standard preparation instructions.

VOA-039 - Revision 4 - 10/17/2018

0000007

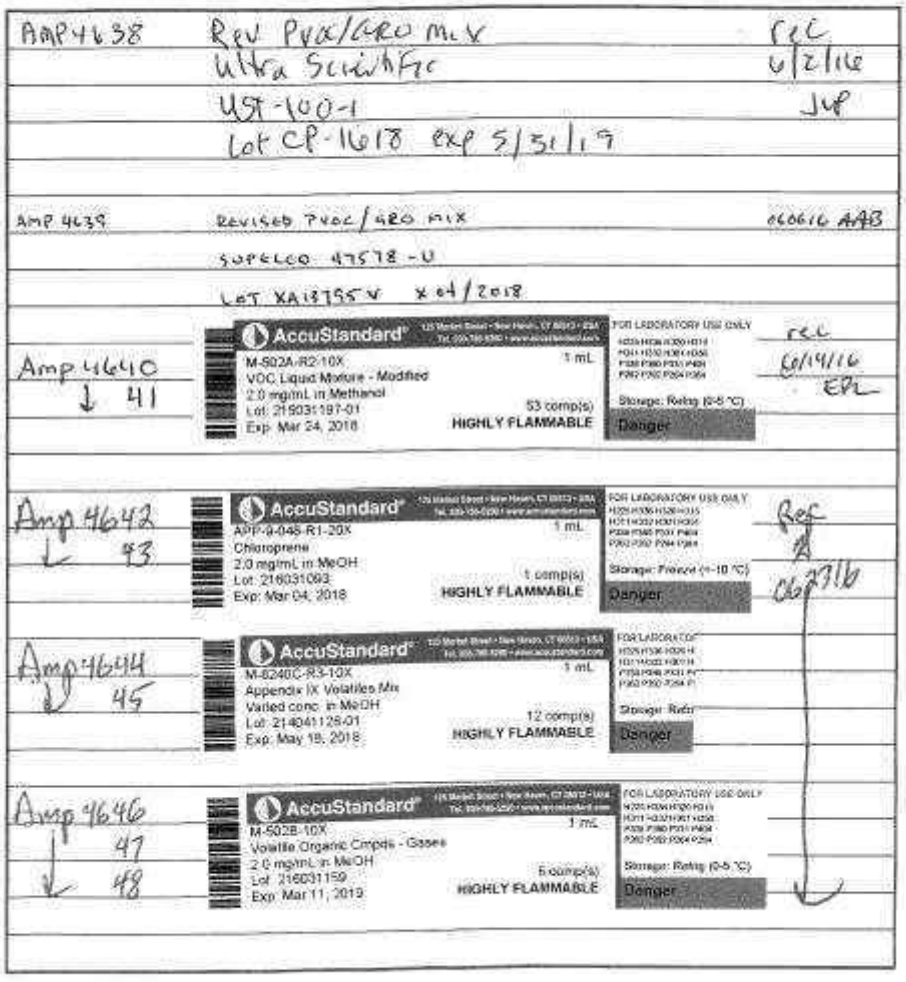
TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
STOCK STANDARDS RECEIVED
GCMS VOLATILES LABORATORY

REVIEWED BY/DATE



VOA-022 – Revision 1 – 10/15/2013

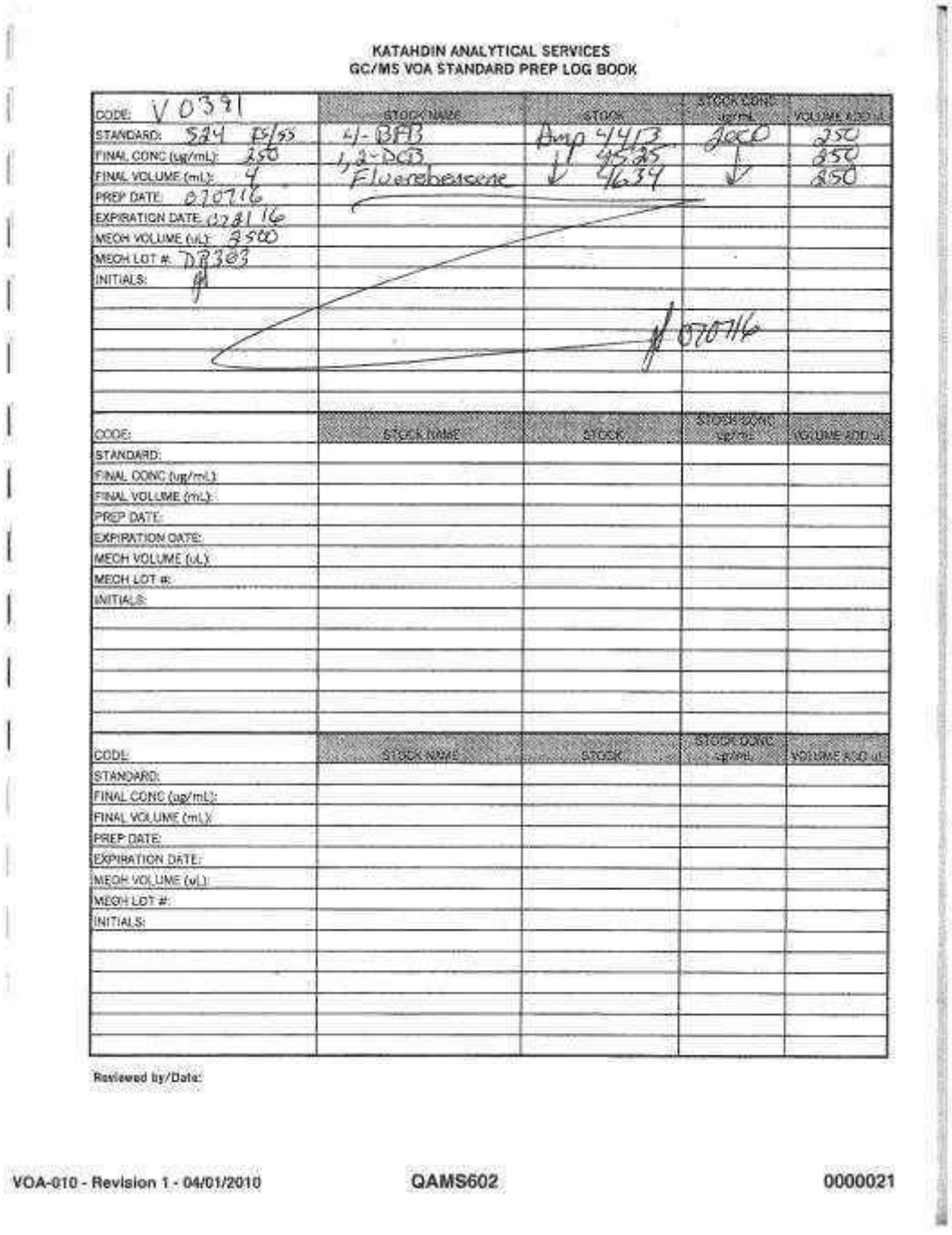
QAMS589

0000013

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V0391	STOCK NAME	STOCK	STOCK CONC (ug/mL)	VOLUME ADD (uL)
STANDARD: 524 ES/SS	4-BFB	Amp 4413	2000	250
FINAL CONC (ug/mL): 250	1,2-DCB	4525	↓	250
FINAL VOLUME (mL): 4	Fluorobenzene	4634	↓	250
PREP DATE: 070716				
EXPIRATION DATE: 072816				
MEOH VOLUME (uL): 4500				
MEOH LOT #: DB303				
INITIALS:				
			070716	

CODE:	STOCK NAME	STOCK	STOCK CONC (ug/mL)	VOLUME ADD (uL)
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

CODE:	STOCK NAME	STOCK	STOCK CONC (ug/mL)	VOLUME ADD (uL)
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

Reviewed by/Date:

VOA-010 - Revision 1 - 04/01/2010 QAMS602 0000021

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Prepared By: GC/MS Group Date: 7/98

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 5035	Format changes, added pollution prevention, minor changes throughout	DN	1·23·01	1/30/01
02 5035	Reorganiced sections 4, 5, 6, 7 and 8.	MRC	07.02.09	07.02.09
03 5035	Editted section 6.4.3 to include the addition of 5mL of H2O to Sample	LAD	020305	020305
04 5035	Balance weighs to 0.1g grammatical corrections formatting corrections	LAD	04/06	04/06
05	Added 3585 Reference. Sections 6.1.2.3, 6.4.3 and 7.2.2: changed 20mL to 5mL.	LAD	09/08	09/09

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Small changes to sections 3.2, 4.2.2.1, and 7.1.1 to address the differences between Purge and Trap Autosamplers. Added references.	LAD	03/12	03/12
07	Sec 6.1.1.2 - Changed 20mL to 5mL. Secs 6.1, 6.2, 6.4: edited and added steps for clarity. Changed required balance accuracy from 0.1 g to 0.01 g throughout. Minor formatting and grammatical changes. Updated references	LAD	03/18	03/18

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-214-07**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-214-07**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

1.0 SCOPE AND APPLICATION

This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 5.0 to 200 µg/kg range.

Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent by method 3585. These samples are also purged using Method 5030.

Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analyses using Methods 5030, 5035 and 3585. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of soils by method 5035 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

2.0 SUMMARY OF METHOD

2.1 **Low concentration soil method:** generally applicable to and soils and other solid samples with VOC concentrations in the range of 5.0 to 200 µg/kg. Volatile organic compounds (VOCs) are determined by collecting an approximately 5 g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. If the samples are sent to the laboratory in an Encore sampling device, the laboratory extrudes the sample into this vial containing a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free laboratory reagent grade water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 **High concentration soil method:** generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent (e.g., methanol) to dissolve the volatile organic constituents. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5 g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that contains a known aliquot of a water-miscible organic solvent (e.g., methanol). An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent. Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is diluted in the appropriate solvent. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) and surrogates are added to the solution that is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared in n-hexandecane according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free laboratory

reagent grade water or water miscible solvent and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free laboratory reagent grade water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free laboratory reagent grade water is not necessary.

3.4 The laboratory where volatile analysis is performed should be free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

A standard 40 ml VOA vial is used (e.g. ESS pre-cleaned certified 40 ml clear Type I borosilicate glass vials, open-top/polypropylene with 0.125 inch septa).

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. The Purge and Trap autosampler systems at Katahdin meet the following criteria:

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5 g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40 °C and holding it at that temperature while the inert purge gas is allowed to pass through the

sample. The device should also be capable of introducing at least 20 mL of organic-free laboratory reagent grade water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging (e.g. using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see Sec. 4.2.2).

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed; it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240 °C – 250 °C) are employed. The analyte 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 29 - 30 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap.

4.2.2.1.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.1.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.1.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35° are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.2 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25 mL glass hypodermic syringes with Luer-Lok (or equivalent) tips (other sizes are acceptable depending on sample volume used)

4.3.2 25 µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent)

4.3.3 Micro syringes: 10 and 100 µL

4.3.4 Syringes: 0.5, 1.0, and 5.0 mL, gas-tight

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60 mL, septum-sealed, to collect samples for screening and dry weight determination

4.4.1.2 40 mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g

4.4.3 Glass scintillation vials – 20 mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples

4.4.4 Volumetric flasks - Class A, 10 mL and 100 mL, with ground glass stoppers

4.4.5 2 mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial

4.4.7 Disposable Pasteur pipettes

4.4.8 Magnetic stirring bars - PTFE or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.2 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.3 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.4 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, laboratory reagent grade water added, cap, and septum.

5.0 REAGENTS AND STANDARDS

5.1 Organic-free laboratory reagent grade water - All references to water in this method refer to organic-free laboratory reagent grade water.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ – free of interferences at the detection limit of the target analytes.

5.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples (low level analysis) and high concentration soil and solid waste samples (high level analysis).

The choice of low or high level is determined by the requirements of the project. However, since the low-level method is only valid for a certain concentration range, a sample for analysis by the high-level method must also be collected to ensure quantification of all target analytes. Katahdin typically supplies three vials prepared for low level analysis and one vial for high level analysis per field sample.

Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

Note: Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards should only be added to the vials once they are back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.1 Low level analysis vials

Low concentration soil samples can be preserved by two different methods. One method involves adding water to the vial prior to sample collection, followed by freezing within 48 hours of sample collection. Alternatively, samples can be preserved using a 20% sodium bisulfate solution. The sampling personnel should examine and pre-test the soils to be collected prior to actual collection in order to make the proper determination for the correct preservation technique. Low concentration soil samples containing carbonate minerals may effervesce upon contact with an acidic preservation solution such as sodium bisulfate. Typically, the water/freezing method is

used unless sodium bilsulfate preservation is specifically requested by the client. The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 Add 5 mL of water or 5 ml of 20% sodium bisulfate solution (depending on the preservation method) to the vial and seal the vial with the screw-cap and septum seal.

6.1.1.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible). It is important that labels and tape not cover the junction of the screw top and vial. Labels and tape must also be applied smoothly (i.e. no wrinkles) to prevent autosampler failures.

6.1.1.4 Weigh the prepared vial to the nearest 0.01 g and record it on the label.

6.1.2 High level analysis - no preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 40 mL glass vials with septum seals (see Sec. 4.4).

6.1.3 High level analysis vials - methanol preservation

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.3.1 Add 5 mL of methanol to each vial.

6.1.3.2 Seal the vial with the screw-cap and septum seal.

6.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.3.4 Weigh the vial to the nearest 0.01 g and record it on the label.

Note: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of > 0.01 g) should not be used for sample collection.

6.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol, sample vials may be prepared as described in Sec. 6.1.3. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative (see Sec. 6.1.2.)

Some oily waste samples that are not soluble in methanol may be soluble in PEG (see Sec. 7.3.)

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™, and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

The low-level method uses one or more of these options for the sampling/preservation of soils:

6.2.1 Soil sampled into a vial with 20% sodium bisulfate solution (see Sec 6.1.1).

6.2.2 Soil collected in an EnCore™ sampler and immediately shipped to the laboratory for further preservation (within 48 hours).

6.2.3 Soil collected in a vial with water, sealed in the field and shipped to the laboratory immediately in order to meet the method preservation requirement to freeze within 48 hours of collection (see Sec. 6.1.1).

The high-level method uses one of these options for sampling/preservation of soils:

6.2.4 Soil sampled into a vial with methanol (see Sec. 6.1.3).

6.2.5 Soil collected in an EnCore™ sampler and shipped to the laboratory immediately in order to meet the method requirement of preserving in methanol within 48 hours of collection (see Sec. 6.1.1).

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4 °C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. Samples should be shipped on the day of sampling if at all possible.

6.4 Sample storage

6.4.1 When samples arrive at the laboratory, they must be sorted as follows to ensure proper preservation. The sample storage areas should be free of organic solvent vapors (VOA walk-in or VOA freezer).

6.4.1.1 Samples arriving in a vial with DI water must be frozen (stored at -10 °C) within 48 hours of collection. Store in the VOA freezer.

6.4.1.2 Samples arriving in a vial with sodium bisulfate or methanol should be stored at 4 °C. Store in the VOA walk-in refrigerator.

6.4.1.3 Samples arriving at the laboratory in EnCore™ samplers need to be extruded into the appropriate vials for preservation within 48 hours of sample collection. For low level analysis, extrude into vials prepared as in Sec. 6.1.1. For high level analysis, extrude into vials prepared as in Sec. 6.1.3. After extruding the samples, store the vials appropriately (see Secs 6.4.1.1 and 6.4.1.2).

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, or the addition of 5 mL of water and storage at -10 °C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Such steps should be outlined in the project plan.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

7.0 PROCEDURES

This section describes procedures for the low concentration soil method (low level analysis), the high concentration soil method (high level analysis), and the procedure for oily waste samples. High level samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

For the high concentration soil and oily waste samples, the surrogate compounds may either be spiked into the solvent at the time of extraction or the laboratory reagent grade water containing an aliquot of the extract prior to analysis.

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.1 Low concentration soil method - approximate concentration range of 5 to 200 µg/kg (The concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.1.1 Purge and Trap Autosampler Operation

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated by the analytical method to be used. When a GC/MS method is used, internal standard calibration is employed.

Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 10 mL of water, to heat the sample to 40 °C, and to hold the sample at 40 °C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.2 Sample purge-and-trap

This method is designed for a 5 g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed, and MUST remain sealed in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.2.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.1.2.2 Without disturbing the hermetic seal on the sample vial, add 10 mL of organic-free laboratory reagent grade water and the appropriate internal standards and the surrogate compounds for the analytical method to be employed as described in Sec. 5.0 of Method 5000. This is carried out either manually or using the automated sampler. Other volumes of organic-free laboratory reagent grade water may be used. However, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free laboratory reagent grade water. Prior to purging, heat the sample vial to 40 °C for 1.5 minutes, or as described by the manufacturer.

7.1.2.3 For the sample selected for matrix spiking, add the appropriate matrix spiking solution for the analytical method to be employed, as described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions.

7.1.2.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a transfer line to a trap packed with suitable sorbent materials.

7.1.3 Sample Desorption

Non-cryogenic interface - After the 11 minute purge, place the purge-and trap system in the desorb mode and preheat the trap to 245 °C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/min for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.4 Trap Reconditioning

After desorbing the sample for 1 to 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245 °C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10

minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2 High concentration soil method – for samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. (If a sample is not soluble in methanol, it may be soluble in PEG.) An aliquot of the extract is added to organic-free laboratory reagent grade water containing surrogates, internal and matrix spiking standards (added manually or by the autosampler), purged according to Method 5030, and analyzed by an appropriate determinative method. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Remove a representative aliquot with a spatula.

7.2.2 For soil and solid waste samples that are soluble in methanol or PEG, add 5.0 g (wet weight) of sample to a tared 40 mL VOA vial using a calibrated (refer to Katahdin SOP, CA-102, Balance Calibration) top loading balance. Record the weight to 0.01 g. Add 5 mL of methanol to the vial containing the sample and shake for two minutes.

Note: The steps in Secs. 7.2.1, 7.2.2, and 7.2.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.2.3 For soil and solid waste samples that were collected in methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field.

7.2.4 For each new lot of methanol, add an appropriate aliquot of the methanol to 20 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 11.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. (If a sample is not soluble in methanol, it may be soluble in PEG.) However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free laboratory reagent grade water, purged according to Method 5030, and analyzed using an appropriate determinative method.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.3.2. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane and shake for two minutes.

7.3.2 For oily samples that are soluble in methanol or PEG if the waste was not preserved in the field, tare a 10-mL volumetric flask, or a VOA vial, weigh 1 g (wet weight) of the sample into the tared vessel and add 10.0 mL methanol or PEG with a calibrated syringe. If a vial is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis. Invert the vial a minimum of three times to mix the contents.

7.3.4 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field, and proceed with Sec. 7.3.5.

7.3.5 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.3.6 Add an appropriate aliquot of the methanol or PEG to 5.0 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 11.0 in Method 5030 and follow the procedure for purging oily waste samples.

7.4 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample. Refer to Katahdin SOP, CA-717, for determination of % dry weight.

Note: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free laboratory reagent grade water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

8.3 Sample Quality Control for Preparation and Analysis - See the appropriate analytical method to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.0 METHOD PERFORMANCE

Refer to appropriate analytical method.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5035, SW-846, USEPA, Revision III, June, 1997.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5035A, SW-846, USEPA, Draft Revision I, July, 2002.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 3585, SW-846, USEPA, Revision IIIB, Nov., 2004.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5030C, SW-846, USEPA, Revision III, May, 2003.

"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Office of Solid Waste and Emergency Response, U.S. EPA

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-214-07	METHOD 5035, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	(1) Use methanol prep for all high concentration soils. (2) For high concentration soils, leave all extract in the vial with the soil for storage.	(1) For high concentration soils from an unknown source, perform a solubility test. (2) For high concentration soils, pipet approximately 1 mL of extract into a GC vial for storage.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D

Prepared By: Semivolatile Group Date: 02.11.09

Approved By:

Department Manager: [signature] Date: 2-11-09

Operations Manager: Deborah J. Radeau Date: 2.11.09

QA Officer: Susie Dimond Date: 02.10.09

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated to reflect DOD QSM version 4.1 compliance and new standard preparation procedures.	EM	08/09	08/09
02	Edited Sections 5.3.2 and 7.5.1 to reflect current calibration levels. Added Table 2 - DoDQSM QC criteria. Renumbered Tables 2-6. Added references.	LAM	08/10	08/10
03	Added new instrumentation. Added LOD and LOQ verification information. Added DDT breakdown equation. Added TNI reference. Minor changes to reflect current techniques.	LAM	01/12	01/12
04	Sect. 1 & 7 - Changed reporting through quickform to reporting through KIMS. Sect. 8 - Added Marginal Exceedance criteria.	LAM	04/13	04/13
05	Sect. 7 - Corrected typo and formatting. Sect. 10 - Added and updated references. Added Table 3 - DoDQSM5.0 QC Requirements Renumbered Tables 3→8.	LAM	07/14	07/14

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Table 1- Corrected ICV acceptance criteria. Changed KAS INC. to KAS throughout.	LAD	08/15	08/15
07	Sect. 5 - Added standards to title. Sect. 7 - Updated GC/MS operating parameters added resolution criteria for structural isomers.	LAD	03/16	03/16
08	updated for SW846 Update V references.	LAD	10/16	10/16
09	Sect. 7 - Added % Error calculation Sect. 9 - Added LLOQ reference	LAD	03/17	03/17
10	Sect. 1, 9 and Table 1 - Added LLOQ definition and verification acceptance criteria, clarified PQL, LOQ and LLOQ. Added SC does not allow for non-linear calibration model.	LAD	06/17	06/17

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-226-10, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-226-10, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270D.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions:

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount; a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. Analyzed immediately after calibration,

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation Of Aqueous Samples For Extractable Semivolatile Analysis", SOP CA-512, "Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

Target and surrogate compounds are identified and compared to the mass spectra obtained from the analysis of standard solutions containing the same compounds. A relative response factor is established for each target compound and surrogate against an internal standard during the most recent initial or continuing calibrations. The identified compound is then quantitated using the relative response factor, the amount of internal standard in the sample, the initial volume of sample, and any other factors, such as dilutions.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890, 6890 and/or 6890N

4.2 Mass Spectrometers (MS): HP5973, HP5972, HP5970 and/or 5975B

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As and HP 7683s

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W

Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

4.10 1.8 mL vials with 350uL inserts

4.11 Crimp tops with Teflon lined septa

5.0 REAGENTS AND STANDARDS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

The standards are prepared on an as needed basis (but not less than every 6 months) and stored in screw cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard stock mix that contains those compounds commonly considered 8270 and those compounds commonly considered Appendix IX compounds. The compound dinoseb should <u>not</u> be added to this stock as it is only available in methanol. This will be added separately to each calibration level. Use Table 5 as a guide. The stock should be prepared at 125 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source

other than that used to make the calibration standards, prepare separate standards mixes (A and B) such that Standard Mix A contains those compounds commonly considered 8270 and Standard B Mix contains those compounds commonly considered Appendix IX compounds. Use Table 5 as a guide. Each stock should be prepared at 100 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP, Pentachlorophenol, Benzidine and DDT at a final concentration of 25 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts should be refrigerated until analysis. Extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: L8270CXX.M (all samples and standards)

Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument has a unique ID)

DFTPP tuning acquisition: DFTPP390.M

NOTE: All acquisition parameters must be identical for L8270CXX.M and DFTPP390. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID. This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID.

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:
    - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.
    - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the “Position and run” command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

Mass	Criteria
51	30 to 60% of mass 198
68	< 2% of mass 69
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 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

All ion abundances must be normalized to m/z 198, the nominal base peak.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

The following are the GC/MS operating conditions for injection of DFTPP.

Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	275°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0-6.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at approximately 60 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using “Edit Sample Log Table”. For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Department Manager, or senior chemist within the GC/MS group.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

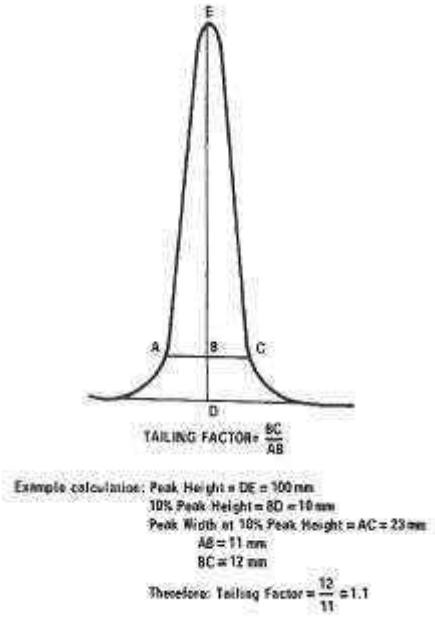
If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

Tailing Factor = $\frac{BC}{AB}$

.where: AC = the width at 10% height
DE = height of the peak
B = the height at 10% of DE

Example:



In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

The Enviroquant system uses the following formulas to determine the % breakdown of DDT and Endrin -

% Breakdown DDT = $\frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} * 100$

% Breakdown Endrin = sum of degradation peak areas (aldehyde + ketone) * 100
sum of all peak areas (endrin + aldehyde + ketone)

Degradation of DDT to DDE and DDD should not exceed 20%.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270D

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated. The calibration consists of a six point curve. The calibration levels are 10, 25, 50, 75, 100 and 125 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	SVOA Stock Soln Added (uL)	1000 ug/mL dinoseb Standard (uL)	$MeCl_2$ Added (uL)	Final Vol (uL)	IS Added (uL)
10	16	2	182	200	2
25	40	5	155	200	2
50	80	10	110	200	2
75	120	15	65	200	2
100	160	20	20	200	2
125	100	0	0	100	1

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C hold 0.5 minutes 20°/min. to 260°C, hold 0.0 minutes 5°/min to 280°C, hold 0.0 minutes 18°/min to 300°C, hold 4.39 minutes
Final Column Temperature hold	300°C
Run Time	21 minutes
Scan Start Time	2.5 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

The conditions are set up in the method files L8270CXX.M.

After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the Mean RRF and %RSD for all analytes. If information is needed concerning the use of these programs, consult the Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for

each target analyte must be less than or equal to 20%.

It is recommended that a minimum response factor (Table 8) for target analytes be achieved as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Therefore the minimum response factors in Table 8 must be verified at the lowest calibration level.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 20% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 20%, then a calibration option outlined in section 11 of method 8000D will need to be employed.

Option 1 (Section 11.5.2 of method 8000D - Rev. 4, 07/14), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target reports r^2. This is calculated by either calculating r or squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control".

Corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

Option 2 (Section 11.5.3 of method 8000D - Rev. 4, 07/14), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Please note that some options may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration models for compliance work originating in their state.

If more than 10% of the compounds in the initial calibration exceed the 20% RSD limits and do not meet the minimum correlation coefficient of determination criteria in option 1 or 2, the GCMS system is considered out of control and the calibration must be repeated. Note: Maintenance may have to be performed.

Internal standard (IS) responses and retention times in all standards must be evaluated immediately after data acquisition; if the RT for any IS changes by more than 0.50 minutes from the latest daily calibration standard, corrections must be made to the chromatographic system. If the extracted ion current profile (EICP) area for any IS changes by more than a factor of two (-50% to +100%), corrective action must be performed.

Each GC/MS system must be calibrated following system corrective action, including ion source cleaning or repair and column removal or replacement.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270. The SSTD050 in the curve may be used as the calibration verification standard as long as it meets the calibration verification acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate

the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\ Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in 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, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

7.5.2.3 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. The percent difference for each target analyte must be less than or equal to 30%. For clients requiring DOD criteria, all project analytes must be within +/- 20% of

true value.

7.5.2.4 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.5.3 Continuing Calibration

A calibration verification check standard must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 50 ng/uL.

After quantitation of the 50 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences.

- All target analytes must have a % difference of +/- 20%D in order to be considered in criteria.

- All target analytes should meet the minimum RRF criterion as in ICAL (Table 8) in order to be considered in criteria.

These conditions must be met before method blank and/or sample analysis can begin.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.
If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.

- Change the septum; clean the injection port; install a clean, silanized

quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different gas chromatographic retention times. Sufficient gas chromatographic 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. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis
by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add a minimum of 100 uL of sample extract and 1.0 uL (for each 100 ul of sample) of the 4000 ng/uL IS stock to the vial and then cap. This gives a 40 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who analyzed the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Tables 1, 2 and 3). These tables give acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or Istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual

integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. All manual integrations are initialed, dated and given a code which describes the reason for the manual integration.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.
For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is, to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

7.8.1 Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.

7.8.2 In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.

7.8.3 Let cool; drain nitric acid and thoroughly flush the liner with water.

7.8.4 Bake briefly in the muffle oven until liner is dry and cool to room temperature.

7.8.5 Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.

7.8.6 Take out the liner and rinse it thoroughly with toluene.

7.8.7 Rinse the liner thoroughly with purge and trap grade methanol.

7.8.8 Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform

other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Tables 1, 2 and 3 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Tables 1, 2 and 3, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Tables 1, 2 and 3 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Tables 1, 2 and 3 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (laboratory reagent grade water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.
Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14
- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270 analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. For DoD ELAP work, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the mid-point of the initial

calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out-of-criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances. South Carolina does not allow for marginal exceedances for compliance work originating in their state.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also

can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.
Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds. Nominal limits of 60-140% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

8.6 QC Requirements

Refer to Tables 1, 2 and 3 for a summary of QC requirements, acceptance criteria, and corrective actions. Tables 1, 2 and 3 criteria are intended to be guidelines for analysts. The table does not over all possible situations. If any of the QC

requirements are outside the recovery ranges listed in Tables 1, 2 and 3, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Tables 1, 2 and 3 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8270D. Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IV and V, July 2014, Method 8270D.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IV and V, July 2014, Method 8000D

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-803, Laboratory QA: Self Inspection System, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD 20% for all compounds. If not met: Option 1) Linear least squares regression: r ≥ 0.995 Option 2) Non-linear regression: coefficient of determination (COD) r^2 ≥ 0.99 (6 points for second order) Up to 10% target analytes may be outside of the above criteria % Error ≤ 30% Refer to section 7.5.2.1 for additional information.	Perform instrument maintenance if necessary. Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	All target analytes: ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst and annually there after.	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL/LLOQ	1) Investigate source of contamination 2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.	1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. 2) If an LCS/LCSD was performed and only one was unacceptable, narrate. 3) If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. 4) If the LCS rec. is high but the sample results are <PQL, narrate. 5) Otherwise, reprep a blank and the remaining samples.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Current statistical limits	1) Check chromatogram for interference; if found, flag data 2) If not found, check instrument performance; if problem is found, correct and reanalyze 3) If still out reextract and analyze sample 4) (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits and section 8.5 of this SOP.	1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. 2) (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

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TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	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.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since 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 analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	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. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3

DOD QSM 5.0/5.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	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 performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	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: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time 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.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (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. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TABLE 3

DOD QSM 5.0/5.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	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 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 analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3

DOD QSM 5.0/5.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	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. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	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 with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 4

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-226-10	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4

Target and Surrogates:

Pyridine (not on TCL list)
N-Nitrosodimethylamine (not on TCL list)
Aniline (not on TCL list)
Phenol
Bis (2-chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol (not on PP list)
2-Methylphenol (not on PP list)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)
4-Methylphenol (not on PP list)
N-Nitroso-di-n-propylamine
Hexachloroethane
Ethyl methanesulfonate (8270 C)
Methyl methanesulfonate (8270 C)
2-Picoline (8270 C)
N-Nitrosomethylethylamine (Appendix IX)
N-Nitrosodiethylamine (Appendix IX)
N-Nitrosopyrrolidine (Appendix IX)
N-Nitrosomorpholine (Appendix IX)
2-Fluorophenol (surrogate)
Phenol-d6 (surrogate)

Internal Standard: Naphthalene-d8

Target and Surrogates:

Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Acetophenone (8270 C)
Benzoic acid (not on PP list)
Bis (2-chloroethoxy) methane
2,4-Dichlorophenol
2,6-Dichlorophenol (8270 C)
1,2,4-Trichlorobenzene
a, a-Dimethyl-phenethylamine (8270 C)
Naphthalene
4-Chloroaniline (not on PP list)
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
N-Nitrosodi-n-butylamine (8270 C)
N-Nitrosopiperidine (8270 C)
o-toluidine (Appendix IX)
o, o, o-Triethylphosphorothioate (Appendix IX)
Hexachloropropene (Appendix IX)
Isosafrole (Appendix IX)
Nitrobenzene-d5 (surrogate)

Internal Standard: Acenaphthene-d10

Target and Surrogates:

Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol (not on PP list)
1-Chloronaphthalene (8270 C)
2-Chloronaphthalene
2-Nitroaniline (not on PP list)
Dimethyl phthalate
Acenaphthylene
3-Nitroaniline (not on PP list)
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran (not on PP list)
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethyl phthalate
4-Chlorophenylphenyl ether
Fluorene
4-Nitroaniline (not on PP list)
1-Naphthylamine (8270 C)
2-Naphthylamine (8270 C)
Pentachlorobenzene (8270 C)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamene (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)
Dinoseb (Appendix IX)

Internal Standard: Chrysene-d12

Target and Surrogates:

Benzidine (not on TCL list)
Pyrene
Butylbenzyl phthalate
3,3'-Dichlorobenzidine
p-Dimethylaminoazobenzene (8270 C)
Benzo (a) Anthracene
Bis (2-ethylhexyl) phthalate
Chrysene
3-Methylcholanthrene (8270 C)
Aramite (Appendix IX)
Chlorobenzilate (Appendix IX)
3,3'-Dimethylbenzidine (Appendix IX)
2-Acetylaminofluorene (Appendix IX)
Terphenyl-d14 (surrogate)

Internal Standard: Perylene-d12

Target and Surrogates:

Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (a) pyrene
Indeno (1,2,3-cd) pyrene
Dibenz (a, h) anthracene
Dibenz (a, j) acridine (8270 C)
Benzo (ghi) perylene
7,12-Dimethylbenz (a) anthracene (8270 C)
Hexachlorophene (Appendix IX)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 6

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

RSD 20% for all compounds.
If not met:
Option 1) Linear least squares regression: $r \geq 0.995$
Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order)
Up to 10% of target analytes may be outside of the above criteria
Refer to section 7.5.2.1 for additional information.
Recommended minimum RF criteria for analytes listed in Table 8.

Continuing Calibration Check Criteria

All target analytes: $\leq$ 20%D
Recommended minimum RF criteria for analytes listed in Table 8.

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 7

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
2-Picoline	93	66,92
Aniline	93	66,65
N-Nitrosodimethylamine	42	74,43
Phenol	94	65,66
Bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Benzyl alcohol	108	77,79
2-Methylphenol	107	107,108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	107,108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 7 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Hexachlorobenzene	284	142,249
1,2-Diphenylhydrazine	184	77,92
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Di-n-butyl phthalate	149	150,104
Carbazole	167	166,139
Fluoranthene	202	101,203
Benzidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α-Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120

TABLE 7 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Dinoseb	211	163, 240
Diallate	86	234,43,70
4-Nitroquinoline-1-oxide	174	101,128,75,116
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
p-Dimethylaminoazobenzene	225	120,77,105,148,42
7,12-Dimethylbenz(a)anthracene	256	241,239,120
3-Methylcholanthrene	268	252,253,126,134,113
Aramite	185	191,319,334,197,321
Chlorobenzilate	251	139,253,111,141
3,3'-Dimethylbenzidine	212	106,196,180
2-Acetylaminofluorene	181	180,223,152
Dibenz(a,j)acridine	279	280,277,250
Hexachlorophene	196	198,209,21,406,408
Phenol-d6 (surrogate)	99	42,71
2-Fluorophenol (surrogate)	112	64
2,4,6-Tribromophenol (surrogate)	330	332,141
Nitrobenzene-d5 (surrogate)	82	128,54
2-Fluorobiphenyl (surrogate)	172	171
Terphenyl-d14 (surrogate)	244	122,212
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	68
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	94,80
Chrysene-d12 (istd.)	240	120,236
Perylene-d12 (istd.)	264	260,265

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in Table 7 after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

Table 8

RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100

Table 8

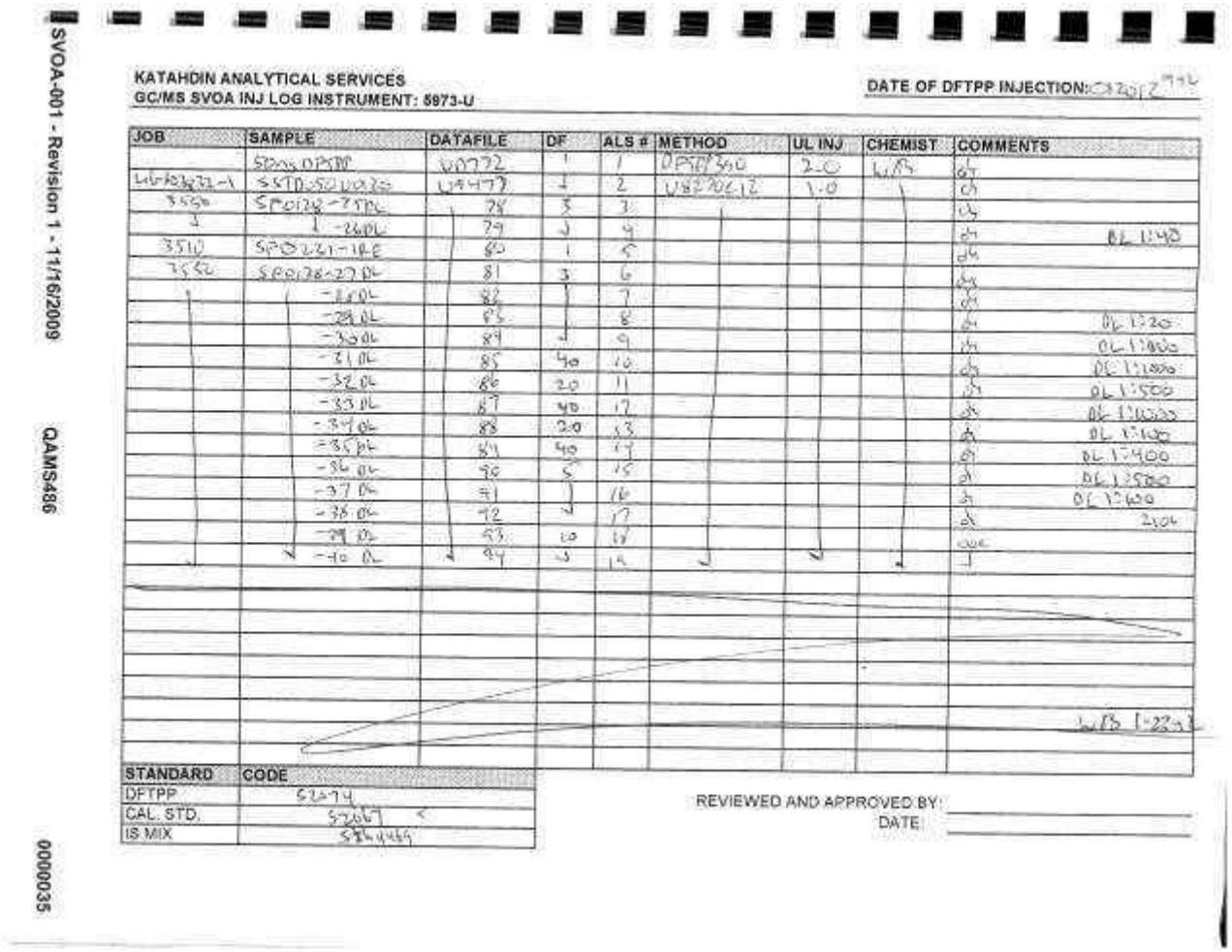
RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION (CONT.)

Semivolatile Compounds	Minimum Response Factor (RF)
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



SVOA-001 - Revision 1 - 11/16/2009

QAMS486

0000035

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-U

DATE OF DFTPP INJECTION:

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	SSTD50PSM	0772	1	1	DFTPP30	2.0	WB	
LL43632-1	SSTD50LL02	L4477	1	2	LL8270C-12	1.0		
3550	SP0128-7DL	78	5	3				
	-2DL	79	1	4				BL 1:40
3510	SP0221-1DL	80	1	5				
3550	SP0128-27DL	81	5	6				
	-1DL	82		7				
	-28DL	83		8				BL 1:20
	-30DL	84		9				BL 1:100
	-31DL	85	40	10				BL 1:1000
	-32DL	86	20	11				BL 1:500
	-33DL	87	40	12				BL 1:1000
	-34DL	88	20	13				BL 1:100
	-35DL	89	40	14				BL 1:400
	-36DL	90	5	15				BL 1:500
	-37DL	91	1	16				BL 1:400
	-38DL	92	1	17				2x4
	-39DL	93	10	18				DUP
	-40DL	94	1	19				
								WB 1-22-18

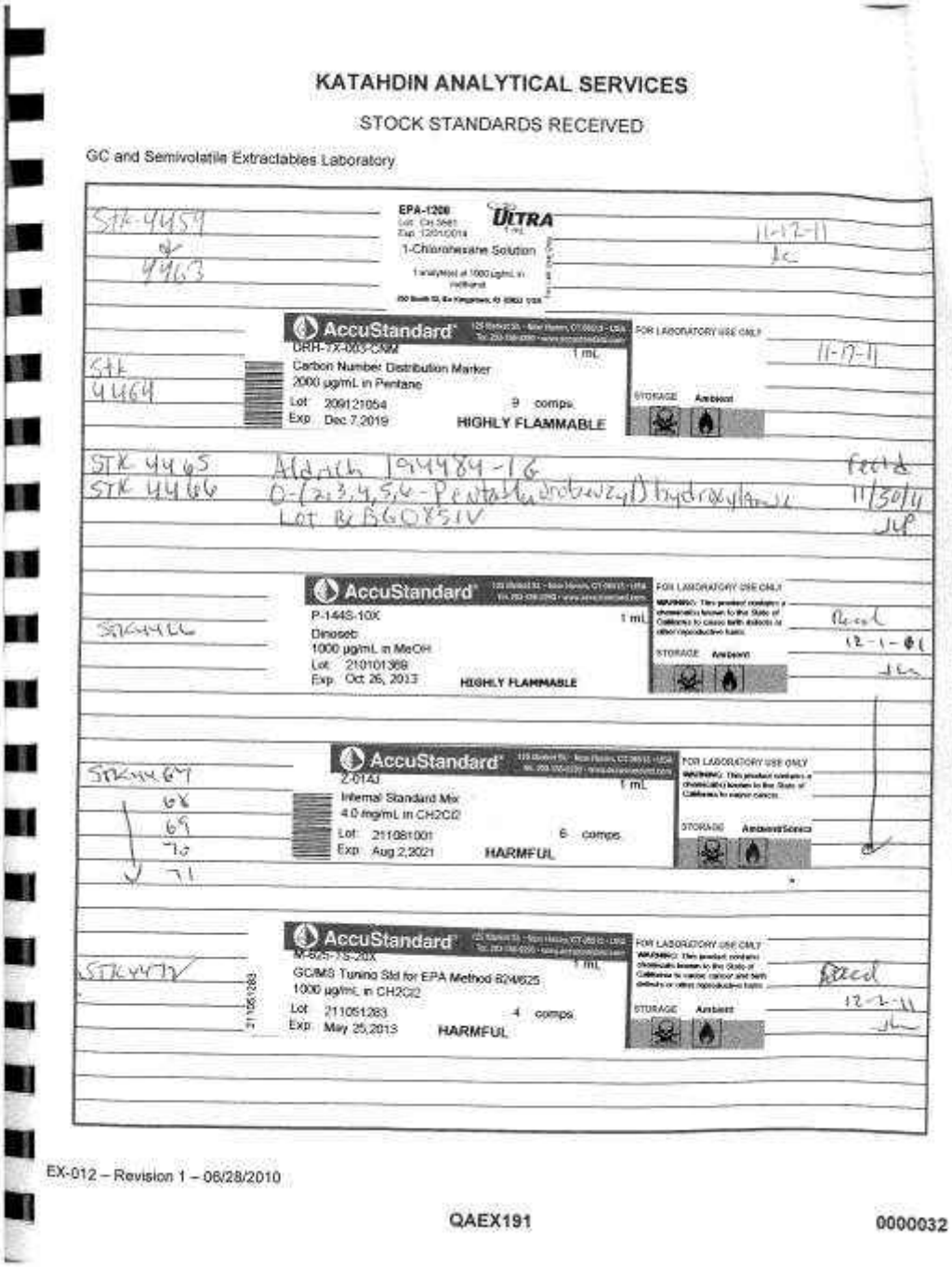
STANDARD	CODE
DFTPP	S2474
CAL. STD.	S2467
IS MIX	S2449

REVIEWED AND APPROVED BY:
DATE:

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

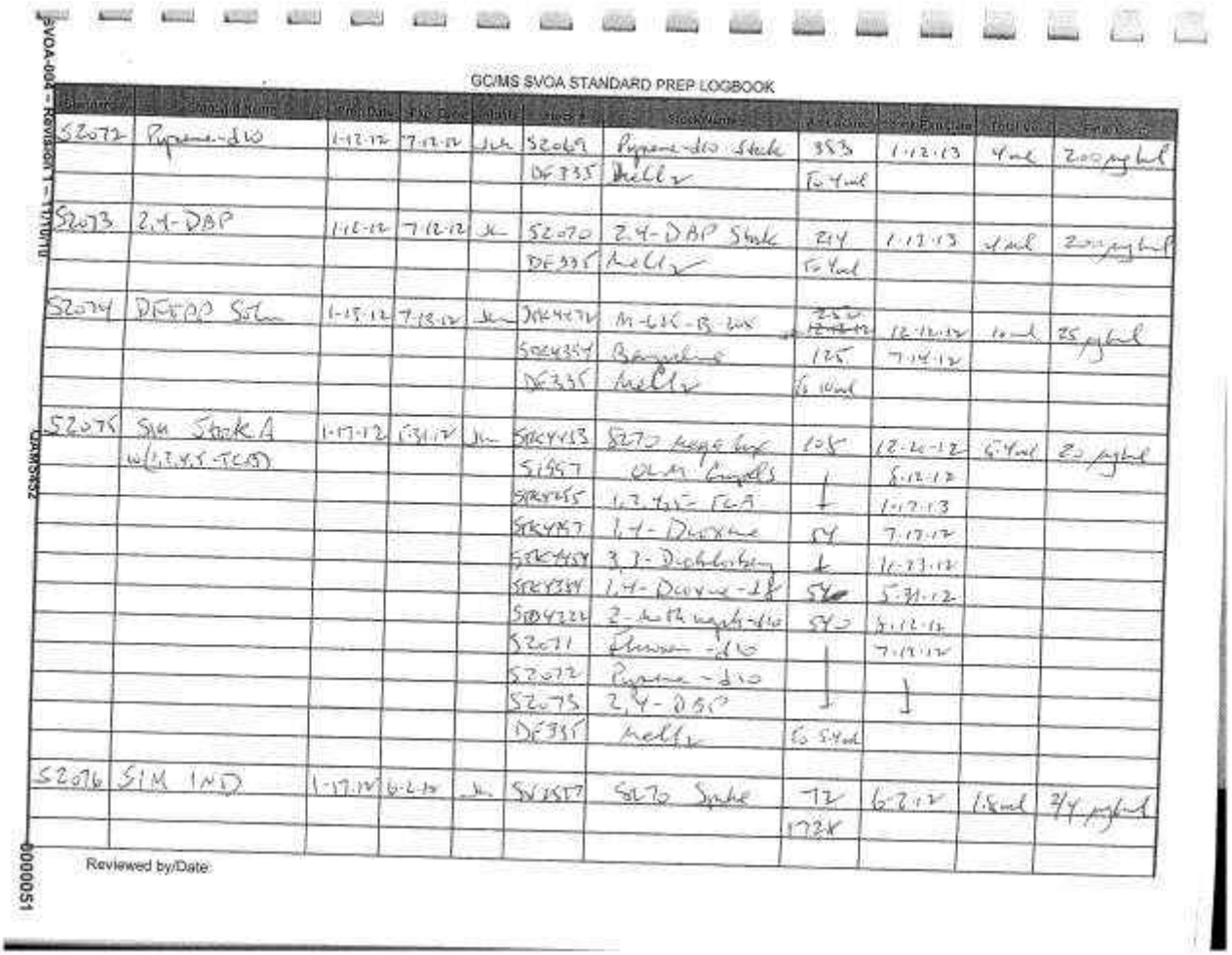


TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

SVOA-004 – Revision 1 – 11/10/10



GC/MS SVOA STANDARD PREP LOGBOOK

Standard #	Standard Name	Prep Date	Exp Date	Initials	Stock #	Stock Name	Lot Number	Exp Date	Total Vol	Final Conc
S2072	Pyrene-d10	1-12-12	7-12-12	JL	S2069	Pyrene-d10 Stock	383	1-12-13	4 ml	200 µg/ml
					DF335	Mell 2	To 4 ml			
S2073	2,4-DBP	1-12-12	7-12-12	JL	S2070	2,4-DBP Stock	214	1-12-13	4 ml	200 µg/ml
					DF335	Mell 2	To 4 ml			
S2074	DFTPP Soln	1-13-12	7-13-12	JL	STK4472	M-625-B-205	252 ~~12-12-12~~	12-12-12	10 ml	25 µg/ml
					STK4354	Benzidine	125	7-14-12		
					DF335	Mell 2	To 10 ml			
S2075	SIM Stock A	1-17-12	5-31-12	JL	STK4453	8270 Mega Lux	105	12-20-12	5.4 ml	20 µg/ml
	w/1,2,4,5-TCB				S1957	OLM Cupels	↓	8-12-12		
					STK4255	1,2,4,5-TCA	↓	1-17-13		
					STK4457	1,4-Dioxane	54	7-17-12		
					STK4454	3,3-Dichlorbenz	↓	11-23-12		
					STK4354	1,4-Dioxane-d8	54	5-31-12		
					STD4222	2-Methnaph-d10	54	8-12-12		
					S2071	Fluoren-d10	↓	7-12-12		
					S2072	Pyrene-d10	↓	↓		
					S2073	2,4-DBP	↓	↓		
					DF335	Mell 2	To 5.4 ml			
S2076	SIM IND	1-17-12	6-2-12	JL	SV2527	8270 Synthe	72	6-2-12	1.8 ml	2/4 µg/ml
							1728			

Reviewed by/Date:

0000051

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: J.C. Gomez

Review Date: 1-22-19

SOP Number: CA-226-10

SOP Title: Analysis of Semivolatile Organic Compounds by Capillary column GC/MS: SW 846 Method 8270D

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-24-19

QAO Signature: Leslie Dimond **Date:** 01.25.19

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-213
Revision History
Cover Page
Page 1

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: [signature] Date: 020101

Operations Manager: John C. Benton Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: [signature] Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 & Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2 Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited Section 7.5.1 - initial cal table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address different SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10
09	Sect. 7.4 - Added additional tune information. Sect. 7.6 - Added 100 ul minimum extract vol. & 1 ul IS is added for each 100 ul aliquot. Sect. 7.5.4 - Added RRT information Sect. 9.0 - Added MDL, LOD and LOQ information Table 4 - Added 1,4-Dioxane-d6 Surrogate	LAD	05/11	05/11
10	Sect. 7 - Changed sample volume from 1ul to 2ul. Sect. 8 - Added 10% rule for non-DoD clients. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added and updated references. Updated Figure 1. Added Addendum 1 - low level 1,4-Dioxane analysis	LAD	05/12	05/12
11	Sect. 1 and 7 - Removed Quick form reporting and added KIMS. Sect. 8 and Table 1 - Added the Surrogate 1,4-Dioxane-d8. Throughout - Fixed typos and made minor changes.	LAD	03/13	03/13
12	Sect. 4 - Updated instrument and column models. Sect. 7 - updated calibration levels and prep. Sect. 8 - Added marginal exceedance criteria. Updated ms/MSD acceptance criteria. Tables - Added DoD QSM 5.0 QC Requirements. Updated Fig. 2 & 3	LAD	04/14	04/14
13	Sect. 5 - Added Standards to title. Sect. 7 & Appendix 1 - Updated GC/MS operating conditions. Appendix 1 - Corrected 1,4 dioxane primary and secondary ions. Add 1,4 dioxane-d4 ions. Changed KAS INC to KAS throughout	LAD	03/16	03/16

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 10-Updated method references. Sect 7 -Added percent error calculation and relative std error calculation. Sect 1.1-updated definitions. Sect. 9.0-update. Sect. 7-changed the initial cal. std concentrations. Added compounds to Tables 5 & 7.	LAD	09/17	09/17
15	Sect. 7- updated SSTD30 preparation. Changed CCV to SSTD2.0 and concentration to 2.0ng/mL. Updated Runlog Example	LAD	01/19	01/19

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-15, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-15, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process. Refer to section 8 for Method Blank acceptance criteria

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term LOQ must be used for DoD work. Refer to section 9 for specific LOQ/LLOQ verification requirements

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer

to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890.

4.2 Mass Spectrometers (MS): HP5975 or HP5973

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: RTX5 SIL MS - 30m, 0.25mm I.D., 25um film thickness, columns (Restek) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS AND STANDARDS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 4. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will “boost” the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 4. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

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7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument is given a unique identifier)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (Each instrument is given a unique identifier). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (Each instrument is given a unique identifier).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	275°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using “Edit Sample Log Table”. For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no evidence of peak tailing. For clients requiring DOD criteria, the tailing factors for these two compounds should not exceed 2.

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 2.0, 7.0, 10.0 and 15.0 ng/uL. This is done to determine instrument sensitivity and the

linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds need to be calibrated at higher concentrations. A second standard (Mix B) is prepared containing these compounds. The two standards are combined as in the example below. The full aliquot is used and spiked with the appropriate amount of IS.

Example –

Calibration Mix A is prepared containing ALL analytes at 20 ng/ul.
Calibration Mix B is prepared containing only phenols and phthalates at 20 ng/ul.

For the low standard, 10 ul of mix A and 40 ul of mix B are combined and diluted to 1000 uL with MeCL2. Internal standards are then added prior to analysis.

Calibration Level	Cal-Mix A (All Analytes) Added (uL)	Cal-Mix B (Phenols and Phthalates) Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)	Final Conc. Everything but Phenols and Phthalates (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)
SSTD 0.2	10	40	950	1000	0.20	1.0
SSTD 0.50	25	75	900	1000	0.50	2.0
SSTD 2.0	40	50	310	400	2.0	4.5
SSTD 7.0	70	NA	130	200	7.0	7.0
SSTD 10	100	NA	100	200	10	10
SSTD 15	150	NA	50	200	15	15

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 3.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C hold 0.5 minutes 20°/min. to 260°C, hold 0.0 minutes 5°/min to 280°C, hold 0.0 minutes 18°/min to 300°C, hold 4.39 minutes
Final Column Temperature hold	300°C
Run Time	21 minutes
Scan Start Time	2.5 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the six calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.

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Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.

Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
$\acute{x}$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, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD2.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 2.0 ng/uL.

After quantitation of the 2.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 2.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may precede using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

For projects or clients requiring DoD QSM 4.1, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap.

This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for

analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.
If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

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8.2 Surrogate Recoveries

The five surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10, Pyrene-d10 and 1,4-Dioxane-d8) must meet the current statistically derived or nominal acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Non-Conformance Report (NCR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.

For projects or clients requiring DoD QSM compliance, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained

for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits or nominal limits with the following sporadic exceedance allowances, for DoD clients.

# of Analytes	# of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For non-DoD clients corrective action is only taken if greater than 10% of the analytes of interest are outside of the laboratory established acceptance limits.

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8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

All MS/MSD samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8270C and Method 8270D.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. % Error must be ≤ 30%. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	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 performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	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: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time 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.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (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. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	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 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 analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	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. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	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 with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-15	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4
Target and Surrogates:
1,4-Dioxane
1,4-Dioxane-d8 (surrogate)
Benzaldehyde
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
2-Methylphenol
3&4-Methylphenol
2,2'-Oxybis(1-chloropropane)
Nitrobenzene
Hexachloroethane
Acetophenone
N-nitroso-di-n-propylamine
1,3-dichlorobenzene
1,4-dichlorobenzene
1,2-dichlorbenzene
Internal Standard: Naphthalene-d8
Target and Surrogates:
Naphthalene
1-Methylnaphthalene (dredge)
2-Methylnaphthalene
2-Methylnaphthalene-D10 (surrogate)
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
4-Chloroaniline
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
1,2,4-trichlorobenzene
1,2,4,5-tetrachlorobenzene
Internal Standard: Acenaphthene-d10
Target and Surrogates:
1,1'-Biphenyl (dredge)
2,6 Dimethylnapthalene (dredge)
Acenaphthylene
Acenaphthene
Fluorene
2-Fluorene-d10 (surrogate)
2,4-Dibromophenol (surrogate)
2-Chloronaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
Dimethylphthalate
2,6-Dinitrotoluene
2,4-Dinitrotoluene
2,4-Dinitrophenol
2,3,4,6-Tetrachlorophenol
Diethylphthalate
4-Chlorophenyl-phenyl ether
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Dibenzofuran
4-Nitrophenol
Internal Standard: Phenanthrene-d10
Target and Surrogates:
Pentachlorophenol
1-Methylphenanthrene (dredge)
Phenanthrene
Hexachlorobenzene (special)
Anthracene
Fluoranthene
Carbazole
Di-n-butylphthalate
4-Bromophenyl-phenyl ether
Atrazine
Internal Standard: Chrysene-d12
Target and Surrogates:
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Pyrene
Benzo(a)Anthracene
Chrysene
Bis-(2-ethylhexyl)phthalate
Pyrene-d10 (surrogate)
Internal Standard: Perylene-d12
Target and Surrogates:
Perylene (dredge)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene (dredge)
Di-n-octylphthalate
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane-d8	96	66
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
1,3-dichlorobenzene	146	148, 111
1,4-dichlorobenzene	146	148, 111
1,2-dichlorobenzene	146	148, 111
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
1,2,4-trichlorobenzene	180	182, 145
1,2,4,5-tetrachlorobenzene	216	214, 179
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

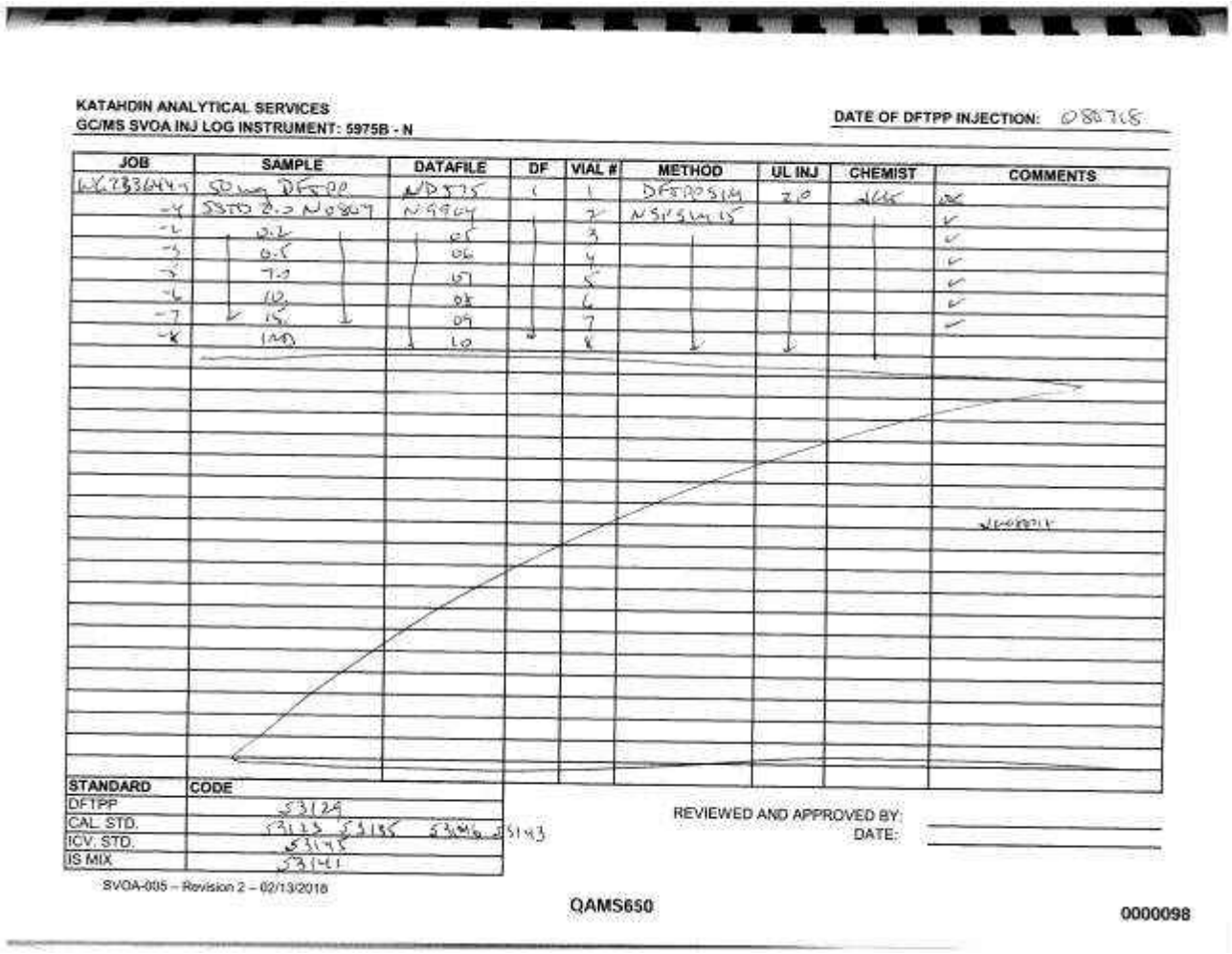
The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5975B - N

DATE OF DFTPP INJECTION: 080718

JOB	SAMPLE	DATAFILE	DF	VIAL #	METHOD	UL INJ	CHEMIST	COMMENTS
WG233644-1	50 ng DFTPP	ND575	1	1	DFTPP SIM	2.0	[illegible]	✓
-4	SSTD 0.2 N0809	N9964		2	NSPSIM15			✓
-2	0.2	05		3				✓
-3	0.5	06		4				✓
-5	7.0	07		5				✓
-6	10	08		6				✓
-7	15	09		7				✓
-8	1ND	10		8				

[illegible]

STANDARD	CODE
DFTPP	53129
CAL. STD.	53123 53135 53146 53143
ICV. STD.	53145
IS MIX	53141

REVIEWED AND APPROVED BY:
DATE:

SVOA-005 – Revision 2 – 02/13/2018

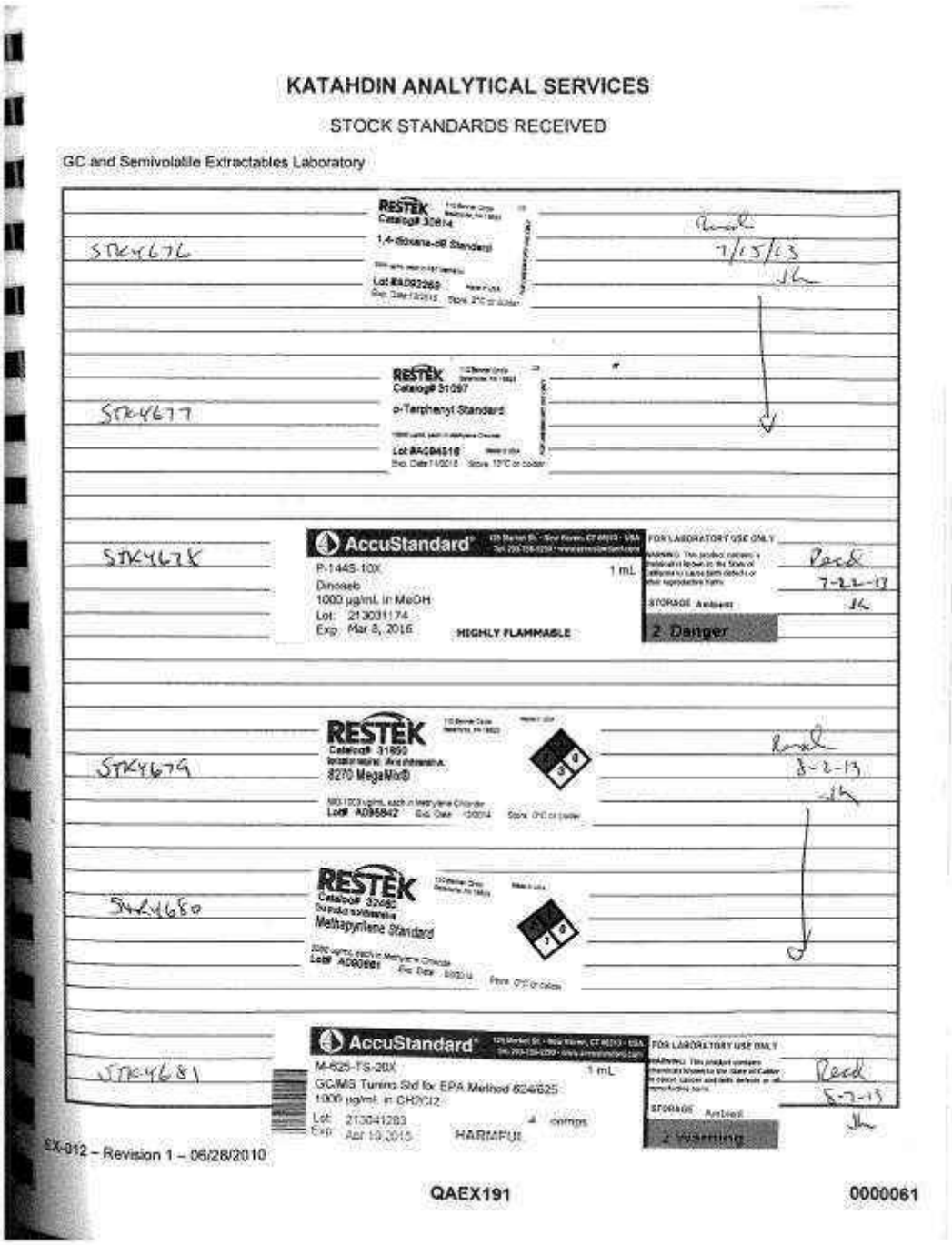
QAMS650

0000098

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GC and Semivolatile Extractables Laboratory

STK4676 — RESTEK Catalog# 32874 1,4-dioxane-d8 Standard — Lot #A092269 — Recd 7/15/13

STK4677 — RESTEK Catalog# 31097 o-Terphenyl Standard — Lot #A094516

STK4678 — AccuStandard P-144S-10X 1 mL Dinoseb 1000 µg/mL in MeOH Lot: 213031174 Exp: Mar 8, 2016 HIGHLY FLAMMABLE — FOR LABORATORY USE ONLY — STORAGE Ambient — 2 Danger — Recd 7-22-13

STK4679 — RESTEK Catalog# 31850 8270 MegaMix® — Lot# A095842 — Recd 8-2-13

STK4680 — RESTEK Catalog# 32460 Methapyrilene Standard — Lot# A090661

STK4681 — AccuStandard M-625-TS-20X 1 mL GC/MS Tuning Std for EPA Method 624/625 1000 µg/mL in CH2Cl2 Lot: 213041283 Exp: Apr 19, 2015 HARMFUL 4 comps — FOR LABORATORY USE ONLY — STORAGE Ambient — 2 Warning — Recd 8-7-13

EX-012 – Revision 1 – 06/28/2010

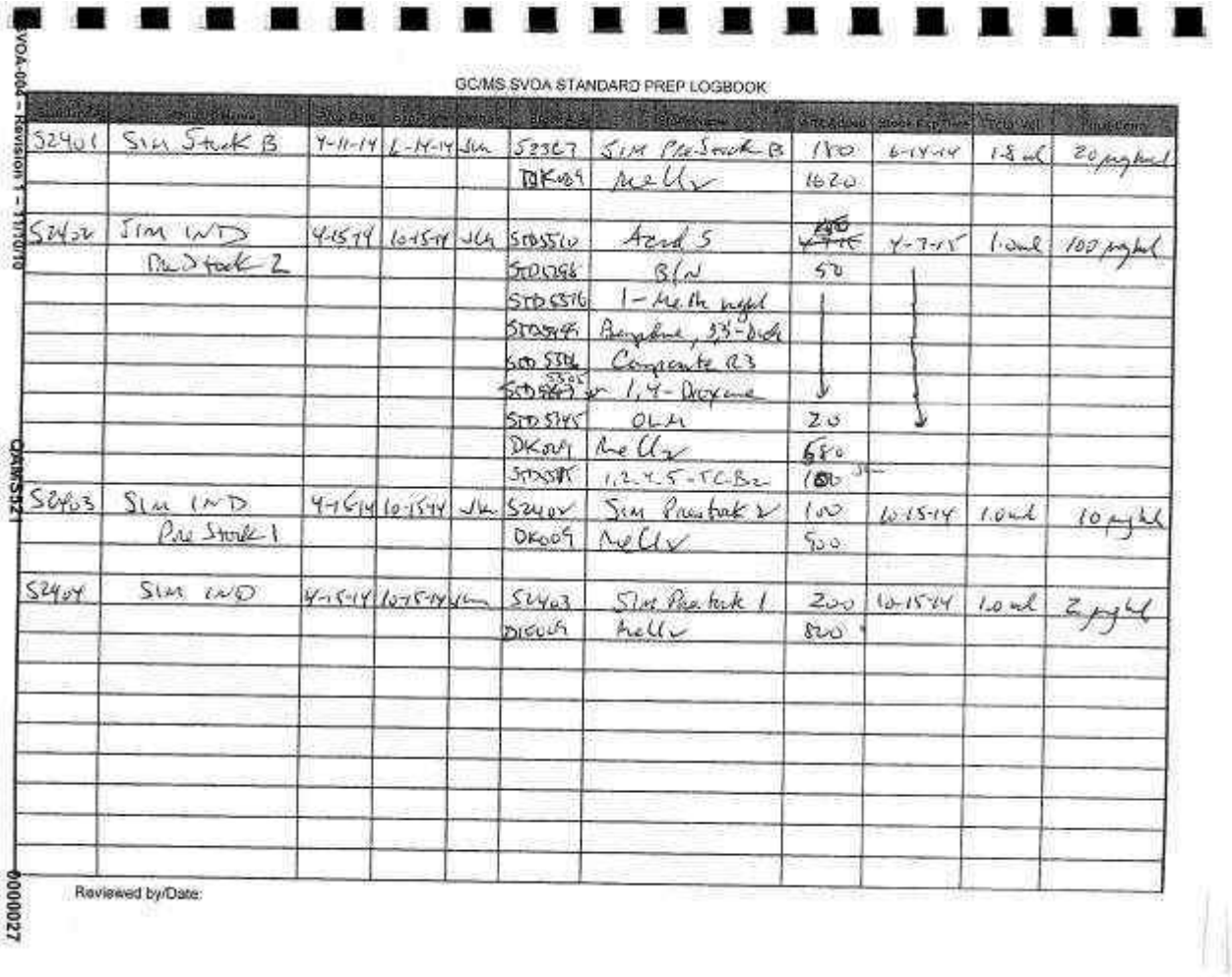
QAEX191

0000061

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY



SVOA-004 – Revision 1 – 11/10/10

GC/MS SVOA STANDARD PREP LOGBOOK

S2401	SIM Stock B	4-11-14	4-14-14	JLH	53367	SIM PreStock B	180	4-14-14	1.8 mL	20 µg/mL
					DK009	MeCl2	1620			
S2402	SIM IND PreStock 2	4-15-14	10-15-14	JLH	STD5510	Acenaphthene-d5	~~450~~ 500	4-7-15	1.0 mL	100 µg/mL
					STD5496	B/N	50			
					STD5516	1-Methylnaphthalene-d10				
					STD5495	Benzidine, 3,3'-Dichloro-				
					STD5514	Component B3				
					STD5497	1,4-Dioxane				
					STD5495	OLM	20			
					DK009	MeCl2	680			
					STD5517	1,2,4,5-TCB-d2	100			
S2403	SIM IND PreStock 1	4-16-14	10-15-14	JLH	S2402	SIM PreStock 2	100	10-15-14	1.0 mL	10 µg/mL
					DK009	MeCl2	500			
S2404	SIM IND	4-15-14	10-15-14	JLH	S2403	SIM PreStock 1	200	10-15-14	1.0 mL	2 µg/mL
					DK009	MeCl2	820			

Reviewed by/Date:

CA-MS-521

0000027

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

ADDENDUM 1

LOW LEVEL 1,4-DIOXANE ANALYSIS

The following are differences from the standard 8270 C or D SIM analysis:

GC Operating Conditions – The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP (Sect. 7.4) with the following exceptions:

Column Temperature Program	35°C hold 3 minutes 20°/min. to 300°C
Final Column Temperature hold	300°C
Run Time	16.25 minutes
Scan Start Time	2.3 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

Stock Standards – 1,4-Dioxane and 1,4-Dioxane each at 20 ug/mL

Calibration Standards – Use the above stock standards to prepare calibration standards at concentrations 0.25 ug/mL, 0.50 ug/mL, 1.0 ug/mL, 2.0 ug/mL, 4.0 ug/mL and 6.0 ug/mL. The 1.0 ug/mL is also the continuing calibration verification standard.

Sample analysis – Add 1 uL of internal standard (Section 5.3.2.4) aliquot of sample.

The ions for 1,4-Dioxane are 58 and 88.
The ions for 1,4-Dioxane-d8 are 64 and 96.

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.24.00	10/24/00
02	Addition of compounds to Figure 2.	DN	3.28.02	3.28.02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from < 39°C to < 30°C. Removed respirator reference. Added KAEHS manual. Added KAS SOP CA-108 reference for additional subsampling information.	LAD	02/09	02/09
08	Removed targeting sample weights. Added KAT SOP SD-902 reference. Updated logbook page example. Added GPC cleanup is required for all samples. Removed decanting samples	LAD	08/10	08/10
09	Minor modifications made to sections 5 & 7 to reflect current practices. Updated Section 9, to include LOD/LOQ requirements. Changed 7.6 & 7.7 to add surrogate and spikes after sodium sulfate is added. Updated references in Section 10.	LAD	04/12	04/12
10	Sect. 5 - Updated surr. prep. for SIM and Scan surr. - now in 1 mix. Sect. 7- updated spiking info. for SIM/Scan surr. mix. Clarified decanting samples. Sect. 10- Added & updated references. Updated fig. 1.	LAD	06/14	06/14
11	Sect. 4 and 7 - Updated for new sonicator. Changed KAS INC to KAS throughout.	LAD	08/15	08/15
12	Title changes for sections 1.4 + 5.0. Updated method references for NELAC, DOD + SW846. Removed old sonicater parameters. Removed a duplicate paragraph. Clarified sample weight.	LAD	09/17	09/17
13	Sect. 7- updated method Blank and LCS initial weight criteria. Sect. 10- updated references. Updated Logbook example	LAD	11/18	11/18

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Added use of tongue depressors. Corrected typographical errors.	CAD	04/19	04/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-14**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-512-14**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550C in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the

current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

An 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Pasteur pipets - disposable, 5 ¾ ".

4.3 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.4 Sonicator ultrasonic processor XL – QSonica Model Q500 (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.5 Vacuum filtration flask - 500 mL Erlenmeyer

4.6 Filter paper, 70 mm, Whatman #4

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Spatula - stainless steel

4.10 Wooden Tongue Depressors

4.11 Balance - capable of accurately weighing ± 0.01 g.

4.12 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent).

4.13 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.14 Powder funnels, 100 mm diameter, 35 mm stem

4.15 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.16 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.17 Nitrogen evaporation apparatus.

4.18 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

4.19 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS^{TM} with internal UV detection

5.0 REAGENTS AND STANDARDS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 200 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 SCAN/SIM Surrogate Spiking Solution - A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution -

Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.6 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.7 Base/Neutral and Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2.0 ug/mL for base/neutral and 4.0 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs

- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Sonicator horns tuned
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples should be cleaned using gel permeation chromatography (GPC) to reduce matrix interferences.

The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP SD-902, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples. Fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Do not decant any water on the sediment sample.

Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2 Mix with a stainless steel spatula or wooden tongue depressor to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique “, for more detailed guidance on subsampling to ensure reproducibility.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out an approximate, greater than 30 g portion of sample into a

labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or wooden tongue depressor. Keep the spatula or wooden tongue depressor in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 $\pm$ 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the SCAN/SIM surrogate spiking solution using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **after** the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution. **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.8 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **after** the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.8.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.8.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.8.3 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.9 Turn sonicator on

7.9.1 Set pulse timer to 1 ½ minutes.

7.9.2 Set pulse to 1 and 1. This sets the sonicator to pulse for 1 second and rest for 1 second, with the timer set for 1 ½ minutes this equals a 3 minute total sonication

7.9.3 Set amplitude to 40%. Record this in the logbook.

7.9.4 These settings are stored in the unit and do not have to be entered with each use.

7.9.5 When done sonicating turn off the unit.

7.9.6 Refer to the Operating Manual for further information.

7.10 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.11 To the mixed and spiked blank and LCS, add approximately 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.12 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula or wooden tongue depressor to loosen up the mixture prior to extracting.

Rinse the spatula or wooden tongue depressor with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.13 Sonicate for 3 minutes. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written in the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using approximately 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula or wooden tongue depressor. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 39°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.23 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.24 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.25 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.
Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3550C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The 2009 TNI Standards

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

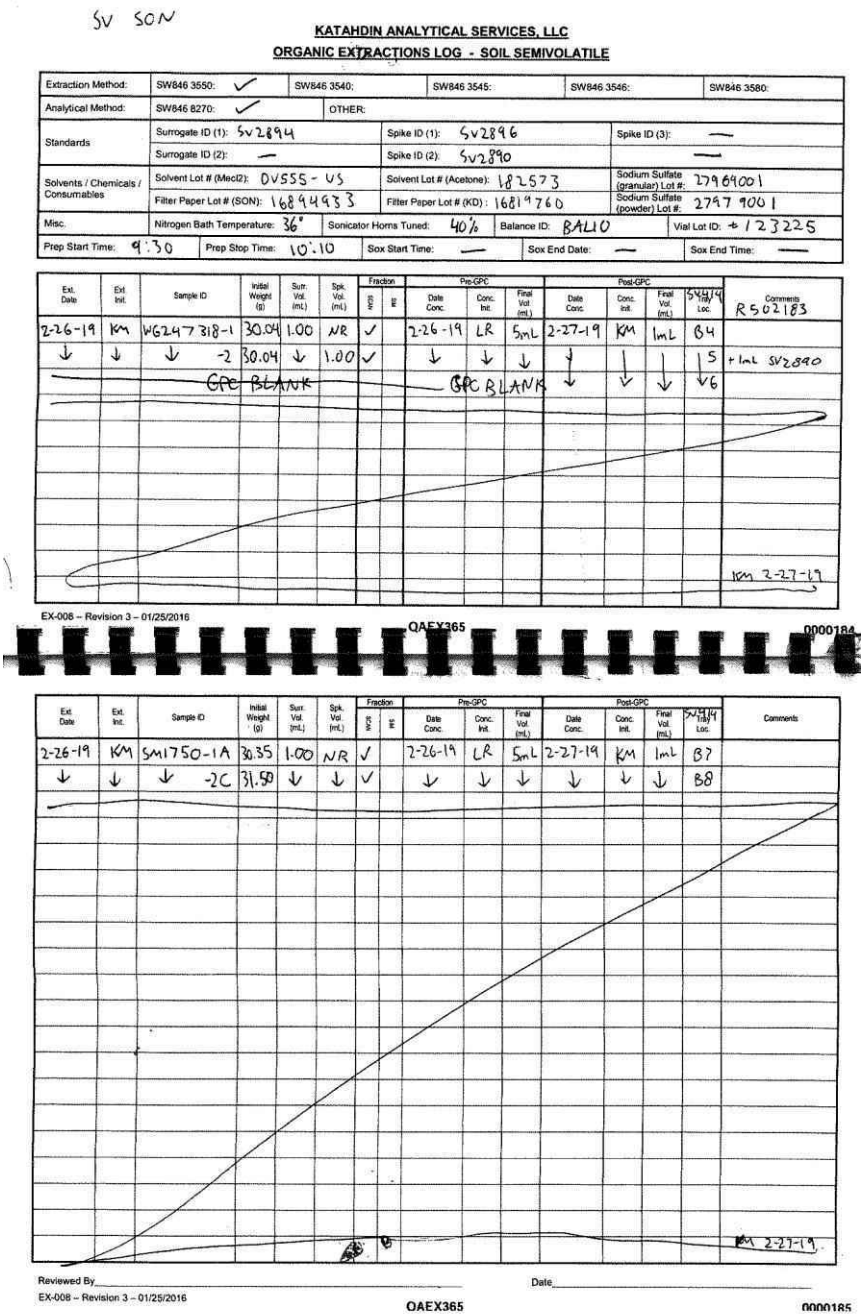
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-13	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



SV SON

KATAHDIN ANALYTICAL SERVICES, LLC
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method:	SW846 3550: ✓	SW846 3540:	SW846 3545:	SW846 3546:	SW846 3580:
Analytical Method:	SW846 8270: ✓	OTHER:			
Standards	Surrogate ID (1): SV2894		Spike ID (1): SV2896	Spike ID (3): —	
	Surrogate ID (2): —		Spike ID (2): SV2890	—	
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): DV555-US		Solvent Lot # (Acetone): 182573	Sodium Sulfate (granular) Lot #: 27969001	
	Filter Paper Lot # (SON): 16894933		Filter Paper Lot # (KD): 16819760	Sodium Sulfate (powder) Lot #: 2797 9001	
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Horns Tuned: 40%	Balance ID: BAL10	Vial Lot ID: ⊕ 123225	
Prep Start Time: 9:30	Prep Stop Time: 10:10	Sox Start Time: —	Sox End Date: —	Sox End Time: —	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction SV/S	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	SVHW Tray Loc.	Comments
2-26-19	KM	WG247318-1	30.04	1.00	NR	✓		2-26-19	LR	5mL	2-27-19	KM	1mL	B4	R502183
↓	↓	↓ -2	30.04	↓	1.00	✓		↓	↓	↓	↓	↓	↓	5 V6	+ 1mL SV2890
		GPC BLANK						GPC BLANK							

KM 2-27-19

EX-008 – Revision 3 – 01/25/2016 OAEX365 0000184

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction SV/S	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	SVHW Tray Loc.	Comments
2-26-19	KM	SM1750-1A	30.35	1.00	NR	✓		2-26-19	LR	5mL	2-27-19	KM	1mL	B7	
↓	↓	↓ -2C	31.50	↓	↓	✓		↓	↓	↓	↓	↓	↓	B8	

KM 2-27-19

Reviewed By_____ Date_____

EX-008 – Revision 3 – 01/25/2016 OAEX365 0000185

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to Sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. Changed CLLE extraction time to 18 → 24 hours. Added information on determining initial Sample volume. Added extracted Sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥ 11. If not add more NaOH and continue extracting. Added information for initial Volume determination. Added Reference to CA-109. Updated logbook example. Added if extract goes dry - reextract.	LAD	10/09	10/09
07	Sect. 5 - Removed baking and rinsing $NaSO_4$. Added 1,4-Dioxane to SIM Surrogate Mix. Sect. 7 added acid to B/N SIM., removed to let separate for 10 minutes minor edits throughout.	LAD	03/12	03/12

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08	Removed Sect. 7.1.9, determining the sample initial volume. Sect. 7.1.4 has this information. Figures 1 and 2 updated.	LAD	05/13	05/13
09	Sect. 5 - Updated prep of SIM/scan Surrogate mix. Sect. 7 - Updated Surrogate spiking directions. Updated Figure 1.	LAD	06/14	06/14
10	Sect. 7 - For separatory funnel, corrected extraction sequence (acid then basic), and that the pH is determined after first shake. Updated Solvent Lot Check Form. Changed KAS INC → KAS	LAD	08/15	08/15
11	Change order of pH checking and spike stds addition. Replace use of HCL with H_2SO_4 to adjust pH. Added test for residual chlorine. Updated method references for NELAC, DOD + SW 846.	LAD	09/17	09/17
12	Sect. 1 - Corrected order of pH extraction. Sect 7 - Updated for current practice. Sect. 10 - Updated references. Updated logbook page example.	LAD	03/19	03/19

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-502-12**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste

stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by separatory funnel and CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a separatory funnel or a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS AND STANDARDS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, certified by the manufacturer/vendor as purified.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) – Prepared in an icebath by slowly adding a volume of concentrated H_2SO_4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC and/or GC/MS analysis.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. SCAN/SIM Surrogate Spiking Solution – A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	20 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.
Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.3 Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutrals and 4.0 ug/mL for acids. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem.

5.5.4 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Potassium iodide starch paper

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

Follow the proper procedures for maintaining Internal Chain of Custodies for samples when removing and replacing samples in storage locations. This procedure is described in KAS SOP SD-902, “Sample Receipt and Internal Control”, current revision.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order

consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS).

7.1.3.1 To prepare method blank, add 1 L reagent water to a sample bottle. Pour this into the CLLE body. Be sure that no water leaks into the round bottom flask. Repeat for the LCS.

7.1.3.2 If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis.

7.1.3.3 This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 The initial volume of a sample is determined by comparing the meniscus of the sample to a reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.5.1 Transfer the sample to a CLLE body slowly, being sure that no water leaks into the round bottom flask.

7.1.5 To prepare a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL SCAN/SIM surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

REMEMBER: If both SCAN and SIM analysis are required, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.1.7 To LCS/LCSD and MS/MSD add 1.0 mL of the appropriate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.7.1 For SVOA Scan Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2).

7.1.7.2 For SIM Analysis - add 1.0 mL of Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.3).

7.1.7.3 For SVOA Appendix IX Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2) and 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.4).

7.1.8 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.1.9 For each blank, LCS, MS and sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.10 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.11 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.12 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.13 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.14 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.11 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered with foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the “N-Hi” satellite.

7.1.12 Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples may be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent.

7.2.3.1 To prepare method blank, add 1 L reagent water to a sample bottle. Pour this into the separatory funnel. Repeat for the LCS.

7.2.3.2 If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis.

7.2.3.3 The blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL SCAN/SIM surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use. (sect. 5.5.2). **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL of the appropriate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 For SVOA Scan Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2).

7.2.7.2 For SIM Analysis - add 1.0 mL of Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.3).

7.2.7.3 For SVOA Appendix IX Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2) and 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.4).

7.2.8 For each blank, LCS, MS and sample, rinse the original sample container, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH ≤ 2 with 1:1 H_2SO_4 after addition of surrogates and spikes. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.2.10 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes.

7.2.11 After the first shake dip a glass stirring rod into the sample and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.2.12 Allow phases to separate. Drain the methylene chloride layer into an amber collection bottle.

7.2.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time. Collect the methylene chloride layer in the same amber collection bottle.

7.2.15 Repeat the extraction for a third time. Collect the methylene chloride layer in the same amber collection bottle.

7.2.16 Following the third shake, adjust the pH to $\geq$ 11 with 10N NaOH. Add enough 10N NaOH to adjust the pH to $\geq$ 11.

7.2.17 Add 60 mL methylene chloride to each separatory funnel and extract the samples in the same manner described in 7.2.11 – 7.2.14. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.18 After the first shake dip a glass stirring rod into the sample and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be ≥ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.19 Repeat 1 more time with 1 more 60 mL aliquot of methylene chloride. Collect the methylene chloride layer in the same amber collection bottle

7.2.20 Sample waste should be poured into the “N-Hi” satellite.

7.2.21 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

7.3.1 For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.2 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts. Record the lot numbers for filter paper, sodium sulfate crystals and methylene chloride in the extractions logbook.

7.3.3 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to

complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.4 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.5 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.6 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.7 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.8 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.9 Transfer all of the extract to a 1.8 mL screw cap vial. Using methylene chloride, adjust the final volume of each extract to 1 mL by comparison to an appropriate reference vial.

Store in refrigerator until GC/MS analysis.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:
- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:
- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Methods 3510 and 3520, current revisions.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of Semivolatiles Logbook Page
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Figure 3 LCS/Matrix Spike Component List
Figure 4 Appendix IX LCS/Matrix Spike Component List

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-12	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-12	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 $\pm$ 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE

S/S
SEP

KATAHDIN ANALYTICAL SERVICES, LLC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3510 (SEP) ✓	SW846 3520 (CLLE)	SW846 3535 (SPE)
Analytical Method:(check one)	SW846 8270 ✓	SW846 8270 SIM ✓	EPA 625 (mark meniscus w/ marker to determine IV)
Surrogate ID: SV2885	Surrogate ID:	Spike ID: SV2869 SV	Spike ID: SV2887 SIM
Methylene Chloride Lot #: DN249-US	pH Paper Lot # HC857466	KI Starch Paper ID: 082117	Note samples requiring TRC neutralization in comments section.
pH (1st Extraction) ≤2	H_2SO_4 Lot # RE0334	pH (2nd Extraction) ≥11	NaOH Lot # 184664
$NaSO_4$ Lot # RE0341	Filter Paper Lot #: 1684 3524	Boiling Stones ID: 3-27-2014	Vial Lot #: 403039
Nitrogen Bath Temperature: 36°C			
Prep Start Time: 9:00	Prep End Time: 11:30	CLLE Start Time: —	CLLE End Date & Time: —

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location SV/SIM	Initials	Comments
12-26-18	AC	WG1243501-1 WG1243502-1	1000	1mL	NR	✓	✓	1mL	12-26-18	A7	JMS	SV- R497250
		WG1243501-2	↓	↓	1mL	✓		↓	↓	2	↓	SIM- R497251
		-3	1060		↓	✓				3		MS TL2690-1F1
		-4	1060			✓				4		MSD -1I1
		-5	200		NR	✓				5		diluted to 1000 PBT Blank 1498
↓	↓	WG1243502-2	1000	↓	1mL	↓	✓	↓	↓	6	↓	

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location SV/SIM	Initials	Comments
12-26-18	AC	TL2607-1	200	1mL	NR	✓		1mL	12-26-18	A7	JMS	TCLP diluted to 1000
		TL2659-1	↓	↓	↓	↓		↓	↓	8	↓	↓
		-2								9		
		-3								10		
		TL2661-1								B1		
		TL2690-1 B1	1060							2		ms/msd
		-2K	1030							3		
		-3L	1050							4		
		-4J	1060							5		
		-5I	1060							6		side by
		-6K	1060							7		
↓	↓	TL2691-27A	1030	↓	↓	↓	✓	↓	↓	8	↓	

Reviewed By _____ Date _____

EX-007 – Revision 5 – 11/08/2017 QAEX367 0000117

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT LOT CHECK LOGBOOK

KATAHDIN ANALYTICAL SERVICES

SOLVENT LOT CHECK

SOLVENT:_____

LOT#:_____

DATE RECEIVED:_____

DATE CONCENTRATED:_____

CONCENTRATED BY:_____

PREP METHOD:_____

TRAY LOCATION:_____

ANALYZED BY:_____

PASS/FAIL:_____

EX-027 – Revision 1 – 08/15/2015

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

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QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Duncan C. Hughes Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DN	1.22.01	1/22/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DN	5.23.01	5.23.01
03 8082	Changed to practice of reporting higher value. Other minor changes to sections 7.5.2, 7.7.3 & to Table 2.	DN	5.21.02	5.21.02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included Target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New Checklist added wording to sect. 8	LAD	020305	020305

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06 8082	Changed PCB 1260 to Aroclor 1260. Removed references to 3541. Updated table 2. Added instructions to shake extract before vialing	LAD	04/06	04/06
07	Added waste streams to sect. 1.0. Added ICV to definitions, sect. 5, sect. 7 and Table 1. Added wording regarding 2nd column confirmation criteria and flagging rules to sect. 7.7.4. Added CCV criteria to sect. 7.5.3 and Table 1. Added wording regarding MI to sect. 7.7.3	LAD	08/07	08/07
08	Added tissue, wipe and oil matrices. Added extraction method 3535. Added DDT analog interference, Std. information and analysis frequency criteria. Added HTs are a recommendation. Added note that 2 detectors must be used for dual column. Updated method references. Removed calibration and surrogate method mod. from Table 2. Added more info to linear calib. Added extraction references,	LAD	02/09	02/09
09	Added Chemstation to definitions. Clarified that Surrogates are added to only the aroclor 1660 standards, NOT ALL standards.	LAD	05/09	05/09
10	Revised sections 7, 8, and 10 to reflect compliance with the DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM Ver. 4.1 QC criteria. Minor changes to Table 1.	LAD	04/10	04/10
12	Removed Sect. 4.5 - Analytical balance. Removed Sect. 5.2.4 - DDT analog standard. Removed Sect. 7.5 - DDT analog standard analysis requirement. Table 1 - Added aver. cal criteria and corrected CCV & LCS acceptance criteria. Added and removed references to Sect. 10. Updated Figure 2 - data review checklist. Added PCBs 1262 & 1268	LAD	07/11	07/11
13	Added Extraction method 3546. Removed Quickforms references. Added reporting from KIMS. Updated Figures 1 and 2.	LAD	02/13	02/13

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 7 - Added additional information for failing CCVs. Sect. 10 – Updated references. Added Table 3 – DoD QSM 5.0 QC Requirements.	LAD	08/14	08/14
15	Sect. 5 - Added Standards to title. Sect. 7 - Added ICV requirements for all PCBs. Changed 1248 calibration from 6-point to single point. Fixed typos. Reworded Sect. 7.5.3 for clarification. Sect. 8 - Added MDL/LOD/LOQ information. Updated Fig. 2.	LAD	03/16	03/16
16	Sect. 7 - Added %D Error calculation. Sect. 8 - Added LLOQ reference	LAD	03/17	03/17
17	Sect. 1, 8, 9 and Table 1 - Added LLOQ definition and acceptance criteria. Clarified PQL, LOQ and LLOQ. Updated CCV acceptance criteria. Updated logbook page and data review checklist	LAD	06/17	06/17
18	Sect 7 - Added % Difference and % Drift calculations. Sect. 8 - Added continuing calibration plan. Sect. 10 - Updated [illegible] references. Removed QSM 4.2 QC [illegible].	LAD	12/17	12/17
19	Sect. 1 - Updated definitions. Sect 2 - Added GPC Cleanup. Sect. 7 - Edited for clarification and to reflect current practices. Minor edits throughout to reflect current practice and correct typographical errors	LAD	01/19	01/19

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-329-19** titled **ANALYSIS OF AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

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Recipient: _____ Date: _____

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of aqueous, solid, tissue, wipe and oil samples for PCBs by EPA Method 8082A as performed by Katahdin Analytical Service including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1016 (AR1016), Aroclor-1221 (AR1221), Aroclor-1232 (AR1232), Aroclor-1242 (AR1242), Aroclor-1248 (AR1248), Aroclor-1254 (AR1254), Aroclor-1260 (AR1260), Aroclor-1262 (AR1262) and Aroclor-1268 (AR1268). Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD).

1.1. Definitions

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term LOQ must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

INDEPENDENT CALIBRATION VERIFICATION STANDARD (ICV): A solution prepared from a stock standard solution independent of the calibration mix that is used to verify the calibration.

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ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, samples and spiked samples prior to analysis. They are also included in the AR1660 standard used for calibration and the calibration verification standard. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

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1.2 Responsibilities

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

1.2.2 It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

1.2.3 It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

1.4.1 Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of

the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

1.4.2 Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

1.4.3 Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the PCB Vial Waste (H).

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3640A – Gel Permeation Chromatography (GPC) cleanup, Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

Compounds from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides including the DDT series.

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4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD). Note: Two detectors must be employed when using dual columns.

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Refrigerator for storage of extracts and standards.

5.0 REAGENTS AND STANDARDS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in

standards prep logbook. A separate standard is used for each Aroclor. The concentrations of the working calibration standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The analytes AR1016, AR1260 and the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) are combined into one mixture called AR1660. AR1016 and AR1260 are prepared at the aroclor concentrations above, while the surrogates are at the respective concentrations 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

5.2.3 Independent Calibration Verification standard (ICV): Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentration of the ICV PCB standard is 1.0 ug/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 while solid samples use methods 3540, 3546 or 3550. Tissue samples are extracted using method 3540 or 3550. Wipes and oils are generally extracted using method 3580.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 ml/min Helium, Ar/Methane or Nitrogen
Column flow: 6 ml/min
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 160(0) - 5/min - 260(10)
Run time: 30 min
Injection size: 2 ul

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7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of AR1660, AR1242, and AR1254 are prepared and routinely analyzed.

Six-point calibration standards of AR1221, AR1232, AR1248, AR1262 and AR1268 are also prepared and routinely analyzed on the primary PCB instrument. At a minimum, a single point calibration standard is analyzed for these Aroclors. If using a single point and the Aroclor is required for a project and is detected in a sample, then the GC would be calibrated for the Aroclor and the samples would be reanalyzed.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Five characteristic peaks from each Aroclor are used to generate the calibration curve. AR1221 is the exception where only 2 peaks are used. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the five peaks can be prepared in Target using the peak height against the concentration of the standard.

7.3.1.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

Where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.3.1.2 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.3.1.4 A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990.

The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for work originating in their state.

7.3.2.3 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in 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, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

7.3.3 All six point calibration curves (AR1660, AR1242 AR1454 and if any other Aroclor is required) must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.3.4 The working calibration curve must be verified prior to sample analysis and every 10 samples thereafter by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 20%, reanalyze all samples since the last successful calibration verification. A new calibration curve may need to be prepared for that analyte.

The average result for 5 (2 for AR1221) peak heights of the Aroclors is used for quantitation.

For clients or projects requiring DoD QSM, current version, compliance, if the CCV fails the above criteria and reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ±

0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 Gas chromatographic analysis

7.5.1 Shake samples and let them sit for one minute before vialing for analysis.

7.5.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes. The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

7.5.3 Samples are analyzed in a batch referred to as an analytical sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by sample extracts interspersed with mid-concentration calibration standards.

When an Initial Calibration is not performed, the calibration must be verified with a Calibration Verification Standard (CV) prior to sample analysis. The calibration for each Aroclor detected in the subsequent analysis must be verified. Typically, for samples whose PCB composition is unknown, CVs for AR1660, AR1242 and AR1254 are analyzed. At a minimum, the AR1660 calibration must be verified because this calibration includes the surrogates.

A CCV must be analyzed at the beginning of every 12-hour clock, after every 10 samples and at the end of the analytical sequence. The CCV concentration should alternate between 1.0 ug/mL and 0.25 ug/mL standards.

The calculated Aroclor concentration in the CV must not exceed a difference (or drift) of ± 20%.

$$\% \text{ Drift} = \frac{\text{Measured Amount} * 100}{\text{True Amount}}$$

Where: Measured Amount = concentration determined by the calibration
True Amount = concentration of the analyte ion the standard

$$\% \text{ Difference} = \frac{CF_v - CF_{Mean} * 100}{CF_{Mean}}$$

Where: CF_v = calibration factor calculated for the calibration verification standard.
CF_{Mean} = mean calibration factor form the initial calibration

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If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected.

If reanalysis cannot be performed, data must be qualified and explained in a narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.5.1.1 If a CV analyzed before or after a batch of samples has a greater than 120% recovery and the analyte was not detected in the associated samples, then reanalysis may not be necessary.

7.5.1.2 If a CV analyzed after a batch of samples has a less than 80% or greater than 120% recovery and the analyte was detected in the associated samples, then reanalysis is necessary to ensure accurate quantitation.

7.5.1.3 If a CV analyzed after a batch of samples has a greater than 120% recovery and the analyte was not detected in the associated samples, then reanlaysis is necessary to ensure that the detector response had not deteriorated to the point that the analyte would not have been detected even though it was present. However, in some cases, where obvious sample matrix carry over is affecting the closing CV, the data may be reported with narration.

7.5.2 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.5.3 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.5.3.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the

height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.5.3.2 Five Aroclor (two for AR1221) concentrations are calculated using the peak heights of the characteristic peaks of the Aroclor. These concentrations are then averaged to determine the concentration of that Aroclor.

7.5.4 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.5.5 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665). **Note:** Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.5.6 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.6 Calculations

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in section 7.5.4.1.1. However, if a sample contains more than one Aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = (C) (Vt)/ (Vs)

Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/ (Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

All recoveries must meet the most recent laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or

a matrix effect, the data is narrated. If the surrogate recoveries are low and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.

For DoD QSM (current version) use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.
Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 “Manual Integration on GC/MS, GC, HPLC and IC Datasystems” for more information.

7.7.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged and narrated. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. In some cases a non-confirming analyte may be reported. In these cases the analyte must be Q-flagged and narrated...

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV. The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

If reporting data that has an RPD that is >40%, the data must be flagged with a “J” indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the other column. In this scenario, the result would be reported from one column and need to be “Q” flagged to indicate that it was not confirmed on a second column.

7.7.5 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is

provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. The DoD QSM states that a method blank is considered to be contaiminated if the concentration of any analytein the blank exceeds ½ of the LOQ and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.3 LCS, MS/MSD and Surrogate Spike Concentrations and Corrective Actions:

8.3.1 The LCS and the MS/MSD are spiked at the same concentration with AR1660. The spike concentrations are:

Compound	WATER ug/L	SOILS mg/kg
AR1660	5.0	0.17

8.3.2 The surrogate spike concentrations in the final extract are:

Compound	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

8.3.3 LCS and MS/MSD acceptance criteria and Corrective Action:

All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM (current version), use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, 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 narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM (current version), apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be </= 30% between MS and MSD.

8.3.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.4 Non-conformance Report: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report (NCR) must be initiated as soon as possible to document resolution.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target

variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8082A.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8000D

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

Katahdin Analytical Services, SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin Analytical Services, SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES

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Table 2 DoD QSM Version 5.0 QC Requirements
Table 3 Summary of Method Modifications
Figure 1 Instrument Run Log
Figure 2 Review Checklist
Figure 3 PQLs

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6pt calibration of Aroclor 1660, 1242, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	Average Model – at least 5 points, % RSD </= 20% Linear Model – at least 5 points, correlation coefficient (r) ≥ 0.995 Quadratic Model – at least 6 pt calibration, coefficient of determination (r^2) ≥ 0.990 % Error must be ≤ 30% or RSE must be ≤ 20%	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample,5 or 6-pt calibration (depending on calibration model) of identified compound with reanalysis of sample.
Independent Calibration Verification	Immediately following calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock.
CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	± 20 % D	(1) Evaluate the samples: If the %D >+20% and sample results are <PQL, narrate. (2) If %D >±20% only on one channel, narrate. If %D >±20% for closing CV, and is likely a result of matrix interference, narrate. (3) Otherwise, reanalyze all samples back to last acceptable CV.
Method blank	One per prep batch	No analyte detected >PQL / LLOQ	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. (3) Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Laboratory statistically derived limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are <PQL, narrate. (5) Otherwise, reprep a blank, QC and the remaining samples.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.
Sample Duplicate	One sample duplicate per ten samples if requested	RPD ≤20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	(1) Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	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 >/= 0.995; Option 3: non-linear least squares regression (quadratic) for each (quadratic) for each analyte: r2 >/= 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time 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.	NA.	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA.	NA.	Calculated for each analyte and surrogate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e. Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. 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 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 analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	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. Apply Q-flag to specific Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD = 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	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 with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation of positive results (second column)	All positive results must be confirmed.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD </= 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 3

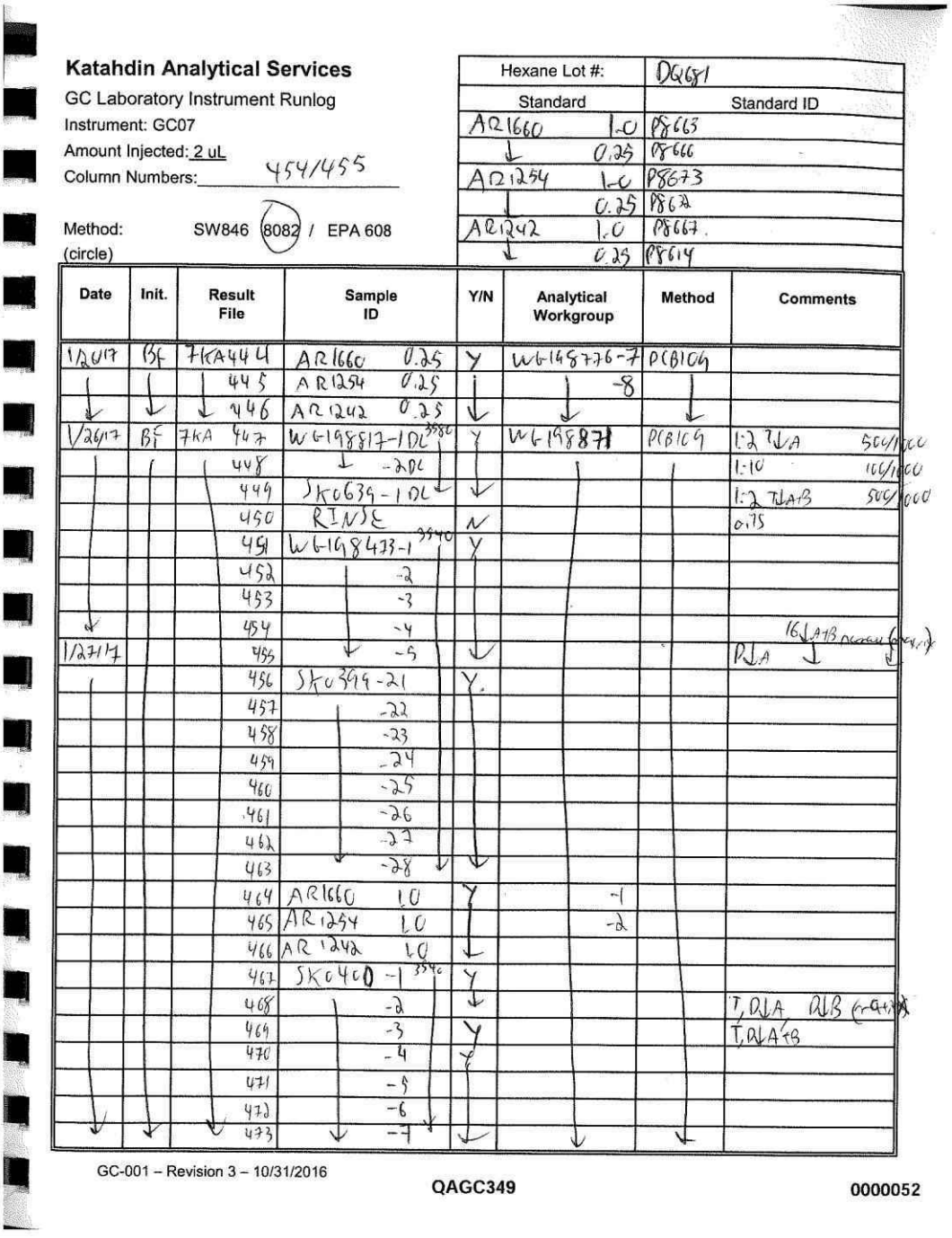
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-19	METHOD 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	9.3 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Spikes		
QC – LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL Practical Quantitation Level – three to ten times the MDL.	EQL Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG



Katahdin Analytical Services
GC Laboratory Instrument Runlog
Instrument: GC07
Amount Injected: 2 uL
Column Numbers: 454/455

Method: SW846 8082 / EPA 608
(circle)

Hexane Lot #:	DQ681
Standard	Standard ID
AR1660 1.0	P8663
↓ 0.25	P8666
AR1254 1.0	P8673
↓ 0.25	P8672
AR1242 1.0	P8667
↓ 0.25	P8614

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
1/26/17	BF	7KA444	AR1660 0.25	Y	WG198776-7	PCB109	
↓	↓	↓ 445	AR1254 0.25	↓	↓ -8	↓	
↓	↓	↓ 446	AR1242 0.25	↓	↓	↓	
1/26/17	BF	7KA 447	WG198817-1DL 3580	Y	WG198871	PCB109	1:2 T↓A 500/1000
↓	↓	↓ 448	↓ -2DL	↓	↓	↓	1:10 100/1000
↓	↓	↓ 449	SK0639-1DL	↓	↓	↓	1:2 T↓A+B 500/1000
↓	↓	↓ 450	RINSE	N	↓	↓	0.75
↓	↓	↓ 451	WG198473-1 3540	Y	↓	↓	
↓	↓	↓ 452	↓ -2	↓	↓	↓	
↓	↓	↓ 453	↓ -3	↓	↓	↓	
↓	↓	↓ 454	↓ -4	↓	↓	↓	16↓A+B [illegible]
1/27/17	↓	↓ 455	↓ -5	↓	↓	↓	P↓A ↓ ↓
↓	↓	↓ 456	SK0399-21	Y	↓	↓	
↓	↓	↓ 457	↓ -22	↓	↓	↓	
↓	↓	↓ 458	↓ -23	↓	↓	↓	
↓	↓	↓ 459	↓ -24	↓	↓	↓	
↓	↓	↓ 460	↓ -25	↓	↓	↓	
↓	↓	↓ 461	↓ -26	↓	↓	↓	
↓	↓	↓ 462	↓ -27	↓	↓	↓	
↓	↓	↓ 463	↓ -28	↓	↓	↓	
↓	↓	↓ 464	AR1660 1.0	Y	↓ -1	↓	
↓	↓	↓ 465	AR1254 1.0	↓	↓ -2	↓	
↓	↓	↓ 466	AR1242 1.0	↓	↓	↓	
↓	↓	↓ 467	SK0400-1 3540	Y	↓	↓	
↓	↓	↓ 468	↓ -2	↓	↓	↓	T, D↓A D↓B [illegible]
↓	↓	↓ 469	↓ -3	Y	↓	↓	T, D↓A+B
↓	↓	↓ 470	↓ -4	Y	↓	↓	
↓	↓	↓ 471	↓ -5	↓	↓	↓	
↓	↓	↓ 472	↓ -6	↓	↓	↓	
↓	↓	↓ 473	↓ -7	↓	↓	↓	

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QAGC349

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TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

REVIEW CHECKLIST ☐ Full Package

PRIMARY

Verbal Due Date _____ . (☐ **Verbals Rev. turned in** *DATE:* _____ *Int.* _____) **DueDate**_____.

Client:	**Verbal Review**	**Primary Review**	**Secondary Reveiw**
Method: **Level :**	Date:	Date:	Date:
SDG No.	Initials:	Initials:	Initials:
Login No.			Approved: ☐ Yes

DODQSM (4.2) ☐ **DODQSM (5.0)** ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐

(*REPORT ND's to -* ***POL*** ☐ ***MDL*** ☐ ***LOD*** ☐)

	Verbal	Final
Verify the above checked criteria are being used throughout the package.	☐	☐
Verify QC limits and PQLs are correct (LCS, Form 2, Form1)	☐	☐
Merged results (Report single ROA ☐) (Report both ROAs ☐)	☐	☐
Extraction Method and Version & Analysis Method and Version Correct.	☐	☐
Date Sampled, Extracted, Analyzed are correct.	☐	☐
Total Solids is entered on the Quantitaion Report and Form1	☐	☐
Flagging of all ROAs correct (DOD ☐ / Florida ☐).	☐	☐
Manual integrations. Date, Initialed and Coded? (Narrate Level 4 samples only).	☐	☐
Were manual corrections made which may be lost if data needs reprocessing?	☐	☐
Narrate any method deviations. (Blanks, LCS's, ICAL, IND, CCV etc.).	☐	☐
Narrative complete and accurate.	☐	☐
All needed forms & raw data are present & in the correct order in the PDF.	☐	☐
All log book pages included (Runlogs, ICAL pgs, Soil wts, Extr, TCLP, SPLP, grinding & GPC)	☐	☐
Level 3 packages include all three PDF files (SUM , ARC, RAW).	☐	☐
Package PDF's copied to the appropriate To Review folder	☐	☐

Package PDF Requirement Level 3 Reports

SUM - (if all forms) - 1, 2, 3, 4, 5, 6, 7, 8. ☐
ARC - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (samples with raw data), 6, 7. ☐
RAW - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (sample ROAs only), 6, IND recoveries, 7. ☐

SECONDARY REVIEW	
☐ FORM 2 (s) DOD O LAB O	☐ FORM 6 (s) DOD O LAB O
☐ FORM 4 (s)	☐ FORM 7 (s) DOD O LAB O
☐ FORM 5 (s)	☐ FORM 1 Sample(s) LOQ O MDL O LOD O
☐ FORM 8 (s)	☐ Flagging B O L O M O C O
☐ FORM 10 (s)	☐ Manual Integrations
☐ FORM 1 Blank (s) LOQ O MDL O LOD O	☐ Logbook Pages
☐ FORM 3 LCS/LCSD (s) DOD O LAB O	☐ Chromatograms & RTs
☐ FORM 3 MS/MSD (s) DOD O LAB O	☐ Manual changes rechecked if data reprocessed

DOD	
CMPD List	**Exceedences**
< 11	**0**
11 to 30	**1**
31 to 50	**2**
51 to 70	**3**
71 to 90	**4**
> 90	**5**

QA-044 – Revision10 – 04/07/2017

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLs FOR METHOD 8082

ANALYTE	Practical Quantitation Level (PQL) (ug/L)	Practical Quantitation Level (PQL) (ug/kg)
AR1016	0.50	17
AR1221	0.50	17
AR1232	0.50	17
AR1242	0.50	17
AR1248	0.50	17
AR1254	0.50	17
AR1260	0.50	17
AR1262	0.50	17
AR1268	0.50	17

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-500
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JC Burton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Duncan F. McGrath Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·23·00	10/23/00
02	removed references to medium level extraction. New logbook figures minor changes through out	LAD	020305 ~~02~~ LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated solvent exchange, updated Table 1, replaced Fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07
05	Updated LB example Added temp. of nitrogen water bath, lot numbers of filter paper, lot #'s of acids need to be recorded in LB. Change "N-Lo" waste to "K" waste.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from < 37°C to < 30°C. Removed respirator references. Added KAEHS manual. Added CA-108 reference for subsampling	LAD	02/09	02/09
07	Removed targeting sample weights Minor changes to section 7. Updated logbook example.	LAD	08/10	08/10
08	Section 5 - Removed baking and rinsing Na_2SO_4, added pH determination for MeCl$_2$, added solvent lot check for acetone. Section 7 - Added information for alternative final volumes, changed spike addition from before $MgSO_4$ to after, added MS/D criteria. Minor changes to reflect current practice.	LAD	03/12	03/12
09	Sect. 4 - Updated Sonicator make and model. Sect. 7 - Changed where hexane is added for solvent exchange. Added decanting information. Updated sonicator setup. Updated Figure 1 - logbook example.	LAD	04/14	04/14
10	Sect. 5 - Added extra LCS compound lists for project specific pesticides/PCBs. Sect. 7 - Updated procedures, Updated sonication instructions. Removed references to old sonicator, Mentioned additional LCSs, Removed redundancies. Minor formatting changes and grammatical corrections throughout. Updated figure 1 - logbook example	LAD	09/17	09/17
11	Added PCB Congener information. Added wooden tongue depressors. Updated references.	LAD	03/19	03/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis in accordance with SW-846 Method 3550, current revision..

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should

also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest

the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors with 85 mm plate diameter (or equivalent)

4.6 Sonicator – Ultrasonic Processor – Qsonica model Q500 or equivalent

4.7 Spatula - stainless steel

4.8 Wooden Tongue Depressors

4.9 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.10 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.11 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.12 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.13 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.14 Balance – top-loading, capable of weighing to 0.01 g

4.15 Nitrogen evaporation apparatus

4.16 Vials (12 mL, 4 mL, 2 mL, and/or 1.8 mL) screw cap vials with Teflon-lined caps or Teflon/silicon septum caps (new, certified clean by manufacturer)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

5.0 REAGENTS AND STANDARDS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, certified by the manufacturer/vendor as purified.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC/MS analysis. Check the pH by shaking equal portions of methylene chloride and water, and then check the pH of the water layer. The pH must be > 5.

5.4 Acetone and hexane - pesticide grade or equivalent, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC analysis.

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 PCB Congener Surrogate Spiking Solution: Prepare a solution of Tetrachloro-m-xylene (TCX), 2,2',4,5',6-pentachlorobiphenyl (PCB 103) and 2,3,3',4,5,5',6-heptachlorobiphenyl (PCB 192) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix spike/Lab control sample spiking solutions:

5.8.1 For every extraction batch, prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
alpha-Chlordane	0.5

beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
gamma-Chlordane	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5

5.8.2 Extra Pesticide Matrix spike/Lab control sample spiking solutions: The following pesticides may be required for certain projects; if so, prepare a spiking solution in pesticide grade methanol that contains the relevant target analytes listed below:

Analyte	**ug/mL**
2,4'-DDD	0.5
2,4'-DDE	0.5
2,4'-DDT	0.5
Alachlor	10.0
Chlorpyrifos	0.5
Dichlobenil	0.5
Hexachlorobenzene	0.5
Mirex	0.5
cis-Nonachlor	0.5
trans-Nonachlor	0.5
Oxychlordane	0.5

5.8.3 Multi-Response Pesticide Matrix spike/Lab control sample spiking solutions: The following multi-response pesticides may be required for certain projects; if so, prepare a separate spiking solution in pesticide grade methanol for each required multi-response target analyte at the concentrations listed below:

Analyte	**ug/mL**
Toxaphene	10.0
Technical Chlordane	10.0
Chlorothalonil	0.5

Propiconizole (Tilt)	0.5

5.9 PCB Arochlor Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260, both at 5.0 ug/mL. For DoD QSM 5.0 or 5.1 projects, additionally prepare spiking solution in pesticide grade acetone that contains PCB Arochlor 1254 at 5.0 ug/mL.

Analyte	**ug/mL**
Arochlor 1016	5.0
Arochlor 1260	5.0
Arochlor 1254	5.0

5.10 PCB Congener Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in pesticide grade acetone that contains all target analytes listed below:

ANALYTE	**ug/mL**
PCB 8 – 2,4'-dichlorobiphenyl	0.2
PCB 18 – 2,2',5-trichlorobiphenyl	0.2
PCB 28 – 2,4,4'-trichlorobiphenyl	0.2
PCB 44 – 2,2',3,5'-tetrachlorobiphenyl	0.2
PCB 49 – 2,2',4,5'-tetrachlorobiphenyl	0.2
PCB 52 – 2,2',5,5'-tetrachlorobiphenyl	0.2
PCB 66 – 2,3',4,4'-tetrachlorobiphenyl	0.2
PCB 77 – 3,3',4,4'-tetrachlorobiphenyl	0.2
PCB 81 – 3,4,4',5-tetrachlorobiphenyl	0.2
PCB 87 – 2,2',3,4,5'-pentachlorobiphenyl	0.2
PCB 101 – 2,2',4,5,5'-pentachlorobiphenyl	0.2
PCB 105 – 2,3,3',4,4'-pentachlorobiphenyl	0.2
PCB 114 – 2,3,4,4',5-pentachlorobiphenyl	0.2
PCB 118 – 2,3',4,4',5-pentachlorobiphenyl	0.2
PCB 123 – 2,3',4,4',5'-pentachlorobiphenyl	0.2
PCB 126 – 3,3',4,4',5-pentachlorobiphenyl	0.2
PCB 128 – 2,2',3,3',4,4'-hexachlorobiphenyl	0.2
PCB 138 – 2,2',3,4,4',5'-hexachlorobiphenyl	0.2
PCB 153 – 2,2',4,4',5,5'-hexachlorobiphenyl	0.2
PCB 156 – 2,3,3',4,4',5-hexachlorobiphenyl	0.2
PCB 157 – 2,3,3',4,4',5'-hexachlorobiphenyl	0.2
PCB 167 – 2,3',4,4',5,5'-hexachlorobiphenyl	0.2
PCB 169 – 3,3',4,4',5,5'-hexachlorobiphenyl	0.2
PCB 170 – 2,2',3,3',4,4',5-heptachlorobiphenyl	0.2
PCB 180 – 2,2',3,4,4',5,5'-heptachlorobiphenyl	0.2

PCB 183 – 2,2',3,4,4',5',6-heptachlorobiphenyl	0.2
PCB 184 – 2,2',3,4,4',6,6'-heptachlorobiphenyl	0.2
PCB 187 – 2,2',3,4',5,5',6-heptachlorobiphenyl	0.2
PCB 189 – 2,3,3',4,4',5,5'-heptachlorobiphenyl	0.2
PCB 195 – 2,2',3,3',4,4',5,6-octachlorobiphenyl	0.2
PCB 206 – 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	0.2
PCB 209 – 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	0.2

5.11 Store the solutions at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

The holding time for extraction of Pesticide sediment/soil samples by Method 3550 is 14 days from date of sample collection.

The holding time for PCB only sediment/soil is 30 days. SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit.

Samples extracted for both Pesticide and PCB must be extracted within the 14 day hold time.

Analysts should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction and Analytical methods
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sonicator Amplitude
- Sample pH (if applicable)

- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weights
- Surrogate and spike amounts
- Boiling chip date
- Balance ID
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- Lot numbers of the vials the concentrated extracts are stored in.

EXTRACTION OF LOW LEVEL SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of approximately 30.0 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

7.1 Do not decant any water on the sediment sample.

Note: Some work orders may specify to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2 Mix with a stainless steel spatula or tongue depressor to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. **Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique“, for more detailed guidance on subsampling to ensure reproducibility.**

7.3 Weigh out approximately 30.0 g plus (> 30 g) portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Refer to section 7.7 for surrogate addition instructions. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or tongue depressor. Keep the spatula/depressor in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out 30.0 ± 0.05 g of purified sand in a labeled 400 mL beaker. Refer to section 7.7 for surrogate addition instructions. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out 30.0 ± 0.05 g of purified sand into a labeled 400 mL beaker. Refer to sections 7.7 and 7.8 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. Additional LCS's for project specific compounds may also be required. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximately 30.0 g plus (>30 g) portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.7 and 7.8 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

Note: A MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. If samples are to be concentrated to a lower than 10 mL final volume the surrogate

spike must be adjusted so the final concentration in the samples is 0.10 ug/mL. The surrogate spike should be added after the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.8 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. If samples are to be concentrated to a lower than 10 mL final volume the surrogate spike must be adjusted so the final concentration in the samples is 0.5 ug/mL for pesticides and 5 ug/mL for PCBs. The LCS/MS spike should be added after the addition of the sodium sulfate. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.9 To each mixed and spiked sample, blank, LCS/LCSD, and/or MS/MSD in the extraction batch, add approximately 100 mL of the 1:1 methylene chloride/acetone solution.

7.10 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula/depressor to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.11 Sonicator settings:

7.11.1 Set pulse timer to 1 ½ minutes.

7.11.2 Set pulse to 1 and 1. This sets the sonicator to pulse for 1 second and rest for 1 second, with the timer set for 1 ½ minutes this equals a 3 minute total sonication.

7.11.3 Set amplitude to 40%. Record this in the logbook.

These settings are stored in the unit and do not have to be entered with each use.

7.12 Extraction by sonication:

7.12.1 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.12.2 Turn sonicator on and allow 3 minutes for sonication process.

7.12.3 Refer to the Operating Manual for further information.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse flask, funnel, and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.14 Repeat steps 7.9, 7.10, 7,12, and 7.13 two additional times, each time adding approximately 100 mL portions of 1:1 methylene chloride:acetone to the beaker. Before each sonication, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula/depressor. Use the same Buchner funnel and filter for all three sonications for each sample. Decant the extraction solvent into the Buchner funnel after each sonication. On the final (third) sonication, pour the entire beaker contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract.

7.15 Repeat the extraction (steps 7.9 through 7.14) for each sample in the extraction batch.

CONCENTRATION OF LOW LEVEL EXTRACTS FOR PESTICIDES/PCBs

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, proceed to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. PCB Congener samples must be GPC'd. If samples are not to be GPC'd, proceed to step 7.19.

7.19 For a solvent exchange, add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than hexane, this will result in a final extract in hexane only.

Note: For Pesticide / PCB samples originating from South Carolina (see worknotes) do not add the hexane at this step. Solvent exchange will be during the nitrogen blow down procedure.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39 ºC for methylene chloride).. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane (methylene chloride for samples not yet solvent exchanged). The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.23 For samples that still need to be solvent exchanged, reduce the methylene chloride extract to ~ 1 mL. Add 10 mL of hexane to the concentrator tube and reduce to ~ 1 mL again on the N-evap.

7.24 Complete quantitative transfer of the extract to a vial by using hexane: Add hexane to adjust the volume of the hexane extract to the appropriate final volume, in a 12 mL vial (for 10 mL or 5 mL final volumes), a 4 mL vial (for 2 mL final volume), or a 1.8 mL vial (for 1 mL final volume). Rinse sides of tube with hexane and transfer rinses to final vial, taking care not to exceed the intended final volume. Use the appropriate reference vial for samples with 10, 5, 2 or 1 mL final volumes.

7.25 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.26 All sample extracts for 8082 PCB Arochlor analysis that are not GPC’d must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA-525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

Note: The lot number of the acid used in PCB cleanup must be recorded in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other

corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

8.2 A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Final Update IV, February 2007.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

Table 1 Summary of Method Modifications
Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

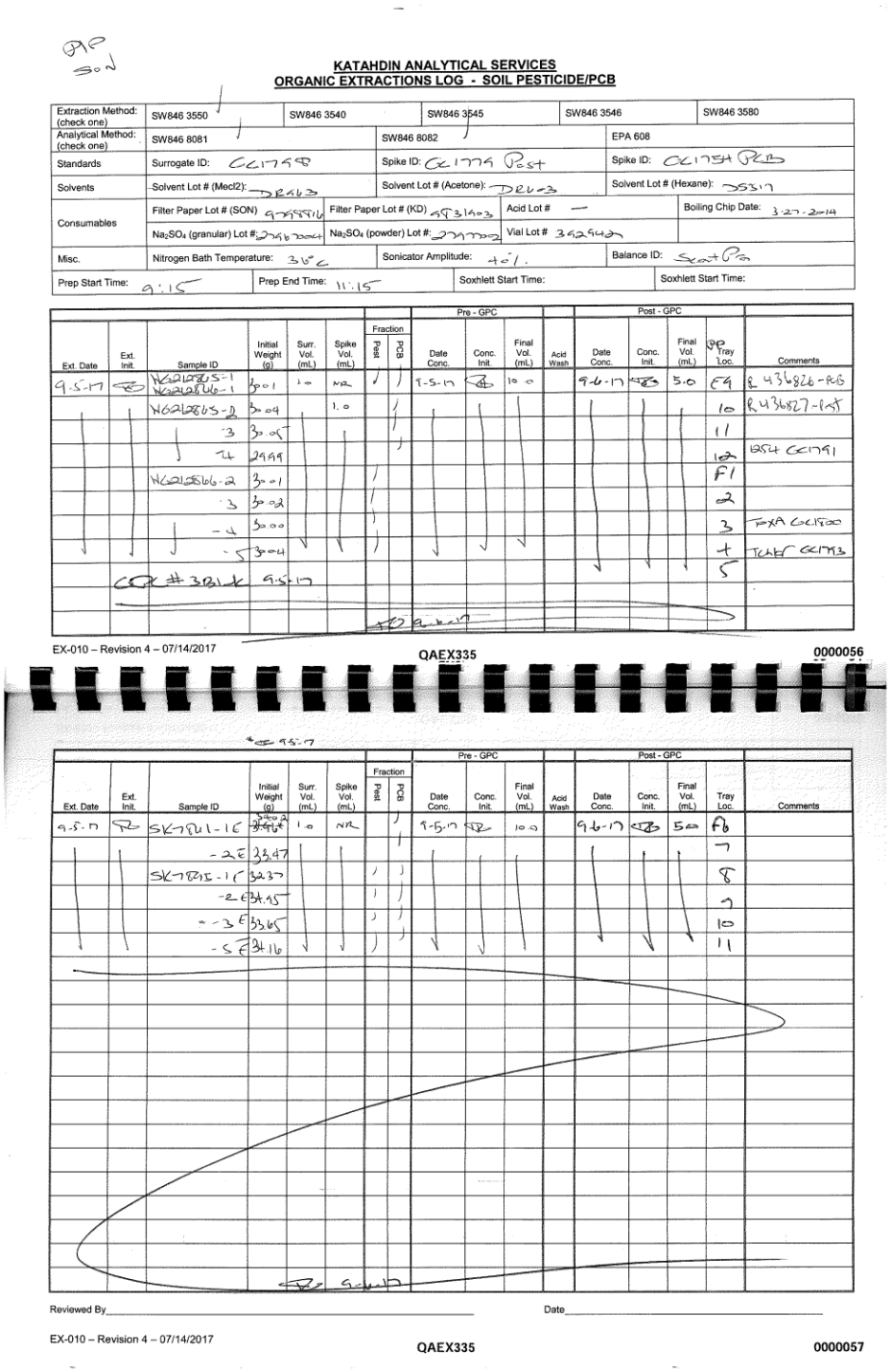
TOPIC	KATAHDIN SOP CA-500-11	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PP
SON



KATAHDIN ANALYTICAL SERVICES
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550	SW846 3540	SW846 3545	SW846 3546	SW846 3580
Analytical Method: (check one)	SW846 8081	SW846 8082	EPA 608		
Standards	Surrogate ID: GC1798	Spike ID: GC1779 Pest	Spike ID: GC1754 PCB		
Solvents	Solvent Lot # (Mecl2): DR463	Solvent Lot # (Acetone): DR603	Solvent Lot # (Hexane): DS317		
Consumables	Filter Paper Lot # (SON) 9798816	Filter Paper Lot # (KD) 9831903	Acid Lot # —	Boiling Chip Date: 3-27-2014	
	Na_2SO_4 (granular) Lot #: 2796004	Na_2SO_4 (powder) Lot #: 2797002	Vial Lot # 392942		
Misc.	Nitrogen Bath Temperature: 36°C	Sonicator Amplitude: 40%	Balance ID: Scout Pro		
Prep Start Time: 9:15	Prep End Time: 11:15	Soxhlett Start Time:	Soxhlett Start Time:		

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	PP Tray Loc.	Comments
9-5-17	[illegible]	WG212865-1 WG212866-1	30.01	1.0	NR	✓	✓	9-5-17	[illegible]	10.0		9-6-17	[illegible]	5.0	E9	R 436826-PCB
↓	↓	WG212865-2	30.04	↓	1.0	↓	✓	↓	↓	↓		↓	↓	↓	10	R 436827-Pest
↓	↓	-3	30.05	↓	↓	↓	✓	↓	↓	↓		↓	↓	↓	11	
↓	↓	-4	29.99	↓	↓	↓	✓	↓	↓	↓		↓	↓	↓	12	1254 GC1791
↓	↓	WG212866-2	30.01	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	F1	
↓	↓	-3	30.02	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	2	
↓	↓	-4	30.00	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	3	TOXA GC1800
↓	↓	-5	30.04	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	4	Tchlor GC1793
		GPC #3B1-K	9-5-17												5	

[illegible] 9-6-17

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*[illegible] 9-5-17

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
9-5-17	[illegible]	SK7841-1E	~~34.96~~ 34.02	1.0	NR		✓	9-5-17	[illegible]	10.0		9-6-17	[illegible]	5.0	F6	
↓	↓	-2E	33.47	↓	↓	↓	✓	↓	↓	↓		↓	↓	↓	7	
↓	↓	SK7895-1E	32.37	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	8	
↓	↓	-2E	34.95	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	9	
↓	↓	-3E	33.65	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	10	
↓	↓	-5E	34.16	↓	↓	✓	✓	↓	↓	↓		↓	↓	↓	11	

[illegible] 9-6-17

Reviewed By_____ Date_____

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TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.15.00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.15.00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced Figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09
07	Removed targeting sample weights. Removed decanting samples prior to extraction.	LAD	08/10	08/10
08	Minor changes to Section 7 to reflect current practices. Updated Section 7.6 for frequency of MS/D's. Added information for MDL, LOD and LOQ to Section 9. Updated References. Added H_2SO_4 to reagents.	LAD	04/12	04/12
09	Sect. 7 - Water bath temp. is set at 80°C, fixed typo, added information @ decanting, Sect. 10 - Updated references. Updated KAS INC. to KAS LLC. Updated Fig. 1.	LAD	12/14	12/14
10	Sect. 5 - Added additional LCS/MS spikes. Sect. 6 - Changed PCB H.T. to 30 days. Sect. 7 - Small edits for clarity and to reflect current practice. Added Attachment A - Total Solids Determination	LAD	01/18	01/18
11	Added wooden tongue depressors, Corrected rheostat setting. Updated references. Updated logbook page example	LAD	03/19	03/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-11**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

I acknowledge receipt of copy ___ of document **SOP CA-524-11**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste

stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

4.1.1 500 mL flat-bottom boiling flask

4.1.2 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Gastight Volumetric Syringes – Various sizes

4.15 Top-loading balance - capable of weighing to 0.01 g.

4.16 Spatulas, stainless-steel

4.17 Wooden Tongue Depressors

4.18 Long forceps, stainless-steel

4.19 Metal clips – for securing Soxhlets to boiling flasks

4.20 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.21 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS™ with internal UV detection

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Sulfuric acid solution (1:1 H2SO4 : H2O) – Prepared in an icebath by slowly adding a volume of concentrated H2SO4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.4 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.5 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.7 Matrix Spike/Lab Control Sample spiking solution

5.7.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone

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Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.7.2 Aroclor 1660 spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7.3 Aroclor 1254 spike solution – prepare Aroclor 1254 in pesticide grade acetone at a concentration of 5.0 ug/mL.

5.7.4 Technical Chlordane spike solution and Toxaphene spike solution – prepare separately, in pesticide grade acetone at a concentration of 10 ug/mL each.

5.7.5 Additional miscellaneous pesticide compounds – Prepare in pesticide grade acetone at a concentration of 0.5 ug/mL each

5.8 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

The holding time for extraction of Pesticide sediment/soil samples by Method 3550 is 14 days from date of sample collection.

The holding time for PCB only sediment/soil is 30 days. SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit.

Samples extracted for Pesticide and PCB must be extracted within the 14 day holdtime.

Analysts should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

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7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Extraction start and end times
- Soxhlett extraction start time and end time and date
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Do not decant any water on the sediment sample

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Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2.2 Mix the sample thoroughly with the stainless steel spatula or wooden tongue depressor. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.3 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.4 Refer to Attachment A for total solids determination.

7.2.5 Please refer to Katahdin Analytical Services SOP CA-108, current revision, "Basic Laboratory Techniques" for more information of subsampling.

7.3 Weigh out an approximate, greater than 30 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or wooden tongue depressor. Keep the spatula/tongue depressor in the sample beaker and cover the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB

analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set should be prepared for every 20 samples. An MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed. To prepare MS/MSD, weigh out two approximate, greater than 30g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas or wooden tongue depressors. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful that none of solid material falls into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL (if FV=10mL, adjust amount for different final volumes) of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL (if FV=10mL, adjust amount for different final volumes) of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 40-45% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 16-24 hours. Be

sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as much solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

7.14 Pest/PCB and Pesticide only extracts will be GPC'd unless there is a time constraint.

If extracts are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. **Extracts that will be GPC'd are not solvent exchanged into hexane.**

7.15 All extracts (GPC and non-GPC) - Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask

three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.16 Extracts not being GPC'd – Begin the solvent exchange of the extracts by adding approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.16.1 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.17 All extracts (GPC and non-GPC) – Please use the same solvent as the extract for rinsing and bringing the extract to the final volume. Methylene chloride should be used with extracts that are to be GPC'd. Hexane should be used for extracts the either have been GPC'd or are not going to be GPC'd.

7.17.1 Place the K-D in a hot water bath set at 75°C. Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane or methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane or methylene chloride.

7.17.2 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.17.3 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane or methylene chloride extract to 10 mL (or a client specified final volume) in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.17.4 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Record the box number and "tray location" for each extract vial.

7.18 Additional cleanup for non-GPC'd extracts:

7.18.1 Extracts for 8082 PCB only analysis must be sulfuric acid washed. The associated Method Blank and LCS must also be sulfuric acid washed. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA-525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

7.18.2 Extracts for combined 8081/8082 analyses must be split. Mix extract well before splitting. One portion, including the Blank, LCS and MS/MSD, must be sulfuric acid washed for 8082 analysis. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

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Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD’s must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.
MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

LIST OF TABLES AND FIGURES

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

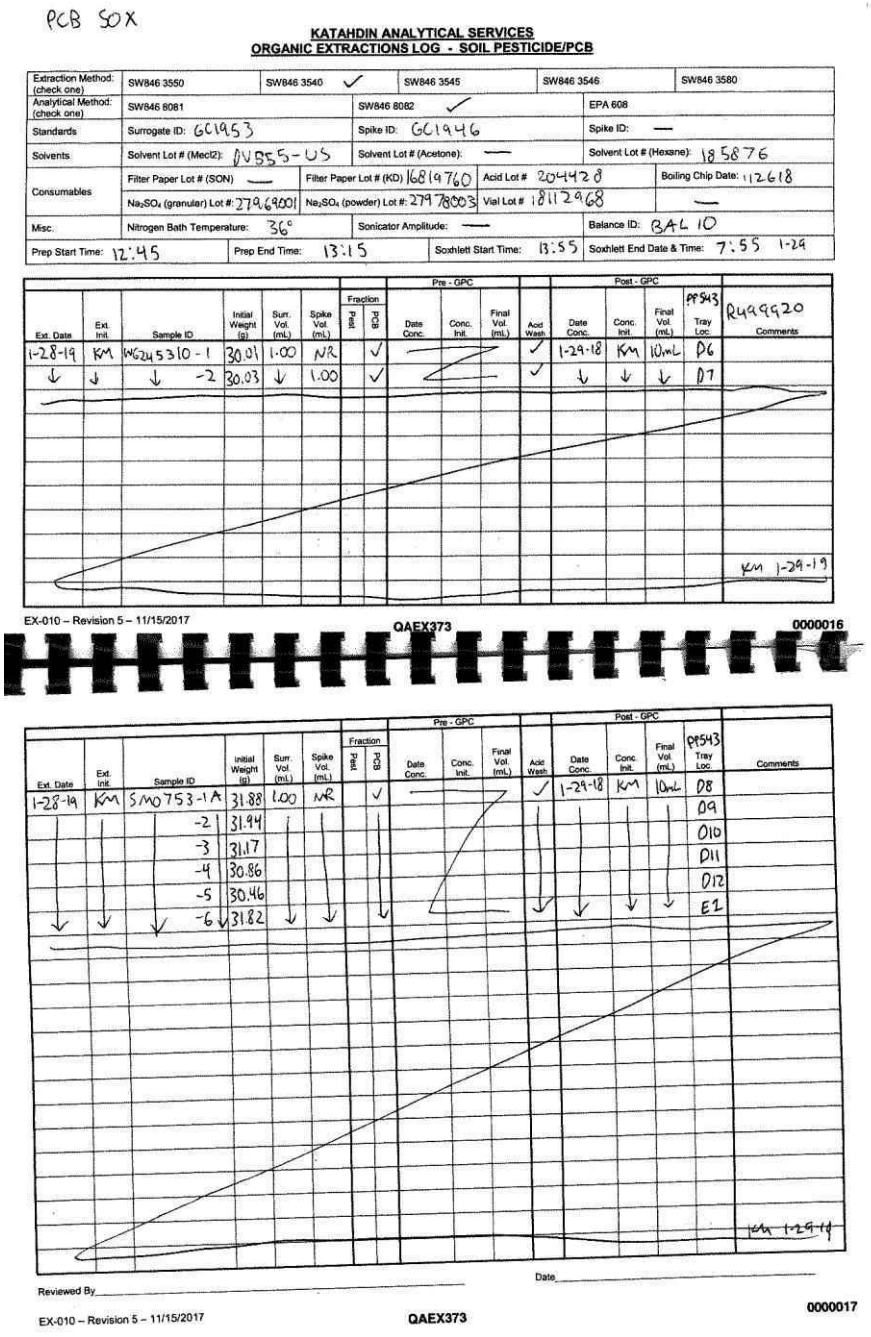
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-11	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PCB SOX

KATAHDIN ANALYTICAL SERVICES
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550	SW846 3540 ✓	SW846 3545	SW846 3546	SW846 3580
Analytical Method: (check one)	SW846 8081	SW846 8082 ✓		EPA 608	
Standards	Surrogate ID: GC1953	Spike ID: GC1946		Spike ID: —	
Solvents	Solvent Lot # (MeCl2): DV855-US	Solvent Lot # (Acetone): —		Solvent Lot # (Hexane): 185876	
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) 16819760	Acid Lot # 204428	Boiling Chip Date: 112618	
	Na_2SO_4 (granular) Lot #: 279.6900	Na_2SO_4 (powder) Lot #: 279 78003	Vial Lot # 18112968	—	
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Amplitude: —		Balance ID: BAL 10	
Prep Start Time: 12:45	Prep End Time: 13:15	Soxhlett Start Time: 13:55		Soxhlett End Date & Time: 7:55 1-24	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Post	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	PP543 Tray Loc.	Comments
1-28-19	KM	WG245310-1	30.01	1.00	NR		✓				✓	1-29-18	KM	10mL	D6	R499920
↓	↓	↓ -2	30.03	↓	1.00		✓				✓	↓	↓	↓	D7	

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Post	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	PP543 Tray Loc.	Comments
1-28-19	KM	SMO753-1A	31.88	1.00	NR		✓				✓	1-29-18	KM	10mL	D8	
		-2	31.94												D9	
		-3	31.17												D10	
		-4	30.86												D11	
		-5	30.46												D12	
↓	↓	-6 ↓	31.82	↓	↓		↓				↓	↓	↓	↓	E2	

KM 1-29-19

Reviewed By _____ Date _____

EX-010 – Revision 5 – 11/15/2017 QAEX373 0000017

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

ATTACHMENT 1

DETERMINATION OF PERCENT DRY WEIGHT

When sample results are to be calculated on a dry weight basis, a second portion of sample should be weighed within 14 days of the sample date.

WARNING: The drying oven should be contained in a hood or be vented. Significant laboratory contamination may result from drying a heavily contaminated sample.

1.0 PROCEDURES

1.1 In accordance with the current revision of Katahdin SOP CA-102, “Balance Calibration”, verify balance calibration at the beginning of the day or prior to beginning analysis. Refer to Section 8.0 for acceptance criteria.

1.2 The oven ID must be recorded in KIMS

1.3 Label and weigh each tin dish before use and record initial (tare) weight in KIMS.

1.4 Samples will be analyzed under this procedure as “total solids” or “decanted total solids”. “Total Solids” will be performed on a well-homogenized sample as received where any aqueous phase is incorporated into the solid matrix prior to aliquotting. “Decanted total Solids” will be performed on sample aliquots designated for extractable organic parameters where it has been determined that the standing aqueous layer must be decanted prior to aliquotting for the organic extractable preparation. Sample login decants the samples during the login process. Decanted samples will marked with a “DEC” on the cap. The decanted sample aliquot will constitute the “original” sample for total solids determination.

1.5 Whether the sample has been decanted or not, remove any obvious non-representative material such as large sticks and rocks. Thoroughly mix the sample with a scupula. Weigh 10-20 g of a representative sample aliquot into a labeled weighing dish. For samples that are analyzed “as received” and contain a significant standing aqueous layer, a larger aliquot may be used. In order to facilitate drying and minimize moisture entrainment, the sample may be spread out across the surface of the pan/dish/crucible. Please refer to Katahdin SOP, CA-108, Basic Laboratory Technique, current revision, for more information on subsampling.

1.6 Record the weight (to the nearest 0.0001g) of the sample plus dish by uploading the weight directly into KIMS. Please refer to the current version of SOP CA-717, Total Solids/Total Volatile Solids Determination in Solid Matrices, for instructions.

1.7 A Method Blank and LCS are prepared at a minimum frequency of 1/20 field samples and a Duplicate is prepared at a minimum frequency of 1/10 field samples or per preparation batch if fewer samples.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.8 A Method Blank consists of a weighing tin/dish/crucible spiked with 0.50 mL of water carried through the entire analytical process. The Method Blank result must be less than the PQL of 0.1 wt%. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

1.9 The LCS is prepared by spiking muffled dry sand with a known amount of laboratory reagent grade water. Weigh out 4.5 g of sand and 0.50 mL (0.50 g) of laboratory reagent grade water and carry LCS through entire analytical process. Recovery for the LCS must be within 90-110% of the expected true value. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

1.10 A Duplicate is prepared by taking a duplicate sample aliquot, 10-20 g, through the procedure as described for the sample. NOTE: The duplicate should be aliquotted at the same time as the original sample.

1.11 Transfer samples to drying oven maintained at 103-105°C. Record “date in”, “time in”, and “temperature in” in KIMS.

1.12 Dry samples for greater than 12 hours. Remove the samples from the oven and cool to ambient temperature in a desiccator. Record “date out”, “time out”, and “temp out” in KIMS.

1.13 Weigh and record in KIMS the final sample plus dish weights to the nearest 0.0001g.

1.14 The raw data (batch number, sample number, pan weight, original sample + pan, and final sample + pan weights) are entered into KIMS for calculation. The calculation performed is as follows:

$$\%\ \text{Total Solids} = \frac{(\text{B} - \text{pan, g})}{(\text{A} - \text{pan, g})} \times 100$$

Where: *A* = weight of original sample+ pan, g, and
B = weight of dried sample + pan, g

1.15 EXPORTING TOTAL SOLIDS DATA INTO KIMS – Please refer to the current version of SOP CA-717, Total Solids/Total Volatile Solids Determination in Solid Matrices, for instructions.

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.16.00

General Manager: [signature] Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 & 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 IND 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~spikes~~ compounds used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added additional compounds to Fig. 2. Added recording of consumable's lot #'s and recording the Nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised Section 7 to reflect current techniques. Added SOP CA-108 reference for sub-sampling. Updated logbook example	LAD	08/09	08/09
07	Removed targeting sample weights. Added to weigh out a minimum 30g sample. Removed decanting samples prior to extracting. Added putting solid waste in the "I" waste stream. Updated Logbook page.	LAD	08/10	08/10
08	Section 5.5 - Added 1,4-Dioxane-d8 and updated 2,4-Dibromophenol's concentration. Section 5.7 - Updated acid spikes concentration. Sections 7.0, 7.6 and 7.13 - minor changes to reflect current practice. Added MDL, LOD and LOQ information to Section 9. Updated references in Section 10.	LAD	04/12	04/12
09	Sect. 5 - Updated Surr. prep. for both SIM and Scan Surr. - now in 1 mix. Sect. 7. Updated spiking info. for SCAN/SIM surr. Clarified decanting soils. Sect. 10 - Added and updated references. Updated Fig. 1.	LAD	06/14	06/14
10	Sect. 1 - Added pollution control to Waste Disposal Sect. 5 - Added standards to title. Sect. 7 - Added to record the date Sox. ends, changed M. BLK and LCS initial weight from >30g to 30g. Updated Fig. 1 - logbook ex. KAS INC → KAS throughout	LAD	08/16	08/16
11	Updated method references for DOD+SW846. Clarified weight of sample. Fixed grammatical errors. Changed water bath temperature.	LAD	09/17	09/17
12	Added Wooden tongue depressors, updated Rheostat setting, updated references, Corrected typographical errors	LAD	04/19	04/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Documentation of Capability”.

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It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream

satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:

4.1.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

4.1.2 500 mL flat-bottom boiling flask

4.1.3 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Wooden Tongue Depressor

4.17 Long forceps, stainless-steel

4.18 Metal clips – for securing Soxhlets to boiling flasks

4.19 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS AND STANDARDS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, pre-baked, certified by the manufacturer/vendor.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 SV SCAN/SIM Surrogate Spiking Solution – A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.6 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral and 4 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH
- Extraction and concentration dates
- Extraction and concentration analyst
- Soxhlet extraction start and end dates and times
- Prep Date and start and end times.
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

Pre-rinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Do not decant any water layer on a sediment sample.

Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2.2 Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, "Basic Laboratory Technique" for more information on subsampling.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out an approximate, greater than 30 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30 g ±0.5g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g ±0.5 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If

combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the SV SCAN/SIM surrogate spiking solution using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on

the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 40-45% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as much solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures along with the glass wool from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries. Put the solid waste in the "I" waste stream.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative

transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality

Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-12	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

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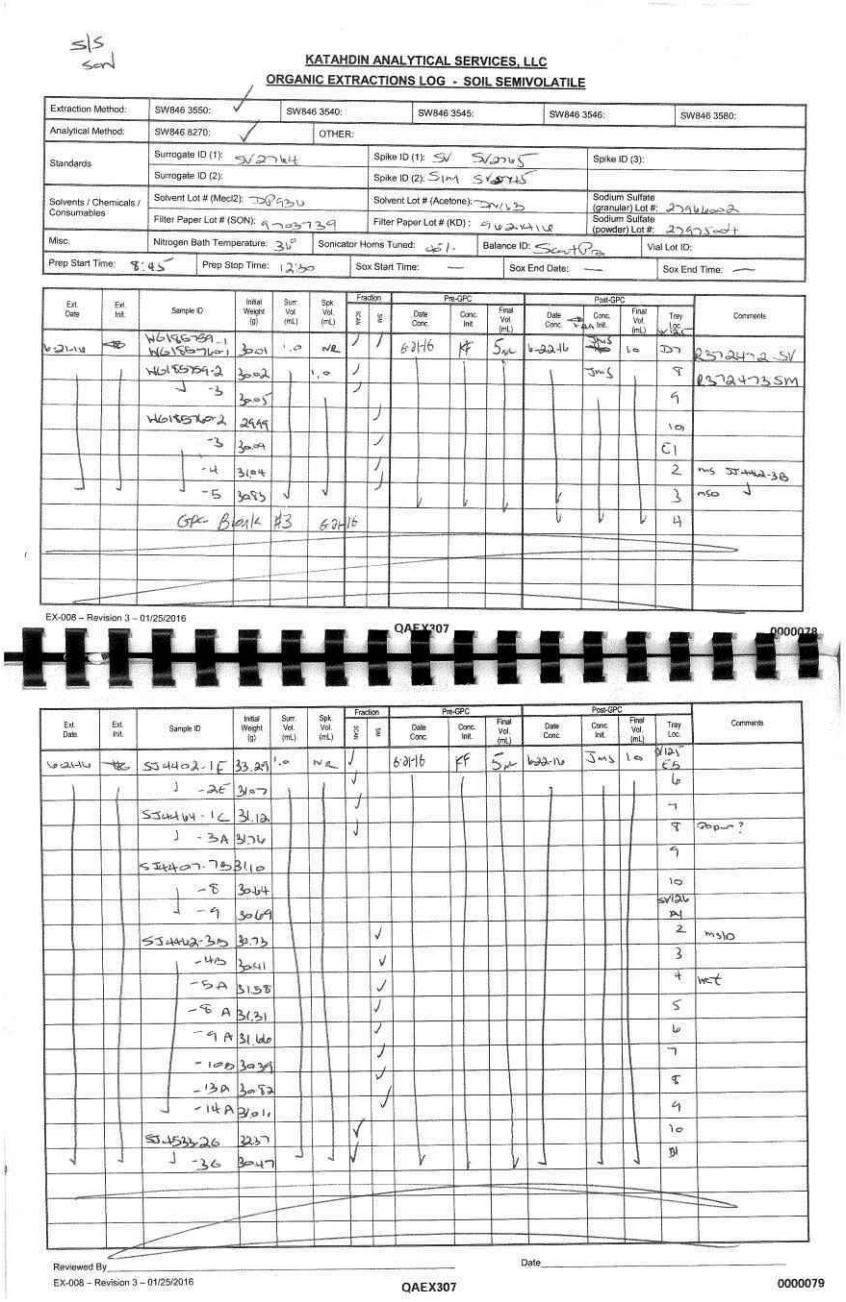
FIGURE 1

EXAMPLE OF LOGBOOK PAGE



S/S
Son

KATAHDIN ANALYTICAL SERVICES, LLC
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method:	SW846 3550: ✓	SW846 3540:	SW846 3545:	SW846 3546:	SW846 3580:
Analytical Method:	SW846 8270: ✓	OTHER:			
Standards	Surrogate ID (1): SV2764	Spike ID (1): SV SV2765	Spike ID (3):		
	Surrogate ID (2):	Spike ID (2): SIM SV2745			
Solvents / Chemicals / Consumables	Solvent Lot # (Mecl2): DP9336	Solvent Lot # (Acetone): DN163	Sodium Sulfate (granular) Lot #: 27966002		
	Filter Paper Lot # (SON): 9703739	Filter Paper Lot # (KD): 9624416	Sodium Sulfate (powder) Lot #: 27975004		
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Horns Tuned: 45%	Balance ID: ScoutPro	Vial Lot ID:	
Prep Start Time: 8:45	Prep Stop Time: 12:30	Sox Start Time: —	Sox End Date: —	Sox End Time: —	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WC	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-21-16	[illegible]	WG185759-1 WG185760-1	30.01	1.0	NR	✓	✓	6-21-16	KF	5 µL	6-22-16	JMS	10	D7	R372472-SV
		WG185759-2	30.02		1.0	✓						JMS		8	R372473SM
		-3	30.05			✓								9	
		WG185760-2	29.99				✓							10	
		-3	30.09				✓							E1	
		-4	31.04				✓							2	ms SJ4462-3B
		-5	30.83				✓							3	mso
		GPC Blank #3			6-21-16									4	

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WC	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-21-16	[illegible]	SJ4402-1E	33.29	1.0	NR	✓		6-21-16	KF	5 µL	6-22-16	JMS	10	D1/25 E5	
		-2E	31.07			✓								6	
		SJ4464-1C	31.12			✓								7	
		-3A	31.76			✓								8	20pm?
		SJ4407-7B	31.10											9	
		-8	30.64											10	
		-9	30.69											SV126 A1	
		SJ4462-3B	30.73				✓							2	mslo
		-4B	30.41				✓							3	
		-5A	31.58				✓							4	wet
		-8A	31.31				✓							5	
		-9A	31.66				✓							6	
		-10B	30.39				✓							7	
		-13A	30.82				✓							8	
		-14A	31.01				✓							9	
		SJ4533-2G	32.37			✓								10	
		-3G	30.47			✓								B1	

Reviewed By _____ Date _____

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

eurofins	Always check on-line for validity. **Determination of Hydrazine, Monomethylhydrazine and 1,1-Dimethylhydrazine in Soil samples by LC/MS/MS**	Level: **Work Instruction**
Document number: **T-SSG-WI9431**		
Old Reference: **1-P-QM-WI-9015096**		
Version: **6**		Organisation level: **5-Sub-BU**
Approved by: **UDM6** Effective Date **02-OCT-2018**	Document users: **5_EUUSLA_Specialty Services_Manager, 6_EUUSLA_Specialty Services_Data Reviewers, 6_EUUSLA_Specialty Services_Sample_Prep, 6_EUUSLA_Specialty_Services_Analyst**	Responsible: **5_EUUSLA_Specialty Services_Manager**

LIMS ID
Analysis DOD - 10346

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Revision Log
Reference
Reference Modifications (USEPA 8315A)
Cross Reference
Scope
Basic Principles
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Preparation of Glassware
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 6		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in D4	Removed old document numbers and replaced with D4 document numbers
Apparatus and Equipment	Reflect current method	Added pH paper to 0.5 pH, Addition of Waters LCMS system,
Reagent and Standard (A.)	Reflect current method	

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-551
Revision History
Cover Page
Page 1

TITLE: GRAIN SIZE ANALYSIS

Prepared By: _____ Date: 10-1-15

Approved By:

Department Manager: _____ Date: 10-1-15

Operations Manager: _____ Date: 10.1.15

QA Officer: _____ Date: 10.01.15

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated title of sections 1.4 + 5.0, Updated method references for NELAC + DoD Added information about hydrometer readings for Sandy samples.	LAD	09/17	09/17
02	Sect. 2 - Updated method summary, Sect. 4 - Added 1/4" Sieve, removed 3/4". Sect. 7 - Updated Sieve only analysis. Updated references and logbook example	LAD	04/19	04/19

TITLE: **Grain Size Analysis**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-551-02,** titled **GRAIN SIZE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-551-02,** titled **GRAIN SIZE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **Grain Size Analysis**

1.0 SCOPE AND APPLICATION

This SOP details the procedure used by Katahdin Analytical Services technical personnel for particle size analysis in soils. This method is applicable to ASTM D422.

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in Grain Size Analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in Grain Size Analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to indicate periodic review of the associated logbooks

1.3 Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation

from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

For Grain size with hydrometer, depending on total solids of a sample, a certain amount is soaked in a dispersing agent overnight. The sample is then split into two portions, the material retained on the #10 sieve and the material passing the #10 sieve. The material retained on the #10 sieve is dried overnight to a constant weight. The sample is then passed through a large size sieve stack. Material passing the #10 sieve may be subjected to hydrometer analysis. After wet washing the sample on a #200 sieve the sample retained is dried overnight, then passed through a small size sieve stack. The material retained on each sieve, large and small sieves are measured and recorded. All measurements, large and small sieves and hydrometer readings are used to determine the particle size distribution of the sample. If the analysis requires sieve only, the sample weight will be determined as with samples including hydrometer. The samples will then be soaked in DI overnight, than wet washed and baked overnight. They will then be sieved through large and small sieves.

3.0 INTERFERENCES

Not Applicable

4.0 APPARATUS AND MATERIAL

4.1 Sieves ASTM E-11 Specifications, Brand Advantech, of the following size(s):

4.1.1 3.0" (75.00 mm)

4.1.2 2.0” (50.00 mm)

4.1.3 1.5” (37.50 mm)

4.1.4 1.0” (25.00 mm)

4.1.5 3/4" (19.00 mm)

4.1.6 1/4 “ (6.3mm)

4.1.7 # 4 (4.75 mm)

4.1.8 #10 (2.00 mm)

4.1.9 #20 (850.0 um)

4.1.10 #40 (425 um)

4.1.11 #60 (250.0 um)

4.1.12 #80 (180 um)

4.1.13 #100 (150.0 um)

4.1.14 #200 (75.0 um)

4.2 Sedimentation Classico Cylinder(s) 1000 mL

4.3 Hydrometer: ASTM 151H Humboldt H-4242

4.4 Drying Oven with temperature range of 60-110°C

4.5 Stainless Steel Spatulas & Spoons

4.6 Metal & Bristle Brushes

4.7 Ro-Tap Sieve Shaker- Gilson Company

4.8 Timers- capable of counting up to 24hours

4.9 Balance, capable of weight measurement to 0.01 g

4.10 Mechanical Stirring Device and Dispersion Cup- Hamiliton Beach Humboldt

4.11 Thermometer: Accurate to 0.5°C

4.12 Mortar and Rubber Tipped Pestle

4.13 Glass beakers- 1000ml and 500ml

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Grade Reagent Water

5.2 Sodium Hexametaphosphate:

5.3 **Sodium Hexametaphosphate Solution**: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon plastic jug with cover. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 The following information must be recorded in the Grainsize logbook (all that are applicable).

- Start/End Date and Time
- Date/Time placement of samples in and out of oven
- Hydrometer(s) Serial Number
- Hydromter(s) Calibration Date
- Balance ID(s)
- Reagant ID
- Initial and final weights
- Analysts Initials
- Any comments regarding the sample extraction

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

7.2 Equipment Calibration

Calibrate the balances being used each day prior to use. Record in the logbook designated for this purpose.

Calibrate or replace the hydrometers every five years

7.3 Total Solids Determination

Refer to SOP CA-717 “Total Solids/Total Volatile Solids Determination In Solid Matrices”

7.4 Sample Preparation

7.4.1 From the calculated percent total solids and the sample characterisitics for each sample the amount needed for the analysis can be determined using Table 1.

7.4.2 After determining the amount of sample to be used, place a 1000ml glass beaker on the balance and tare the balance. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.4.3 Add 125ml sodium hexametaphosphate solution to each beaker containing sample. Stir and mix thoroughly, soak sample in solution for at least 16 hours.

7.4.4 Refer to Katahdin SOP CA-108, current revision, “Basic Laboratory Technique” for more information on subsampling.

7.5 Sample Partition

After sample and solution has soaked for a minimum of 16 hours, the sample slurry is rinsed into a dispersion cup using DI water. Fill the dispersion cup ½ full with DI water and place the cup on the blender to mix for one minute.

7.5.1 If the sample appears to have large gravel, sand, or organic material that does not appear to be amendable for using the blender notify the PM and proceed to the next step without blending.

7.5.2 After the sample slurry has been blended, pour sample through a #10 sieve with pan attached, so sample smaller than a #10 sieve is collected in pan. Transfer contents in pan to a 1000ml graduated cylinder and continue to rinse the dispersion cup through the #10 sieve until transfer is complete. After all sample in the pan is rinsed into the cylinder bring the volume of the cylinder to 1000ml using DI water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature for the hydrometer analysis.

7.5.3 Weigh and label a medium aluminum pan, than transfer the contents of the material retained on the # 10 sieve to the pan. Place the aluminum pan in a drying oven set at 110 $\pm$ 5° C and dry the sample material for at least 16 hours or until constant weight set aside for sieve analysis.

7.6 Hydrometer

Prepare a hydrometer blank by adding 125ml sodium hexametaphosphate and bring to 1000 mL with DI water in a 1000 mL graduated cylinder. Be sure to take readings with a hydrometer and a thermometer while taking readings on actual hydrometer samples. This will provide us with the temperature and solution correction factors later in the procedure. Also prepare a hydrometer rinse bath, used to rinse the hydrometer between uses.

7.6.1 To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

7.6.2 To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait approximately 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the logbook. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

7.6.3 Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the logbook. Rinse the temperature probe in the hydrometer rinse bath.

7.6.4 Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes, proceed to small sieve analysis.

With samples that are mostly sand, the hydrometer reading can drop below the reading of the blank. When this occurs, we will apply the lowest hydrometer reading to the blank reading. We will include this in the technical narrative. The logic behind this issue is due to the high amount of sand, resulting in more density. As a result, less DI water is added when transferring the sample/hexametaphosphate solution to the 1000 mL cylinders. Since the blank has a higher DI water/hexametaphosphate ratio, it increases the reading.

7.7 Sieve Analysis for Large and Small Sieves

Look at the sample material in the aluminum pan and record a description of the non-soil material (such as- sticks, grass, wood, plastic), hardness of material and shape of material in the logbook.

Hardness qualifiers include hard, soft or brittle.
Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

7.7.1 Weigh the 3/4", 1/4", #4 and #10 sieves and enter the weight measurements in the logbook as the tare weight.

7.7.2 Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 4 minutes. Weigh each sieve and record these measurements in the logbook.

Small Sieves

Completely transfer the sample from the graduated cylinder to a #200 wet wash sieve. Make sure the entire sample has been transferred to the #200 wet wash sieve by rinsing the graduated cylinder several time with DI water. Using DI water, wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve into a aluminum tin labeled with the sample's LAB ID.

7.7.3 Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

7.7.4 Samples are sieved through the #20, #40, #60, #80, #100, and #200.

7.7.5 After samples have cooled, stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 4 minutes. Weigh each sieve and record these measurements in the logbook.

7.8 Sieve Only Analysis

7.8.1 Determine the total solids of the sample. Use the Percent Solid Table (Table 1) to determine the sample size to be used. If total solids are not available depending on sample matrix we make an educated guess as to how much sample will be used. Samples are then soaked in DI overnight.

7.8.2 After sample and solution has soaked for a minimum of 16 hours, the sample slurry is rinsed into a dispersion cup using DI water. Fill the dispersion cup ½ full with DI water and place the cup on the blender to mix for one minute.

7.8.3 If the sample appears to have large gravel, sand, or organic material that does not appear to be amendable for using the blender notify the PM and proceed to the next step without blending.

7.8.4 Samples are then wet-washed through the #200 Sieve until samples run clear.

7.8.5 Samples are then completely transferred to a metal tin labeled with appropriate sample ID, and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the tin from the oven and allow it to cool.

7.8.6 Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

7.8.7 For Large Sieve analysis follow 7.7.1-7.7.2

7.8.8 For Small Sieve analysis follow sections 7.7.2-7.7.3

7.9 Calculations

7.9.1 Sample Used (SU): total dry sample

SU =Total Sample Weight ((100-%Moisture)/100)*

HMCF = Hygroscopic moisture correction factor (we assume 1)

TITLE: **Grain Size Analysis**

7.9.2 Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

7.9.3 Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1)
Time, minutes = Time of hydrometer reading from beginning of edimentation
Sqrt - square root
SG - Specific Gravity of soil (assuming a default SG)
Viscosity - is the resistance of a liquid to flow

Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

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)

TITLE: **Grain Size Analysis**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not Applicable

9.0 METHOD PERFORMANCE

Not Applicable

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM Standard D 422-63 (Re-approved 2007) “Standard Test Method for Particle-Size Analysis of Soils”, ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

Katahdin SOP SOP CA-717 “Total Solids/Total Volatile Solids Determination In Solid Matrices”, current revision.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018

LIST OF TABLES AND FIGURES

Table 1 Percent Solids Table for Weight Determination
Table 2 Summary of Method Modifications
Figure 1 Example of Logbook Page

TITLE: **Grain Size Analysis**

TABLE 1

PERCENT SOLIDS TABLE FOR WEIGHT DETERMINATION

Percent Solid Table
Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr.	% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	50	75	100	200		25	50	75	100	200
1	2500	5000	7500	10000	20000	51	49	98	147	196	392
2	1250	2500	3750	5000	10000	52	48	96	144	192	385
3	833	1667	2500	3333	6667	53	47	94	142	189	377
4	625	1250	1875	2500	5000	54	46	93	139	185	370
5	500	1000	1500	2000	4000	55	45	91	136	182	364
6	417	833	1250	1667	3333	56	45	89	134	179	357
7	357	714	1071	1429	2857	57	44	88	132	175	351
8	313	625	938	1250	2500	58	43	86	129	172	345
9	278	556	833	1111	2222	59	42	85	127	169	339
10	250	500	750	1000	2000	60	42	83	125	167	333
11	227	455	682	909	1818	61	41	82	123	164	328
12	208	417	625	833	1667	62	40	81	121	161	323
13	192	385	577	769	1538	63	40	79	119	159	317
14	179	357	536	714	1429	64	39	78	117	156	313
15	167	333	500	667	1333	65	38	77	115	154	308
16	156	313	469	625	1250	66	38	76	114	152	303
17	147	294	441	588	1176	67	37	75	112	149	299
18	139	278	417	556	1111	68	37	74	110	147	294
19	132	263	395	526	1053	69	36	72	109	145	290
20	125	250	375	500	1000	70	36	71	107	143	286
21	119	238	357	476	952	71	35	70	106	141	282
22	114	227	341	455	909	72	35	69	104	139	278
23	109	217	326	435	870	73	34	68	103	137	274
24	104	208	313	417	833	74	34	68	101	135	270
25	100	200	300	400	800	75	33	67	100	133	267
26	96	192	288	385	769	76	33	66	99	132	263
27	93	185	278	370	741	77	32	65	97	130	260
28	89	179	268	357	714	78	32	64	96	128	256
29	86	172	259	345	690	79	32	63	95	127	253
30	83	167	250	333	667	80	31	63	94	125	250
31	81	161	242	323	645	81	31	62	93	123	247
32	78	156	234	313	625	82	30	61	91	122	244
33	76	152	227	303	606	83	30	60	90	120	241
34	74	147	221	294	588	84	30	60	89	119	238
35	71	143	214	286	571	85	29	59	88	118	235
36	69	139	208	278	556	86	29	58	87	116	233
37	68	135	203	270	541	87	29	57	86	115	230
38	66	132	197	263	526	88	28	57	85	114	227
39	64	128	192	256	513	89	28	56	84	112	225
40	63	125	188	250	500	90	28	56	83	111	222
41	61	122	183	244	488	91	27	55	82	110	220
42	60	119	179	238	476	92	27	54	82	109	217
43	58	116	174	233	465	93	27	54	81	108	215
44	57	114	170	227	455	94	27	53	80	106	213
45	56	111	167	222	444	95	26	53	79	105	211
46	54	109	163	217	435	96	26	52	78	104	208
47	53	106	160	213	426	97	26	52	77	103	206
48	52	104	156	208	417	98	26	51	77	102	204
49	51	102	153	204	408	99	25	51	76	101	202
50	50	100	150	200	400	100	25	50	75	100	200

TITLE: **Grain Size Analysis**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-551-02	METHOD ASTM D 422-63
Procedures		
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Accuracy/ Precision		

TITLE: **Grain Size Analysis**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Katahdin Analytical Services, LLC.
Sediment Grain Size - Method ASTM D422

Lab Sample ID	5M2456-4	Start Date/Time	3-20-19: 11:51
Analyst:	JMS	End Date/Time	4-2-19: 12:44
Sample Description:	Muck/organics - removed 35.1g sticks/grass/roots		
Sample Weight	Sample (g)	Date/Time in oven	See pg. 1
Sample Weight (wet)	~~125.0~~ 89.9	Date/Time out of oven	
Sample Weight (dried)	25.98		
		Hydrometer Data	
% Moisture	71.105	Serial Number	152303
		Cal Date:	~~3/25/19: 11:32~~
Sample Split (Oven Dried)	Sample (g)	Low Temp C	16.4
Sample >=#10	0	Low Temp Reading	1.0035
Sample <=#10	25.98	High Temp	17.6
		High Temp Reading	1.0035
Sieve Only:			
Sieve Only, Wet Wash:			
		Soil Gravity	2.65

Gravel/Sand Fraction (Sieves)			
Sample Fraction	Size (um)	Pan Tare	Pan+Sample
3"	75000		
2"	50000		
1.5"	37500		
1"	25000		
3/4"	19000		
1/4"	6300		
#4	4750		
#10	2000		
#20	850	307.2	307.5
#40	425	270.0	270.6
#60	250	248.5	249.0
#80	180	241.9	242.1
#100	150	236.5	236.7
#200	75	315.2	315.8
Pan	Pan	339.9	340.3 Dry

11:45

Silt/Clay Fraction (Hydrometer Test)				
Time (min)	Proposed Read Time	Actual Time (min)	Temp C	Spec. Gravity
2	11:50	11:50 (2)	17.2	1.0155
5	11:53	11:53 (5)	17.0	1.0130
15	12:03	12:03 (15)	17.5	1.0115
30	12:18	12:17 (29)	17.3	1.0105
60	12:48	12:48 (60)	16.6	1.0100
240	15:48	15:48 (240)	16.5	1.0075
1440	11:48	11:44 (1441)	17.5	1.0060

EX-028 – Revision 3 – 03/19/2019

QAEX379

0000002

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-604
Revision History
Cover Page
Page 1

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Burton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Neufeld Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Figures 2 and 3. Changed KAS, INC. to KAS.	LAD	06/15	06/15
07	Sect. 1 - Replaced beaker with digestion vessel. Sect. 7.5 & 7.9 - Added calibrated Pipet.	LAD	06/16	06/16
08	Update figure 1. Change title of section 5.0 to Reagents and Standards. Update method references for NELAC and DoD.	LAD	09/17	09/17
09	Added Thorium to Tables 2 and 3 Sect. 8- Added contingency plan.	LAD	01/19	01/19

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-09**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-09**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever digestion vessels are being heated in the hood. Use caution when handling hot digestion vessels.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter or calibrated pipet, to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter or calibrated pipet to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final

volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

LIST OF TABLES AND FIGURES

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Table 3 Procedure Condensation
Figure 1 Example Page From Metals Sample Preparation Logbook
Figure 2 Preparation of Matrix Spikes, LCSs, and Spiking Solutions: Method 3010
Figure 3 Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-09	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

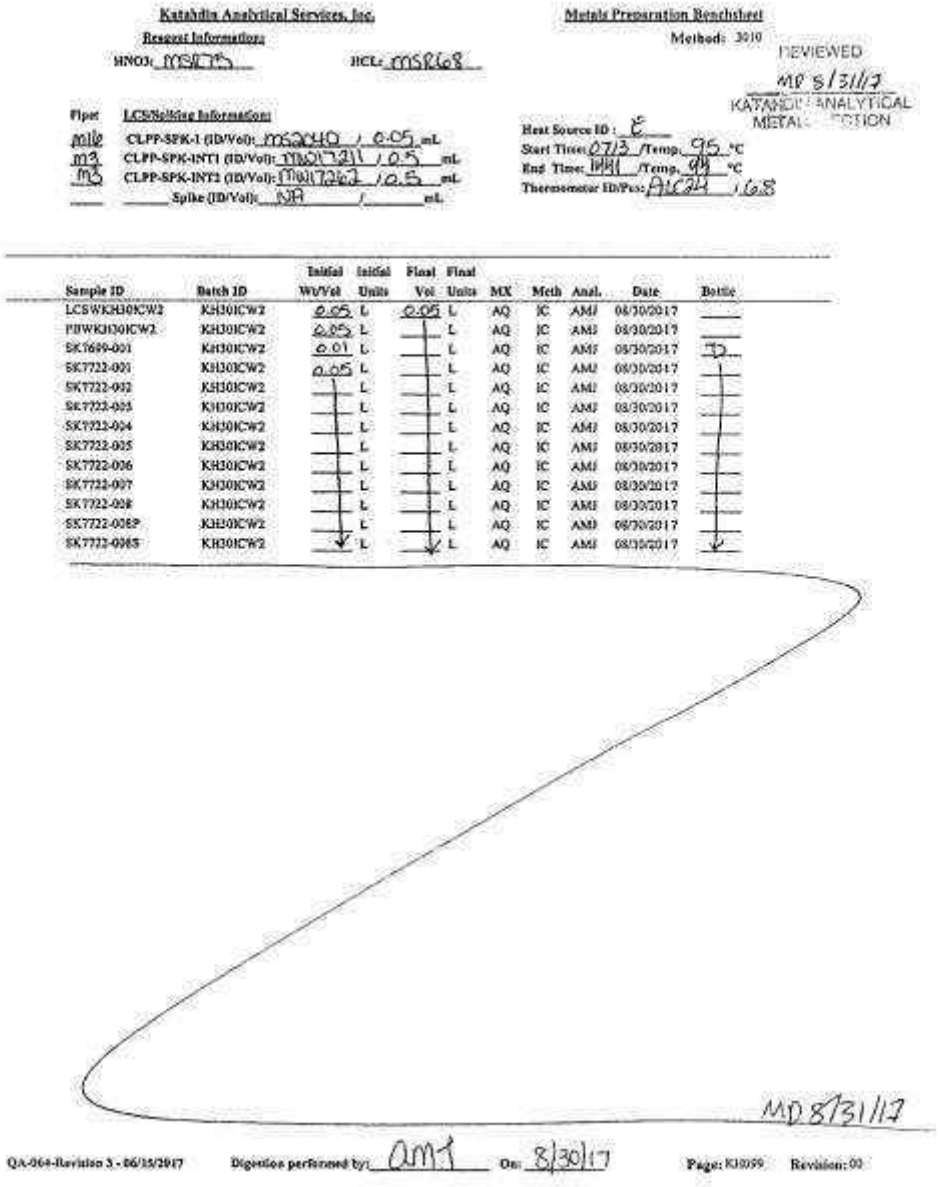
PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:

HNO3: MSR75 HCL: MSR68

Method: 3010

REVIEWED
MD 8/31/17
KATAHDIN ANALYTICAL
METALS SECTION

Pipet	LCS/Spiking Information:
m16	CLPP-SPK-1 (ID/Vol): MS2040 / 0.05 mL
m3	CLPP-SPK-INT1 (ID/Vol): MW17211 / 0.5 mL
m3	CLPP-SPK-INT2 (ID/Vol): MW17262 / 0.5 mL
	Spike (ID/Vol): NA / mL

Heat Source ID: E
Start Time: 0713 /Temp. 95 °C
End Time: 1441 /Temp. 94 °C
Thermometer ID/Pos: AL24 / 68

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWKH30ICW2	KH30ICW2	0.05	L	0.05	L	AQ	IC	AMJ	08/30/2017	
PBWKH30ICW2	KH30ICW2	0.05	L		L	AQ	IC	AMJ	08/30/2017	
SK7699-001	KH30ICW2	0.01	L		L	AQ	IC	AMJ	08/30/2017	D
SK7722-001	KH30ICW2	0.05	L		L	AQ	IC	AMJ	08/30/2017	
SK7722-002	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-003	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-004	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-005	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-006	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-007	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008P	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008S	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	

MD 8/31/17

QA-064-Revision 3 - 06/15/2017 Digestion performed by: AMJ On: 8/30/17 Page: KH059 Revision: 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10,000 mg/L K	High Purity Standards	10.0
	10,000 mg/L Na	High Purity Standards	7.5
	10,000 mg/L Mg	High Purity Standards	5.0
	10,000 mg/L Ca	High Purity Standards	2.5
	1000 mg/L Tl	High Purity Standards	1.0
CLPP-SPK-INT2	1000mg/L Sr	High Purity Standards	5.0
	1000mg/L Sn	High Purity Standards	5.0
	10,000mg/L Si	High Purity Standards	1.0
	1000mg/L B	High Purity Standards	5.0
	1000mg/L Li	High Purity Standards	5.0
	1000mg/L Ti	High Purity Standards	5.0
	1000mg/L Mo	High Purity Standards	1.0
	1000mg/L U	High Purity Standards	1.0
	1000mg/L W	High Purity Standards	1.0
	1000mg/L Th	High Purity Standards	1.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION		
	CLPP-SPK-1	CLPP-SPK-INT1	CLPP-SPK-INT2
Aluminum	2000		
Antimony		10	
Arsenic		10	
Barium	2000		
Beryllium	50		
Boron			50
Cadmium		25	
Calcium		250	
Chromium	200		
Cobalt	500		
Copper	250		
Iron	1000		
Lead		10	
Magnesium		500	
Manganese	500		
Molybdenum			10
Nickel	500		
Potassium		1000	
Selenium		10	
Silicon			100
Silver	50		
Sodium		750	
Strontium			50
Thallium		10	
Tin			50
Titanium			50
Uranium			10
Vanadium	500		
Zinc	500		
Lithium			50
Tungsten			10
Thorium			10

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-605
Revision History
Cover Page
Page 1

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Jol C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Dennis F. Neufeld Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3 + 4 to reflect current practice.	DN	8.29.02	8.29.02
03 3050B	New Title to include ILMOS.3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1 g water to 1.0 g boiling chips. H_2O_2 addition from 3.0 ml then 7.0 mls to 3.0 ml, 2.0 ml then 7.0 ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09
05	Updated Tables 3 and 4 to reflect current spiking procedures.	LAD	09/10	09/10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 7 – Added wording concerning subsampling. Table 3 and 4 – Corrected standard concentrations. Attachment A - Modifications For 8330B Preparation & Digestion. Changed KAS INC. to KAS throughout	LAD	08/15	08/15
07	Update Figure 2. Change title of Section 5.0. Update method references for NELAC + DoD. Minor changes to Table 1 + Section 8.2.	LAD	09/17	09/17
08	Updated Table 3 to include Thallium and corrected Standard concentration of U + W. Updated Table 4 to include Thallium Sect. 8 - added Contingency Plan. Sect 10 updated references	LAD	01/19	01/19

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-605-08**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-605-08**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS.**

Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSO – Laboratory Control Sample for Solids – An aqueous standard that had been brought through the sample preparation process.

LCSS – Laboratory Control Sample for Solids – A solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with

the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from the Environmental Health and Safety Officer, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of $95^{o}C \pm 5^{o}C$. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature

of each hot plate used is measured and recorded each day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated ($4^oC \pm 2^oC$) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Avoid rocks, roots, leaves and other organic or inorganic foreign material. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet.

Refer to Katahdin Analytical Services SOP CA-108, current revision "Basic Laboratory Technique" for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95°C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95°C ± 5°C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 Prepare an appropriate number of laboratory control samples (LCSO for aqueous LCS or LCSS for solid LCS reference material) by weighing appropriate masses of solid reference material or by spiking the LCSO as described in Table 3. The analyte concentrations of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

LIST OF TABLES AND FIGURES

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Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
Figure 1 Procedure Condensation – Method 3050
Figure 2 Example Page from Metals Sample Preparation Logbook
Figure 3 Example Certificate of Analysis for Solid Reference Material
Attachment A Modifications For 8330B Preparation & Digestion

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Aqueous or Solids (LCSO or LCSS)	One each per prep batch of 20 or fewer samples, if specified by project or client	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-08	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures(IV)	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L As,Pb,Sb,Se,Tl	High Purity Standards	1.0 each
	1000 mg/L Cd	High Purity Standards	2.5
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	1000 mg/L Mo	IV or High Purity Standards	1.0
	1000 mg/L B,Li,Sn,Sr,Ti	IV or High Purity Standards	5.0 each
	10000 mg/L Si	High Purity Standards	1.0
	1000 mg/L U	High Purity Standards	1.0
	1000 mg/L W	High Purity Standards	1.0
	1000 mg/L Th	High Purity Standards	1.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

	CONCENTRATION IN SOLUTION, mg/L			
Element	**Matrix Spike**	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**CLPP-SPK-INT2**
Aluminum	2.000	2000		
Antimony	0.100		10	
Arsenic	0.100		10	
Barium	2.000	2000		
Beryllium	0.050	50		
Boron	0.500			50
Cadmium	0.250		25	
Calcium	2.500		250	
Chromium	0.200	200		
Cobalt	0.500	500		
Copper	0.250	250		
Iron	1.000	1000		
Lead	0.100		10	
Lithium	0.500			50
Magnesium	5.000		500	
Manganese	0.500	500		
Molybdenum	0.300			10
Nickel	0.500	500		
Potassium	10.000		1000	
Selenium	0.100		10	
Silicon	5.000			100
Silver	0.050	50		
Sodium	7.500		750	
Strontium	0.500			50
Thallium	0.100		10	
Tin	0.500			50
Titanium	0.500			50
Tungsten	0.100			10
Uranium	0.100			10
Vanadium	0.500	500		
Zinc	0.500	500		
Thorium	0.100			10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet — Method: 3050B

Reagent Information:

HNO3: MSR75 HCL: MSR68 H2O2: MSR62 Filter Paper: 9782497/9831914

Boiling Stones: MSR53

Plate	LCS / Spike	LCS/Spiking Information:
M10	☑ ☑	CLPP-SPK-1 (ID/Vol): MS26440 / 0.1 mL
M3	☑ ☑	CLPP-SPK-INT1 (ID/Vol): MW17211 / 1.0 mL
M3	☑ ☑	CLPP-SPK-INT2 (ID/Vol): MW17262 / 1.0 mL
—	☐ ☐	Spike (ID/Vol): NA / mL

LCSS: NA Balance ID: Bal15

Heat Source ID: E

Start Time: 0749 /Temp. 91 °C

End Time: 1421 /Temp. 94 °C

Thermometer ID/Pos: AUE24 / 4.1

REVIEWED
MD 9/1/17
KATAHDIN ANALYTICAL
METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOKI01ICS1	KI01ICS1	1.0	g	0.1	L	SL	IC	AMJ	09/01/2017	—
PBSKI01ICS1	KI01ICS1	1.0	g		L	SL	IC	AMJ	09/01/2017	
SK7769-001	KI01ICS1	1.88	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001F	KI01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001S	KI01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7789-001	KI01ICS1	1.80	g		L	SL	IC	AMJ	09/01/2017	G
SK7794-001	KI01ICS1	1.95	g		L	SL	IC	AMJ	09/01/2017	A
SK7794-002	KI01ICS1	1.98	g		L	SL	IC	AMJ	09/01/2017	"
SK7794-003	KI01ICS1	1.92	g		L	SL	IC	AMJ	09/01/2017	"
SK7837-001	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-002	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-003	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-004	KI01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-005	KI01ICS1	1.0	g		L	AR	IC	AMJ	09/01/2017	"
SK7837-006	KI01ICS1	1.0	g	↓	L	AR	IC	AMJ	09/01/2017	↓

MD 9/1/17

QA-06S-Revision 2 - 06/16/2017 Digestion performed by: AMJ On: 9/1/17 Page: KI001 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	13000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	328	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	113	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 158
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4279
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	13000*	2840	2160 - 3520
selenium	146	135	104 - 165
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	328	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	146	104 - 275
titanium	3100*	288	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

ATTACHMENT 1

MODIFICATIONS FOR 8330B PREPARATION & DIGESTION

4.0 APPARATUS AND MATERIALS – additional materials

4.1 Sieves - #10 mesh (2 mm) – 8" diameter with covers and collection trays.

4.2 Aluminum drying trays with drying rack

4.3 Heavy duty aluminum foil

4.4 Stainless steel scoopulas

4.5 Dust mask

9.0 PROCEDURES – additional procedures

Prior to the digestion of samples (section 7.1 in SOP):

Spread the entire aliquot of soil onto a drying tray lined with heavy duty aluminum foil and dry in air at room temperature or colder to a constant weight (last two successive dry weights within 3% RPD). Trays should be placed in rack for drying. Record all weights in the Sample Drying Logbook.

Note: Hydric soils and sediments with high moisture content may take several days to dry to constant weight.

Remove the oversize fraction by passing it through a 10-mesh (2 mm) sieve. Be sure to break up caked up soil with a gloved hand. Weigh both fractions – oversize and <2mm. Record all weights in the Sieving & Grinding Logbook.

To obtain a subsample, the entire sample must be mixed with a stainless steel scoopula and spread out on a clean surface (aluminum tray lined with foil) so that it is only 1 or 2 cm thick - preferably in a fume hood designed to prevent the spread of dust and possible inhalation or residue losses. Using the scoopula, obtain at least 30 different increments, i.e., portions (~0.3 g) from randomly chosen locations throughout the entire sample profile for a total of ~10 g. Mix this subsample one more time with the scapula and then obtain an aliquot for metals digestion (beginning with section 7.3).

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Bunton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hufnagle Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (Cal blank) followed by S1 (Mixed Cal Std.) Changes to section 7.5 and Table 3 to reflect this. Made changes to element LOICS in Tables 3, 4, 5, 6 to reflect current practices.	DH	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chkstd. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current Standard concentrations and prep. Updated Table 1 with current practices including NAVY audit findings. Updated Sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up Updated tables to reflect changes in Standard Concentrations and Preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect Changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for Compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10
11	Added yttrium criteria to section 7 and Table 1.	LAD	06/10	06/10
12	Revised Tables 4 → 8 with the following information: -Add palladium and gold; removed tungsten and uranium; removed Stock Standard ~~ICP-CICV~~ 2007ICS-1, changed stock standard ICP-CICV-3 to CL-CAL-3. Added references to section 10.	LAD 09/22/11 LAD	09/11	09/11
13	The changes above had not been finalized in SOP-12. Sect. 9 - Added MDL, LOD and LOQ information. Added Attachment 2 - Analysis of palladium by SW846 6010	LAD	04/12	04/12

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 9 & Table 1 - Fixed typos.	LAD	05|13	05|13
15	Sect. 10- Updated references. Added Table 3 - DoDQSM 5.0 QC Requirements- Renumbered rest of Tables. Updated Tables(6→8). Changed KAS INC to KAS LLC.	LAD	12|14	12|14
16	Sect. 5 & 7 - corrected Table references. Tables 5, 6, 7 & 8 - Updated Standard, Concentrations & sources. Changed KAS LLC to KAS	LAD	05|16	05|16
17	Sect. 1 and 6 - Added Tissue matrix	LAD	07|16	07|16
18	Sect. 8.1 - Changed reagent spiked water to calibration blank solution. Sect. 10 - update method references.	LAD	09|17	09|17
19	Updated Tables to correct concentrations for a few elements. Removed Table 2, DODSM 4.2 QC Requirements. Renumbered Tables	LAD	01|19	01|19

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-19**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-19**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, LLC personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, biological tissue and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution

ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to 500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document

their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual l and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of

the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 4 and 5 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months
Tissue	P, G	25 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multi-element calibration standard (S1 in Table 4) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Tables 1 through 3 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Tables 1 through 3 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and

project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed. Tables 2 and 3 list the QC Check, minimum frequencies, acceptance criteria, corrective actions, flagging criteria and additional comments for work analyzed in accordance with DoD QSM versions 4.2 and 5.0.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of calibration blank solution, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared 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. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements

may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within $\pm$ PQL if the PQL is greater than 0.01 mg/L, within $\pm$ 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run,

unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM 4.2 acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. If DoD QSM 5.0 acceptance criteria are being used, recovery for water and solid matrix samples must fall between the limits stated in Tables 3 & 4 of the QSM. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in

all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 6010C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ® ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the LLCCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 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.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
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.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CVs. 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 analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	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. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike(MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD)	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-18	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 6)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	Calibration Standard 3	Claritas PPT	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	Inorganic Ventures	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn, Au	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 6)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 6)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 6)	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L Li, Sn, Au	High Purity Standards	1.0 each
	10000 mg/L K, Na	High Purity Standards	1.0 each
	1000 mg/l B	High Purity Standards	0.50
	1000 mg/l Zn	High Purity Standards	0.20
	1000 mg/L Cu	High Purity Standards	0.25
	10000 mg/L Si	High Purity Standards	0.20
	1000 mg/L Ti, Tl	High Purity Standards	0.15 each
	1000 mg/L Se, Mo, Co, Ni, Ag, Sr, V, Cr	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba, Be, Cd, Mn, Pb	High Purity Standards	0.05 each
	10000 mg/L Fe, Ca, Mg	High Purity Standards	0.1 each
ICSAB-INT	1000 mg/L K,Na	High Purity Standards	4.0 each
	1000 mg/L B, Li, Mo,Sr,Sn,Ti, Au	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.40
ICP-INT STD (Intermediate)	10000 mg/L Si	High Purity Standards	2.5
	10000 mg/L Ca, Mg, Fe, Al, Na	High Purity Standards	2.4
	10000 mg/L K	High Purity Standards	1.5
	1000 mg/L Au, Li, Sn. Sr	High Purity Standards	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L							
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**
Aluminum	25	10	0.3	500	500	12.5	500	
Antimony	1	0.4	0.008		0.6	0.5		
Arsenic	1	0.4	0.008		0.1	0.5		
Barium	1	0.4	0.005		0.5	0.5		
Beryllium	1	0.4	0.005		0.5	0.5		
Boron	1	0.4	0.05		0.5	0.5		
Cadmium	1	0.4	0.005		1.0	0.5		
Calcium	25	10	0.10	500	500	12.5		
Chromium	1	0.4	0.01		0.5	0.5		
Cobalt	1	0.4	0.01		0.5	0.5		
Copper	1	0.4	0.025		0.5	0.5		
Iron	25	10	0.1	200	200	12.5		200
Lead	1	0.4	0.005		0.05	0.5		
Lithium	1	0.4	0.1		0.5	0.5		
Magnesium	25	10	0.10	500	500	12.5		
Manganese	1	0.4	0.005		0.5	0.5		
Molybdenum	1	0.4	0.01		0.5	0.5		
Nickel	1	0.4	0.01		1.0	0.5		
Potassium	25	13.6	1		20	12.5		
Selenium	1	0.4	0.01		0.05	0.5		
Silicon	25.5	10.0	0.2		2	12.75		
Silver	1	0.4	0.01		0.2	0.5		
Sodium	25	10	1		20	12.5		
Strontium	1	0.4	0.01		0.5	0.5		
Thallium	1	0.4	0.015		0.1	0.5		
Tin	1	0.4	0.1		0.5	0.5		
Titanium	1	0.4	0.015		0.5	0.5		
Vanadium	1	0.4	0.01		0.5	0.5		
Zinc	1	0.4	0.02		1.0	0.5		
Gold	1	0.4	0.1		0.5	0.5		

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L		
	ICP Intermed STD	PQL-INT	ICSAB-INT
Aluminum	240	30	
Antimony		0.8	
Arsenic		0.8	
Barium		0.5	
Beryllium		0.5	
Boron		5	10
Cadmium		0.5	
Calcium	240	10	
Chromium		1.0	
Cobalt		1.0	
Copper		2.5	
Iron	240	10	
Lead		0.5	
Lithium	10	10	10
Magnesium	240	10	
Manganese		0.5	
Molybdenum		1.0	10
Nickel		1.0	
Potassium	150	100	400
Selenium		1.0	
Silicon	250	20	40
Silver		1.0	
Sodium	240	100	400
Strontium	10	1.0	10
Thallium		1.5	
Tin	10	10	10
Titanium		1.5	10
Vanadium		1.0	
Zinc		2.0	
Gold	10	10	10

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	IV-28	QCS-26	CLPP-ICS-A	CLPP-ICS-B4	CL-CAL-3
Aluminum	100	100	5000		
Antimony	100	100		60	
Arsenic	100	100		10	
Barium	100	100		50	
Beryllium	100	100		50	
Boron	100	100			
Cadmium	100	100		100	
Calcium	100	100	5000		1000
Chromium	100	100		50	
Cobalt	100	100		50	
Copper	100	100		50	
Iron	100	100	2000		1000
Lead	100	100		5	
Lithium	100				
Magnesium	100	100	5000		1000
Manganese	100	100		50	
Molybdenum	100	100			
Nickel	100	100		100	
Potassium	1000	1000			1000
Selenium	100	100		5	
Silicon	50	50			
Silver	100	100		20	
Sodium	100	100			1000
Strontium	100				
Thallium	100	100		10	
Tin					
Titanium	100	100			
Vanadium	100	100		50	
Zinc	100	100		100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 2

ANALYSIS OF PALLADIUM BY SW846 6010

Palladium may be analyzed by EPA Method SW846 6010C following the method outlined in this SOP. However, due to significant spectral interferences caused by addition of palladium to the calibration and check standards used in this method, palladium is added to aliquots of the regular standards as needed for analysis. Two stock standards (1000 mg/L) are currently kept for palladium analysis. One is purchased from High Purity Standards and is used for calibration, PQL, ICSAB, and CCV. The other is purchased from Inorganic Ventures and is used as the independent check standard (ICV). Analysts should add palladium stock to the regular standards according to the table below:

Name of Working Standard	Volume of Standard Aliquot (mL)	Volume of Palladium Stock Added (mL)	Concentration of Palladium (mg/L)	Source of Palladium Stock
Calibration Std.	50	0.05	1.0	High Purity
ICV	50	0.02	0.4	Inorganic Ventures
PQL	50	0.005	0.1	High Purity
ICSAB	50	0.025	0.5	High Purity
CCV	50	0.025	0.5	High Purity

Prior to starting the run, a palladium-only standard should be analyzed along with the iron and aluminum standards to evaluate interelement correction factors as outlined in Katahdin SOP CA-632, Section 7.1.

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Joh C. Burton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03·27·01

General Manager: Deborah J. Nadeau Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. Changed Run ID Naming convention added data reduction and reporting procedures updated standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tubing size Sect. 5 - changed acid conc.s Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated Tables 1, 3 → 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4.5 and 6 with current standards. Updated Table 1 with serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL mininum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.18-changed instrument identifier to reflect new instrument; section 8-changed acceptance criteria and ICSAB analyte list; Table 1-updated acceptance criteria and corrective action to QC. Table 3-added all analytes to list-removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 602A. Added Hardness by calculation attachment. Added LLQC requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 8 and QC Tables - Added DoD QSM references and criteria. Section 10 - Added references. Tables 4 → 7 - Added information pertaining to CCV conc. change	LAD	08/09	08/09
07	Added Table 2 with DoD QSM ver. 4.1 QC requirements. Updated Section 4.1, Table 10 and Table 11 with new autosampler information.	LAD	04/10	04/10
08	Sect. 1.1 - Added definitions. Sect. 4.1, 4.2, 5.2 7.9, 7.10, 7.11, 7.16 and 8.7 - minor changes to reflect current practice. Sect. 9 - added MDL, LOD and LOQ information. Sect. 10 - Added, ~~edited references~~. Updated Tables 1, 5, 6, 7, and 8 edited references LAD 042512	LAD	04/12	04/12
09	Sect. 7 - Added reference to autosampler software, added printing calibration and removed printing of run summary	LAD	08/13	08/13
10	Sect. 7 - Updated for changes made in the metals database for importing and handling data. Sect. 10 - updated and added references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 7, Table 1, 2, 3, 6, 8 & 11 - Updated to reflect change from 5 pt. to 2 pt. calibration. Table 7, 8 & 9 - Updated to reflect change in Aluminum PQL	LAD	04/16	04/16
12	Change title of Section 5.0. Update method references for NELAC + DoD. Minor additions/corrections to sections 3.0, 4.2, 7.3.5 and table 5.	LAD	09/17	09/17
13	Table 1 - Added ms/msD, corrected section references - Table 2 - Removed, DoD QSM 4.2 QC Requirements. Renumbered subsequent sections. Table 7 - Added Thorium - Updated Table references throughout SOP	LAD	01/19	01/19

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as “analytes” in Table 4. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions. If Method 6020 is used to determine any analyte not listed in Table 4, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

Internal Standard - Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. Internal standards must be analytes that are not native to the sample.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LDR - Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.
LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon

tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7500 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total (“acid-leachable”) values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7500 ChemStation data system is used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) = (m/z 75 signal) - (2.95) (m/z 77 signal) + (2.548) (m/z 82 signal) – (2.571) (m/z 83 signal), where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).

Similarly:
Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) = (m/z 114 signal) - (0.027) (m/z 118 signal) - (1.84)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 4. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been

found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7500 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used should differ from the analyte of interest by no more than 50 amu. See Table 14 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7500 ICP-MS system, consisting of the Agilent 7500 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7500 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7500 ChemStation software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment includes a Elemental Scientific SC-4 PX Fast Autosampler and Sample

Introduction system, and Bullzip PDF printer set to print to file ICPMS_CP.pdf located in folder PDF_PRINTS on the desktop.

4.2 Peristaltic pump tubing – 3-stop ESI PVC flared black-black (0.76 mm ID) and orange-green-orange (0.38 mm ID). 2-stop ESI PVC flared red-red (1.14 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable-volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area, designed for trace element sample handling must be used. Standards, samples and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker “Instra-Analyzed” trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All

acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Laboratory reagent grade water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 6 and 7 for a listing of stock standards required, and to Table 9 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 8 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 5 for a listing of working standards and for preparation instructions. Refer to Table 7 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent Chemstation software. The main Chemstation screen is accessed by double-clicking the “ICP-MS Top” icon on the Windows desktop. Autosampler tables are edited by selecting “Edit Sample Log Table” from the Sequence menu in the Agilent Chemstation software. In the following discussion, software menu items that are to be selected are printed in boldface. The instrument operating conditions, acquisition parameters, acquisition masses, and internal standards for analysis USEPA Method 6020 are detailed in Table 11.

7.2 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.3 Turn on the water chiller/recirculator.

7.4 Verify that the exhaust hood is in operation.

7.5 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.6 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.7 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.8 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.9 Open ESI autosampler software by double-clicking the "ESI SC" icon. Open the Chemstation software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting **Instrument>>Instrument Control>>Plasma>>Plasma On** and allow the instrument to aspirate calibration blank solution for at least 45 minutes. During this warm-up, select **Tune>>Sensitivity>>Start** to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.10 After the 45 minute warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.11 Generate a tune report by selecting **Tune>>File>>Generate Report**. Evaluate the tune report against the tune specifications listed in Table 12. If the tune passes all specifications, proceed to step 7.14.

7.12 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting **Tune>>Autotune>>Run**. The Chemstation software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 13.

7.13 Repeat step 7.12 until all tune specifications have been met. File the final tune report.

7.14 Aspirate the P/A tuning solution (see Table6) and run a P/A auto tune by selecting **Tune>>Tune>>P/A Factor>>Run**. This will calibrate the pulse and analog modes of the detector. File the P/A report with the Tune report.

7.15 Load the instrument analytical method and calibrations table for USEPA Method 6020 into memory by selecting:

Methods>>Load>>K1PTCAL16.M>>K1PTCAL16.C.

7.16 Edit the sequence template "K6020.S" to create an analytical sequence table listing all of the samples to be analyzed. To do this, select "Edit Sample Log Table" from the **Sequence** menu in the Agilent Chemstation software. Double-click **SMPL** from the menu at the top left. Fill in the sample table with sample IDs, vial numbers, analytical method (K1PTCAL16.M for all samples), dilution factors, and failure actions. When the sample table is complete, select **Print** to print this table. Close the "Edit Sample Log Table" window. Save the sample log table under a new name by selecting **Save** under the **Sequence** menu and then typing the name.

7.17 Load the autosampler racks according to the analytical sequence printout.

7.18 Select **Sequence>>Load and Run Sequence**, and select the appropriate autosampler sequence table from the displayed list. Enter the analyst's initials in the Operator box. Change data file name to appropriate designation. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("J" for the Agilent 75000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("G" for 2013, "H" for 2014, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "JGA16A" is the first run of the day that was performed on January 16, 2013, using the Agilent 7500 ICP-MS. Select **Run**. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 10 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least one multielement calibration standard to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.19 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.20 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.21 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.22 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.23 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.24 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.25 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.26 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.27 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.28 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.29 Follow these steps to create the data import file: Open the FileView program using the "FIVIEW" icon on the Windows Desktop. Select "Data" in left window. Select the data file of interest and double click to move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.30 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K2008.sbl" from this list of options and click "open." Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.31 Select "Quant Info" from the top menu and select "Quant Results" from the displayed options. This will display the data in concentration units.

7.32 Select the "Transpose" from the menu. Click on "file" within the chart to highlight the data.

7.33 Select "Tools" from the top menu and "Copy Selected Data to CSV File" from this list of options. Set the name to the file as "FileName.CSV", e.g., "JGA28A.CSV". Save the file to the ICP-MS DATA folder on metals on server_a.

7.34 Rename the pdf file to the appropriate file name in the PDF_Prints window and save to J-ICMS-Data file in My Network Places. Right click on ICPMS_CP.pdf icon to copy and paste blank file into PDF_Prints window for the next run.

To import data into the Metals Database:

7.35 Open the data file from metals on Server_a. Replace dashes in Cal Blank line with zeros. Replace dashes in Cal Std 1 line with 50 for most all elements. Change aluminum and silicon with 1000 and change sodium, magnesium, potassium, calcium, and iron with 10,000. All cells under metals with ###, replace with 999999. Save file in ICP-MS Data folder on Metals on Server a. Select the "ICPMS Import" icon from the Windows Desktop, the ICPMS Import window will appear. Enter the datafile name without extension, (e.g., "JGA28A") and click "ok."

7.36 When the “Import finished” message appears, close the ICPMS Import window and select the “KIMS_METALS” icon from the Windows Desktop. The Metals Database Main Menu will appear. Select Additional Data Handling and then select Accept Samples by Element. Type in file name and reject any items that fail run QC.

7.37 Select the “Reporting Menu” button. From the Reporting Menu screen select the Batch QC Menu button and then the “Calculate Batch QC” button.

7.38 From the resulting list of QC results, deselect any items that fail run QC. Click on the “Accept Selected Batch QC” button.

7.39 From the Metals Main Menu, select the “Additional Data Handling” button. The Data Menu will appear. Select the “Report Added Compounds” button.

7.40 From the resulting list of sample results, deselect any items that fail run QC. Click on the “Accept Data” button.

7.41 Once all associated data from an analysis run have been processed, go to the RUNLOG INFO table of the metals database. Sort for the file of interest. Add lines for the 6020 and 200.8 Method Tunes. Change the time column accordingly. Go to the Generate Coverage portion of the Export Menu and print the Run Log and Logbook Page for the analysis run.

7.42 To extract Tune Reports and P/A Factor Tuning Report click on metpdf on Imageserver icon. Select J-ICPMS Data folder and select file on interest. Select Document drop down menu>pages>extract>select page numbers and click ok. Close document and save in metdpdf on “imageserver (P:) in J-ICP-MS-INST Tune folder as Filename+Tune.

7.43 Remove “blanks” and “rinses” from pdf file by selecting Document drop down menu>pages>delete>select appropriate pages at the beginning and end of report. Save document with ”RAW” added to the end of the file name. Save in the “ICPMS DATA” section of the “METPDF” directory on the IMAGESERVER.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific

judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least one multielement calibration standard. For each element,

calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where: Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 70 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements in samples bracketed by the failing CCV may not be reported, unless the CCV recovery is greater that 110% and the sample result is less that the PQL. For DoD analyses, results may not be reported without a valid CCV or report flagged results if reanalysis is not possible.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples

analyzed are for the Department of Defense (80% to 120% recovery limits) or other client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as “estimated” with a “J”.

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high

concentrations, and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

8.12 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12.1 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12.2 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.13 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.14 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.16 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine

the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6020A.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Inc., 2000.

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TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	If more than 1 caibration std is used, correlation coefficient (r) $\geq$ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample $\geq$ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit.
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use bracketed sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if $\geq$ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within $\pm$ 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within $\pm$ 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank conc.
Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Matrix Spike Sample (S), or Matrix Spike Duplicate Sample (P)	**At least one per digestion batch of 20 or fewer samples.**	**Recovery within ±25% of true value, if sample <4x spike added.**	**Flag results**
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section **8.13**	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 20% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 70%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	Daily.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration = 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICALhas passed.
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.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the 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 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 analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low Level ICV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the LOQ.
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% 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,	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
			correct problem, and rerun all associated failed field samples.		
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. Flagging is not appropriate.	Results may not be reported without a valid calibration blank.	For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep 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. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).			
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-13	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 4

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	$^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571$ [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	$^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764$ [See note 3] $^{114}Cd = (114)*1 – (118)*0.0268 – (108)*1.84$ [See note 4]
	Calcium	Ca	44	$^{44}Ca = (44)*1 – (88)*0.0169$ [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	$^{54}Fe = (54)*1 – (52)*0.0282$ [See note 8] $^{57}Fe = (57)*1 – (43)*0.03$ [See note 9]
	Lead	Pb	206, 207, 208	$^{208}Pb = (208)*1 + (206)*1 + (207)*1$ [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	$^{98}Mo = (98)*1 – (99)*0.146$ [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	$^{82}Se = (82)*1 – (83)*1.009$ [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	$^{51}V = (51)*1 – (53)*2.95 + (52)*0.378$ [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	$^{115}In = (115)*1 – (118)*0.016$ [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 4 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g “(51)”, indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Continuing Calibration Verification CCV (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z	Lab Prepared	0.50
	ICP-MS CAL 1	Lab Prepared	1.25
Calibration Standard (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.50
	ICP-MS-MIX-Z	Lab Prepared	1.0
	ICP-MS CAL 1	Lab Prepared	2.5
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W Solution (0.5mL each per 50mL and use same day only)	Inorganic Ventures	0.200
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	ICP-MS-CAL 1	Lab Prepared	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Co, Cr, Mo, Mn, Pb, Sb, Sr, U, V	High Purity Standards	0.02
	10,000 mg/L Al, K, Na	High Purity Standards	0.002
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
	Conc. HNO_2	Baker Instra Analyzed	4
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0
	Internal Standard Mix 1	Spex Industries	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate (5% HNO_2/5%HCL)	10,000 mg/L K, Na	High Purity Standards or Inorganic Ventures	2.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	1000 mg/L B	High Purity Standards or Inorganic Ventures	0.40
	10,000 mg/L Al, Ca, Fe, Mg 1000 mg/L Zn	High Purity Standards	0.20 of each
	1000 mg/L As, Se, V, W, Sr, Sn, Mo, Cr	High Purity Standards or Inorganic Ventures	0.10 of each
	1000 mg/L Cu	High Purity Standards	0.06
	1000 mg/L Ba, Mn, Ni	High Purity Standards	0.04 of each
	1000 mg/L U, Be, Cd, Co, Ag, Th, Tl, Pb, Sb	High Purity Standards	0.02 of each
ICP-MS CAL 1 -(5% HNO_2/5%HCL)	1000 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Th, Tl, U, V, Zn	High Purity Standards	0.2 of each
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.02
	Conc. HCL	Baker Instra Analyzed	2
ICP-MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na, Fe, Mg, Ca	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.95
	1000 mg/L B, Sn, Sr, W	High Purity Standards or Inorganic Ventures	0.50 of each
ICP-MS-MIX-Y (1.0% HNO3/ 0.5% HCl)	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.030
	1000 mg/L As, Ba, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, V, Zn	High Purity Standards or Inorganic Ventures	0.30 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	0.1 of each
	1000mg/L Pb		0.30

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	CCV	Cal. Std.	ICV	PQL	P/A Tune Soln.
Aluminum	500.0	1000.0	400.0	20.0	200
Antimony	25.0	50.0	20.0	0.2	200
Arsenic	25.0	50.0	20.0	1.0	
Barium	25.0	50.0	20.0	0.4	
Beryllium	25.0	50.0	20.0	0.2	
Boron	25.0	50.0	20.0	4.0	
Cadmium	25.0	50.0	20.0	0.2	
Calcium	5000.0	10000.0	4000.0	20.0	
Chromium	25.0	50.0	20.0	1.0	200
Cobalt	25.0	50.0	20.0	0.2	200
Copper	25.0	50.0	20.0	0.6	
Iron	5000.0	10000.0	4000.0	20.0	
Lead	25.0	50.0	20.0	0.2	200
Magnesium	5000.0	10000.0	4000.0	20.0	
Manganese	25.0	50.0	20.0	0.4	200
Molybdenum	25.0	50.0	40.0	1.0	200
Nickel	25.0	50.0	20.0	0.4	
Potassium	5000.0	10000.0	4000.0	200.0	200
Selenium	25.0	50.0	20.0	1.0	
Silicon	500.0	1000.0	400.0	100.0	
Silver	25.0	50.0	20.0	0.2	
Sodium	5000.0	10000.0	4000.0	200.0	200
Strontium	25.0	50.0	20.0	1.0	200
Thallium	25.0	50.0	20.0	0.2	
Thorium	**25.0**	**50.0**	**20.0**	**0.2**	
Tin	25.0	50.0	20.0	1.0	
Tungsten	25.0	50.0	20.0	1.0	
Uranium	25.0	50.0	20.0	0.2	200
Vanadium	25.0	50.0	20.0	1.0	200
Zinc	25.0	50.0	20.0	2.0	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Aluminum	100000	100000			
Antimony		20			
Arsenic		20			
Barium		20		10	
Beryllium		20			
Boron		20			
Cadmium		20			
Calcium	100000	100000			
Chromium		20			
Cobalt		20		10	
Copper		20			
Iron	100000	100000			
Lead		20		10	
Magnesium	100000	100000		100	
Manganese		20			
Molybdenum	2000	2000			
Nickel		20			
Potassium	100000	100000			
Selenium		20			
Silver		20			
Sodium	100000	100000			
Strontium		20			
Thallium		20		10	10.0
Thorium		**20**			
Tin		20			
Tungsten		20			
Uranium		20			
Vanadium		20			
Zinc		20			
Bismuth			1000.0	10	
Germanium			1000.0	10	
Indium				10	
Lithium (^{6}Li)			1000.0	10	
Scandium			1000.0	10	
Terbium			1000.0	10	
Yttrium			1000.0	10	10.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Cerium					10.0
Lithium					10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L					
	MS-MIX-Z	ICP-MS PQL Intermediate	ICP-MS-MIX-Y	ICP-MS Method Tune Intermediate	ICP-MS CAL 1	ICP-MS ICSAB Intermediate
Aluminum	95.0	2.0	3.0		0.2	
Antimony		0.02	3.0		0.2	
Arsenic		0.10	3.0		0.2	
Barium		0.04	3.0		0.2	
Beryllium		0.02		1.0	0.2	
Boron	5.0	4.0				0.2
Cadmium		0.02			0.2	
Calcium	500	2.0				
Chromium		0.10	3.0		0.2	
Cobalt		0.02		1.0	0.2	
Copper		0.06	3.0		0.2	
Iron	500	2.0				
Lead		0.02	3.0	3.0	0.2	
Magnesium	500	2.0		10.0		
Manganese		0.04	3.0		0.2	
Molybdenum		0.10	3.0		0.2	
Nickel		0.04	3.0		0.2	
Potassium	500	20.0				
Selenium		0.10	3.0		0.2	
Silicon	100	10.0				5.0
Silver		0.02			0.2	
Sodium	500	20.0				
Strontium	5.0	0.10				0.2
Thallium		0.02		1.0	0.2	
Tin	5.0	0.10				0.2
Thorium		0.02			0.2	
Tungsten	5.0	0.10				0.2
Uranium		0.02			0.2	
Vanadium		0.10	3.0		0.2	
Zinc		0.20	3.0		0.2	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	Instrument Calibration Standard 3 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum		10.0			
Antimony		10.0			
Arsenic		10.0			
Barium		10.0			
Beryllium		10.0			
Boron					
Cadmium		10.0			
Calcium	1000		200.0		
Chromium		10.0			
Cobalt		10.0			
Copper		10.0			
Iron	1000		200.0		
Lead		10.0			
Magnesium	1000		200.0		
Manganese		10.0			
Molybdenum				10.0	10.0
Nickel		10.0			
Potassium	1000		200.0		
Selenium		10.0			
Silver		10.0			
Sodium	1000		200.0		
Strontium					10.0
Thallium		10.0			
Thorium				10.0	
Tin					10.0
Tungsten					
Uranium				10.0	
Vanadium		10.0			
Zinc		10.0			

TABLE 9 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**Internal Standard Mix 1 (Spex)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Bismuth		1000	
Cerium			10000
Germanium		1000	
Indium		1000	
Lithium			10000
Lithium (^{6}Li)		1000	
Scandium		1000	
Terbium		1000	
Thallium			10000
Yttrium		1000	10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 10

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
…	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 11

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program	Acquisition Mode	Spectrum
	Points per Mass	3
	Number of Replicates	3
	Detector Mode	Auto for all elements
	Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
		0.30 sec for Be, As, Cd, Ge
		0.010 sec for Na, Al, K, ^{28}Si
		0.030 for Ca, Fe, Sr
		1.00 sec for Se
		0.050 sec for Mg, Co
	Spray Chamber Temperature	2° C
	Total Acquisition Time	105 sec for 3 replicates
Peristaltic Pump Program	Analysis Speed	0.15 rps
Before Acquisition	Uptake Speed	0.15 rps
	Uptake Time	5 sec
	Stabilization Time	15 sec
After Acquisition (Probe Rinse)	Rinse Speed	0.15 rps
	Rinse Time (sample)	5 sec
	Rinse Time (standard)	5 sec
After Acquisition (Rinse)	Rinse Vial	1
	Uptake Speed	0
	Uptake Time	0 sec
	Stabilization Time	0 sec
Calibration Curve fit	All quantitation masses	Y=ax+(blank)
	All internal standard masses	(Excluded)
	All interference correction masses	(Excluded)
Reporting Parameters	QC Reports	On-Printer
	All Other Reports	Off

TABLE 12

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 13

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 14

REPORTED ISOTOPES AND INTERNAL STANDARDS

ELEMENT	MASS	INTERNAL STANDARD (mass)
Aluminum	27	Scandium (45)
Antimony	123	Terbium (159)
Arsenic	75	Yttrium (89)
Barium	135	Terbium (159)
Beryllium	9	Lithium (6)
Boron	11	Lithium (6)
Cadmium	114	Yttrium (89)
Calcium	44	Scandium (45)
Chromium	52	Yttrium (89)
Cobalt	59	Yttrium (89)
Copper	65	Yttrium (89)
Iron	57	Yttrium (89)
Lead	208	Bismuth (209)
Magnesium	25	Scandium (45)
Manganese	55	Yttrium (89)
Molybdenum	98	Yttrium (89)
Nickel	60	Yttrium (89)
Potassium	39	Scandium (45)
Selenium	82	Yttrium (89)
Silicon	29	Scandium (45)
Silver	107	Yttrium (89)
Sodium	23	Scandium (45)
Strontium	88	Yttrium (89)
Thallium	203	Bismuth (209)
Thorium	232	Bismuth (209)
Tin	118	Terbium (159)
Tungsten	182	Terbium (159)
Uranium	238	Bismuth (209)
Vanadium	51	Yttrium (89)
Zinc	66	Yttrium (89)

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in “Standard Methods for the Examination if Water and Wastewater,” Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-611
Revision History
Cover Page
Page 1

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: John C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Neufeld Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table	DN	1.29.01	1/29/01
03 7471A	Changed LeeMAN PS200 Automated Mercury Analyzer to Cetac MG100 Mercury analyzer. Revised Sect. 10 to show correct reference material. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605
04 7471A	Sect. 5.9 and 5.10 - changed preparation of intermediate mercury standards from daily to monthly. Sect. 7.8 - removed calibration blanks (LCB/CCB). They are prepared in sect. 7.6. Added weighing of boiling chips for the prep blanks. Sect. 8.3 - Removed intermediate standards	LAD	03/08	03/08
05	Revised Sections 8 and 10, and Tables 1 and 2 to update compliance from method 7471A to method 7471B.	LAD	02/09	02/09
06	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DoD QSM version 4.1 compliance.	DN	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added Table 2 with DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10
08	Sect. 4.6 - Changed thermometer type. Added LCSO - A LCS prepped using aqueous mercury LCSspike. Updated type of marker used to label digestion bottles. Updated corrective action for failing PQL standard.	LAD	12/10	12/10
09	Sect. 7 - Changed calibration digestion from digestion of all points to digestion of high point and dilutions of rest. Changed prep from 3 to 0.2g aliquots to 1 w 0.6g aliquot. Added additional prep info. Added Serial dilution and PDS to sect. 8, Added MOL, LOD, LOQ info. to sect. 9. Updated and added references to Sect. 10.	LAD	04/12	04/12
10	Sect. 7 - Corrected Calibration preparation, changed digestion temperature to 95 +/- 3°C. Sect. 10 - Added and updated references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 4 - Added snap top containers and digestion tubes. Sect 5 - Updated Aqua Regia prep. Sect. 7 - Added heat block and digestion tube instructions, minor edits. Removed Table 2, updated fig. 1, added fig. 4 changed ~~KASINC~~ KAS throughout. KASINC to LAD 10/2017	LAD	10/17	10/17
12	Sect. 7 - Updated to reflect current Calibration and Independent Calibration verification Standard preparation. Corrected typos	LAD	01/19	01/19

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-611-11, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-11, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

.

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, sludges and tissue under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process. LCSS utilizes the standard reference material. LCSO is spiked with aqueous mercury LCS spike.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of

hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and

recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps or digestion tubes, for use as digestion vessels.

4.2 Heat source capable of maintaining a constant temperature of 95°C.

4.3 Analytical balance capable of weighing to 0.01 g.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Thermometer, NIST-traceable, covering the range from -10° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not

acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

4.9 4oz graduated snap-cap container, 120 mL capacity.

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water – mercury-free water.

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 1:1 Aqua regia: Prepare an appropriate amount immediately before use. Start with 4 parts laboratory reagent grade water, carefully add one part of concentrated HNO_3 and then three parts of concentrated HCl in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood. Record preparation in "Metals Preparation Laboratory - Reagent Preparation Logbook". Refer to Figure 4 for a cop of a page from this logbook

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared monthly, and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared monthly, and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically-established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to $4^{o}C \pm 2^{o}$	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Glass mercury digestion bottles are reused, and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

7.2 Uncontaminated bottles are then triple-rinsed with laboratory reagent grade water, and are ready for reuse.

7.3 Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water, and are then ready for reuse.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.4 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, Bottle IDs, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the benchsheet. All necessary details of sample preparation (standards preparation information, digestion times, digestion temps, initial weights and final volumes, pertinent observations, etc.) must be recorded on this benchsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.5 Using an industrial marker with super permanent ink, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control sample and matrix spike sample to be digested.

7.6 Calibration Preparation - Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to two standard digestion bottles (250 mL media bottles). Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to each bottle. The blank calibration standard is prepared in the same manner as the high calibration standard except for the addition of mercury standard. Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to the appropriately labeled media bottle. Using a dose cup, add 15 mL potassium

permanganate to each calibration standard. Using a repipettor, add 8 mL potassium persulfate solution to each calibration standard and swirl to mix. Fill each bottle to 100 mL with laboratory grade reagent water. The mercury concentration of this calibration standard is 10.0 ug/L. Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5.0 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount Calibration Blank Solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.7 Initial Calibration Verification Standard (ICV) Preparation - Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to a standard digestion bottles. Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to the bottle. Using a calibrated adjustable pipette, prepare the ICV standard by adding 600 uL of Intermediate Mercury Standard A to the appropriately labeled media bottle. Using a dose cup, add 15 mL potassium permanganate to the standard. Using a repipettor, add 8 mL potassium persulfate solution to the standard and swirl to mix. Fill the bottle to 100 mL with laboratory grade reagent water. The mercury concentration of the ICV will be 6.0 ug/L.

7.8 Prepare an appropriate number of preparation blanks (PBS) by adding 1.0 g of Teflon boiling chips to labeled digestion bottles.

7.9 Prepare an appropriate number of laboratory control samples (LCSS or LCSO) by weighing appropriate masses of solid reference material or by adding 500 uL of Intermediate Mercury Standard A respectively into labeled digestion bottles. The mercury concentration of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material. The mercury concentration of the LCSO will be 5.0 ug/L.

7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.11 Preparation blanks, laboratory control spike and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP. Calibration standards are not digested.

SAMPLE PREPARATION AND DIGESTION

7.12 Do not decant any water on the sediment sample. **Note:** Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

Mix sample with a wooden spatula to ensure homogeneity of the sample. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on sub-sampling to ensure reproducibility.

Weigh an approximate 0.6 g portion of untreated, homogenized sample from the sample container and place in the bottom of a labeled digestion bottle.

7.13 Add 10 Ml of 1:1 Aqua Regia to each sample, standard, and QC sample. Place bottles in a heat source located in a fume hood and heat for 2 minutes at 95 ±3°C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 For glass mercury bottle preparation, add 50 Ml of laboratory reagent grade water and 15 Ml of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. For mercury digestion tube preparation, add 20 Ml of laboratory reagent grade water and 15 Ml of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. For Samples that contain large amounts of oxidizable organic matter may require additional 15 Ml aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 Ml aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95°C. Record initial and final time and temperatures on the mercury preparation benchsheet.

7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 Ml aliquots of potassium permanganate solution as necessary to obtain a persistent purple

color and heat for an additional 30 minutes at 95 ±3 ^{O}c. Record any information regarding additional permanganate aliquots on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for the samples affected.

7.16 For glass mercury bottle preparation, add 6 Ml of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 Ml of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

For mercury digestion tube preparation, quantitatively transfer sample to 4oz snap cap and then add 6 Ml of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Bring sample to 100 Ml final volume with reagent grade water. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, “Operation and Maintenance of the CETAC M6100 Mercury Analyzer”. The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471, and should be used in conjunction with the instructions given in Katahdin SOP CA-629.

7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in “templates”. The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named “SW846-7470-7471”.

7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument’s autosampler. The “standards” autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 Ml capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.

7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into

17 x 100 mm autosampler tubes (15 Ml capacity), which are placed in the one of the "samples" autosampler racks. The "samples" autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the "samples" autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses 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. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B)V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known

standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 2. A linear regression program may be used to obtain the intercept concentration.

7.22 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.23 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported – the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{I \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)

TS = Total Solids (%)

7.24 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.4 Instrument calibration – The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed. For DoD QSM acceptance criteria, samples that are below the reporting limit may be reported if the CCV reads greater than 120%.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. If the PQL fails, results may not be reported from the run until the problem is corrected and a passing PQL has been analyzed.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSS or LCSO), consisting of solid reference material or 500 Ul of Intermediate Standard A carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples that are supplied by the vendor of the solid reference material. The results of the LCSO must fall with in 80% - 120% of its true value which is 5.0 ug/L. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS or LCSO must be within 80% - 120%.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 Serial Dilution – A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.14 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantiaion (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 7471B.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

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TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning of run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration □ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS or LCSO)	One per digestion batch of 20 or fewer samples.	LCSS: Recovery within vendor-supplied acceptance limits. LCSO: Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate (D)	One per digestion batch of 20 or fewer samples.	1)Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes or duplicate samples.	Flag results
	Post-Digestion Matrix Spike Sample (PDS)	When matrix spike or MSD fail	Recovery ±20% of true value	Analyze serial dilution of sample
	Serial Dilution Test (L)	One per digestion batch or when PDS fails	1:5 dilution of sample must agree within 10% with undiluted result	If MS, MSD, PDS, and serial dilution fail, quantitate sample by method of standard additions
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1)Repeat IDL study. 2)Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the 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 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 analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		not listed, use in-house LCS limits if project limits are not specified.	failed analytes, if sufficient sample material is available.	specific analyte(s) in all samples in the associated preparatory batch.	reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 3

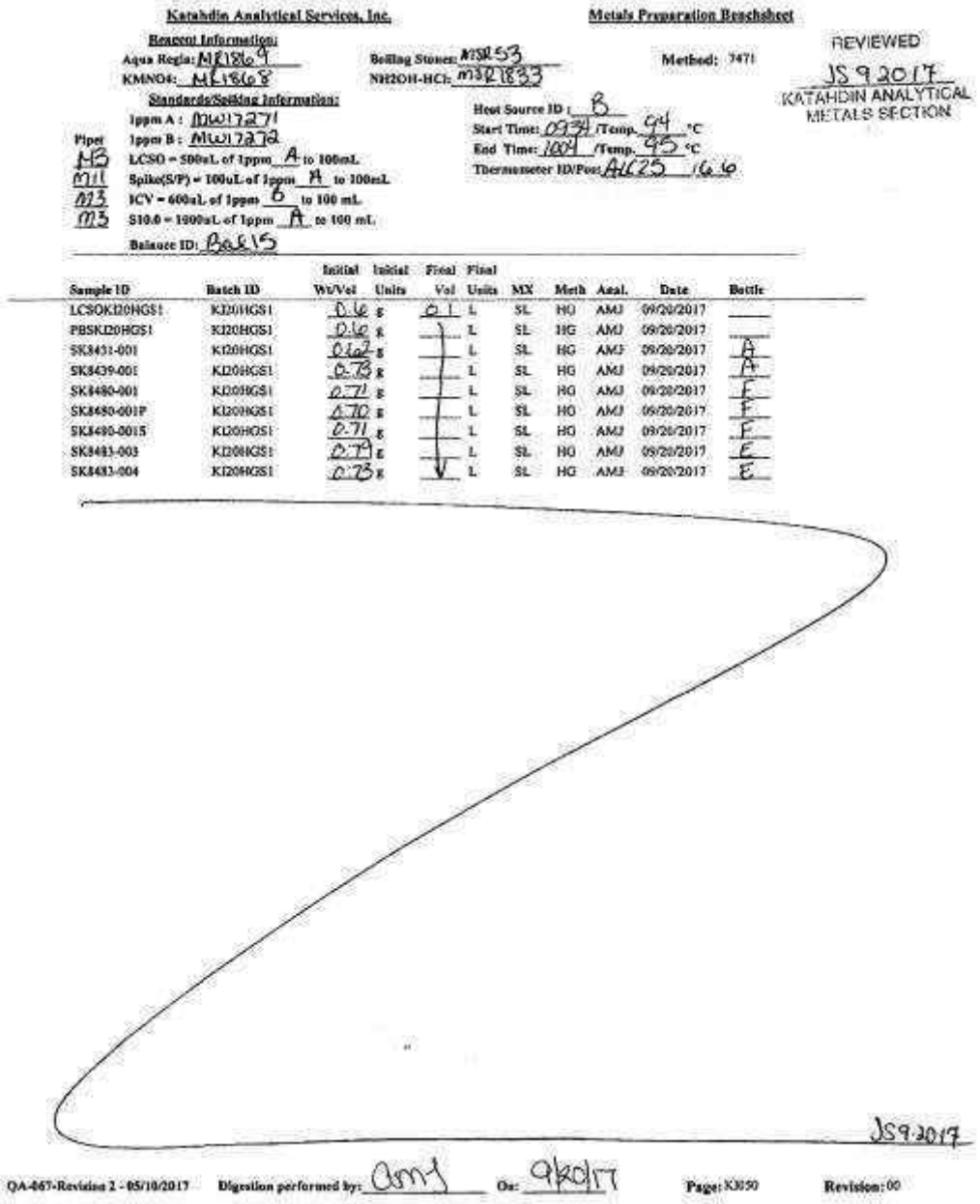
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-12	USEPA Method 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
Qc _ Calibration	Calibration standards are not digested.	Sect. 7.3- Requires Calibration standards are digested
QC – Calibration Verification	1)Known reference sample (ICV) analyzed daily. 2)Calibration verified after every 10 samples with CCV.	1)Known reference sample analyzed quarterly. 2)Calibration verified after every 20 samples.
QC - Calibration Blanks and Method Blanks	Acceptance Criterion: < PQL	Acceptance criteria: Low enough not to interfere with data quality objectives, or <10% of PQL, or <10% of regulatory limit, or <10% of lowest associated sample

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
Aqua Regia: ME1869
KMNO4: ME1868
Boiling Stones: M3R53
NH2OH-HCl: M3R1833
Method: 7471

REVIEWED
JS 9 2017
KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:
1ppm A: MW17271
1ppm B: MW17272
Pipet
M3 LCSO = 500uL of 1ppm A to 100mL
M11 Spike(S/P) = 100uL of 1ppm A to 100mL
M3 ICV = 600uL of 1ppm B to 100 mL
M3 S10.0 = 1000uL of 1ppm A to 100 mL
Balance ID: Bal15

Heat Source ID: B
Start Time: 0934 /Temp. 94 °C
End Time: 1004 /Temp. 95 °C
Thermometer ID/Post: AL25 1666

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOKI20HGS1	KI20HGS1	0.6	g	0.1	L	SL	HG	AMJ	09/20/2017	___
PBSKI20HGS1	KI20HGS1	0.6	g		L	SL	HG	AMJ	09/20/2017	___
SK8431-001	KI20HGS1	0.62	g		L	SL	HG	AMJ	09/20/2017	A
SK8439-001	KI20HGS1	0.73	g		L	SL	HG	AMJ	09/20/2017	A
SK8480-001	KI20HGS1	0.71	g		L	SL	HG	AMJ	09/20/2017	F
SK8480-001F	KI20HGS1	0.70	g		L	SL	HG	AMJ	09/20/2017	F
SK8490-001S	KI20HGS1	0.71	g		L	SL	HG	AMJ	09/20/2017	F
SK8483-003	KI20HGS1	0.79	g		L	SL	HG	AMJ	09/20/2017	E
SK8483-004	KI20HGS1	0.73	g	V	L	SL	HG	AMJ	09/20/2017	E

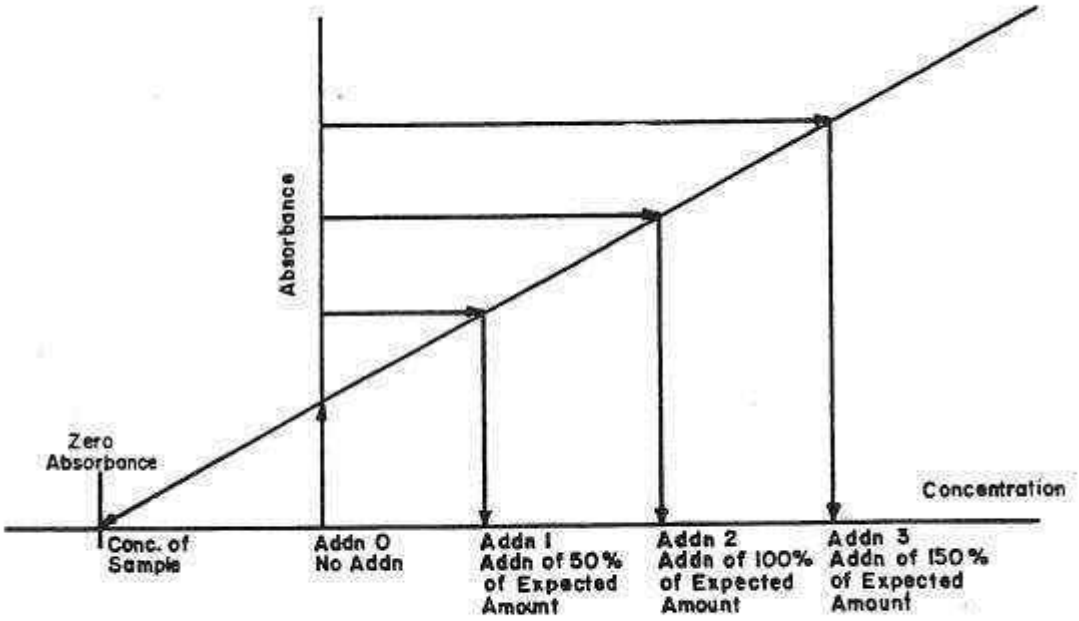
JS 9.2017

QA-667-Revision 2 - 05/10/2017 Digestion performed by: AMJ On: 9/20/17 Page: K3050 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	6630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	860	211	174 - 247
beryllium	80.8	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2920 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	71.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	84.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	860	217	177 - 257
beryllium	80.8	53.0	42.7 - 64.5
boron	129	89.5	58.0 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	161	132 - 192
magnesium	3780*	2160	1660 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.28	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2100 - 3520
selenium	146	135	104 - 166
silver	127	107	49.6 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 4

EXAMPLE REAGENT PREP LOGBOOK PAGE

REAGENT PREP LOG - METALS PREP LAB

Reagent ID	Reagent Name	Prep Date	Exp. Date	Prep Initials	Component	Lot Number	Amount Added	Prep Notes
MR1863	$SnCl_2$ - HCL	9-8-17	12-8-17	AMJ	$SnCl_2$	MSR66/MSR76		FV= 4000mL w/Reag H_2O
↓	↓	↓	↓	↓	Conc HCL	MSR68	280mL	↓
MR1864	1:1 Aqua Regia	9-12-17	9-13-17	AMJ	Conc HCL	MSR68	150mL	FV= 400mL w/Reag H_2O
↓	↓	↓	↓	↓	Conc HNO_3	MSR75	50mL	↓
MR1865	NH_2OH - HCL (12% W/V)	9-14-17	9-14-17	AMJ	NH_2-OH	MSR57/MSR58	480.01	FV= 4000mL w/ Reag H_2O
↓	↓	↓	↓	↓	NaCL	MSR36	480.01	↓
MR1866	1:1 aqua Regia	9-18-17	9-19-17	AMJ	Conc HCL	MSR68	150mL	FV= 400mL w/Reag H_2O
↓	↓	↓	↓	↓	Conc HNO_3	MSR77	50mL	↓
MR1867	5% HNO_3	9-18-17	9-18-18	AMJ	Conc HNO_3	MSR77	500mL	FV= 10L w/Reag H_2O
MR1868	5% (M/V) $KMNO_4$	9-18-17	9-12-18	AMJ	$KMNO_4$	35309	200.00g	FV= 4L w/Reag H_2O AMJ 9/18/17
MR1869	1:1 aqua Regia	9-20-17	9-21-17	AMJ	Conc HCL	MSR73	150mL	FV= 400mL w/Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR77	50mL	↓

ME-011 - Revision 1 - 07/25/2013

QAAA184

0000006

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis P. Hogan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02-16-05	02-16-05
02	Updated Fig. 1 - new prep logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoDQSM version 4.1 QC Requirements.	LAD	04/10	04/10
06	Sect. 4.6 - changed thermometer type. Sect. 7.3 - Changed type of marker used. Table 1 - Added PQL standard corrective action. Table 2 - added comments for calibration blank. Sect. 9 - Added MDL, LOD and LOQ information	LAD	05/11	05/11
07	Sect. 7 - Calibration prep from digesting all to digesting high STD. and diluting down. Added serial dilution and PDS to sect. 8. Added more MDL, LOD & LOQ information to Sect. 9. Updated and added references to Sect. 10	LAD	04/12	04/12
08	DoDQSM 5.0 References added. Sect. 7.4 and Table 3 - Updated Calibration Standard prep - removing digesting all Standards. Added to digest high point	LAD	06/14	06/14
09	Updated Figure 1. Change title of section 5.0, Update method references for NELAC and DoD. Minor additions to sections 4.1, 4.2, 4.6, 7.1, 7.10, 7.12.	LAD	09/17	09/17
10	Removed DoDQSM 4.2 QC Requirement Table. Added DoDQSM 5.0/5.1 QC Requirement Table. Updated references. Updated Logbook Example	LAD	11/18	11/18
11	Sect. 7 - Updated to reflect current calibration and Independent Calibration Verification Standards	LAD	01/19	01/19

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-11,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-11,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of

hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL, 50 mL or 70 mL digestion tubes and appropriate watch glasses, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in preparation of calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95° C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50° to 750° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate

Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL, 50 mL or 70 mL digestion tubes. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using an industrial marker with super permanent ink, label clean sample containers with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control spike and matrix spike and standard to be digested.

7.4 Calibration Preparation - Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to two standard digestion bottles (250 mL media bottles). Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to each bottle. The blank calibration standard is prepared in the same manner as the high calibration standard except for the addition of mercury standard. Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to the appropriately labeled media bottle. Using a dose cup, add 15 mL potassium permanganate to each calibration standard. Using a repipettor, add 8 mL potassium persulfate solution to each calibration standard and swirl to mix. Fill each bottle to 160 mL with laboratory grade reagent water. The mercury concentration of the high calibration standard is 10.0 ug/L.

Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5 ug/L standards are analyzed after calibration as the PQL standard and

the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount calibration blank solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.5 Independent Calibration Verification Standard - Using a bottle-top dispenser add approximately 10 mL of laboratory grade reagent water to a digestion bottle. Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to the bottle. Using a calibrated adjustable pipette, add 600 uL of Intermediate Mercury Standard B to the bottle. Using a dose cup, add 15 mL potassium permanganate to each calibration standard. Using a repipettor, add 8 mL potassium persulfate solution and swirl to mix. Fill to 160 mL with laboratory grade reagent water. The mercury concentration of the ICV standard is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 Preparation blanks, laboratory control spikes and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13 but the standards are not heated.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup or pour directly into graduated sample tube, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that

contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

Some samples may require dilution to 25 mL with potassium permanganate for digestion to be performed in the digestion vessel. Prepare method blank and LCS with equal amounts of potassium permanganate to check for potential mercury contamination.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials, for 50 mL or 70 mL tubes, add ribbed watch glasses, and place them in a preheated water bath or heat source. Monitor the temperature of the bath with a thermometer throughout the digestion. The temperature of the water bath will fall below 90-95° C upon addition of the digestion vials. After the temperature of the bath has risen back to 90-95° C, continue heating the samples at 90-95° C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath or heat source and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 90-95° C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. After color change, samples are filled to 40 mL with laboratory grade reagent water and mixed well. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, “Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer”.

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METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B)V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is

shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{\text{MC X DF X IV}}{\text{FV}}$$

where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a “U” qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard

deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of

Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is

less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.14 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

The 2009 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient $\geq$ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm$ 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within $\pm$ 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm$25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm$ 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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Table 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the 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 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 analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	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. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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TABLE 3

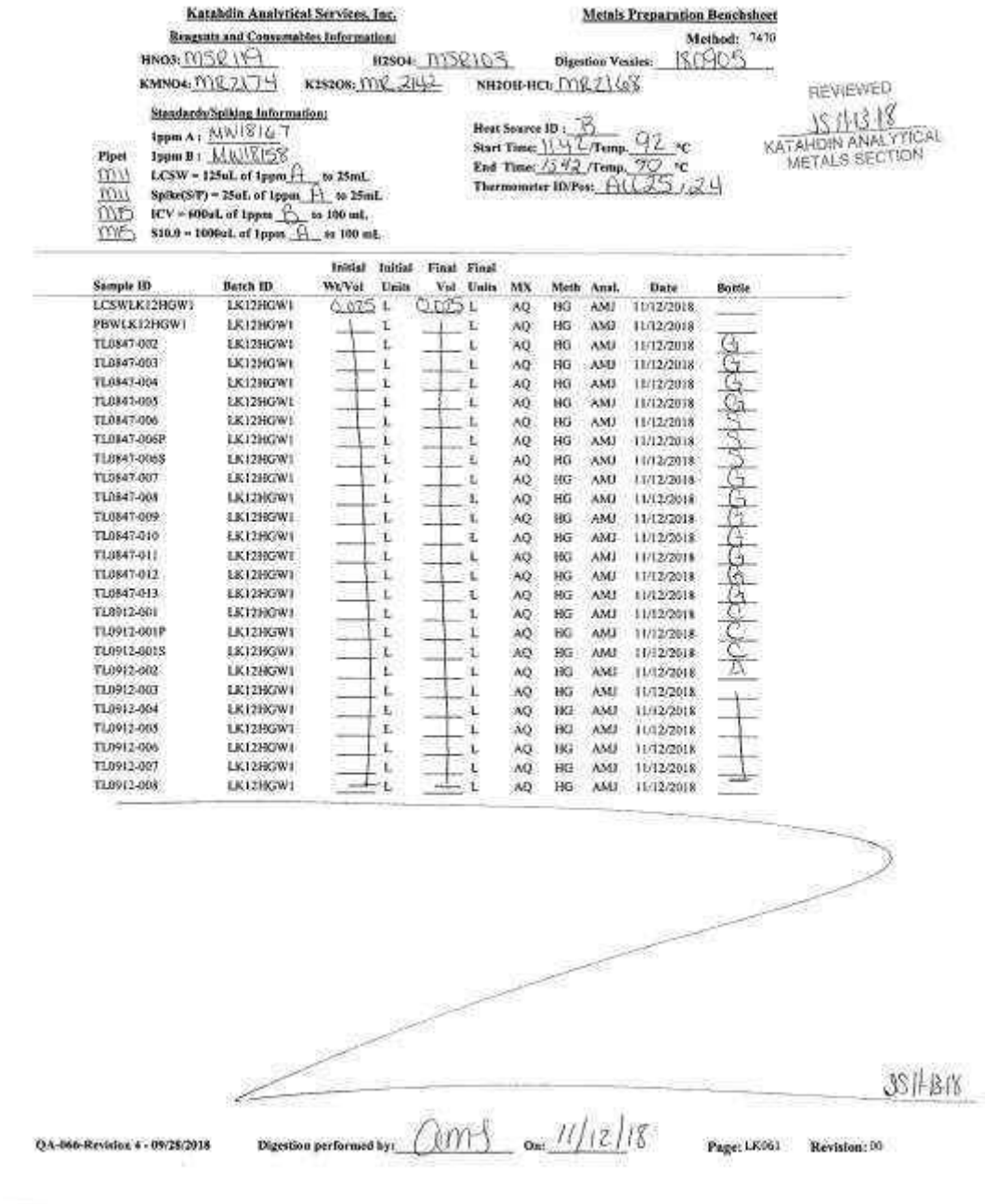
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-11	USEPA METHOD 7470
Reagents	1) Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	1) Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	2) Sampling and gas stream switching performed automatically by mercury analyzer. 3) Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	2) Sampling and gas stream switching performed manually by analyst. 3) Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	4) Known reference sample (ICV) analyzed daily. 5) Calibration verified after every 10 samples with CCV.	4) Known reference sample analyzed quarterly. 5) Calibration verified after every 20 samples.
QC - Calibration Blanks	6) Acceptance criteria employed for 245.1: ± PQL	6) Acceptance criteria stated in 245.1: ± MDL

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. **Metals Preparation Benchsheet**

Reagents and Consumables Information: **Method:** 7470

HNO3: MSR119 H2SO4: MSR103 Digestion Vessies: 180905

KMNO4: MR2174 K2S2O8: MR2142 NH2OH-HCl: MR2168

REVIEWED
15/11/18
KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:

1ppm A : MW18167
1ppm B : MW18158

Pipet
MU LCSW = 125uL of 1ppm A to 25mL
MU Spike(S/P) = 25uL of 1ppm A to 25mL
MS ICV = 600uL of 1ppm B to 100 mL
MS S10.0 = 1000uL of 1ppm A to 100 mL

Heat Source ID : B
Start Time: 1142 /Temp. 92 °C
End Time: 1542 /Temp. 90 °C
Thermometer ID/Pos: ACC25 / 24

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWLK12HGW1	LK12HGW1	0.025	L	0.025	L	AQ	HG	AMJ	11/12/2018	
PBWLK12HGW1	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-006P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-006S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-009	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-010	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-011	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-012	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-013	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0912-001	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-001P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-001S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	A
TL0912-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	

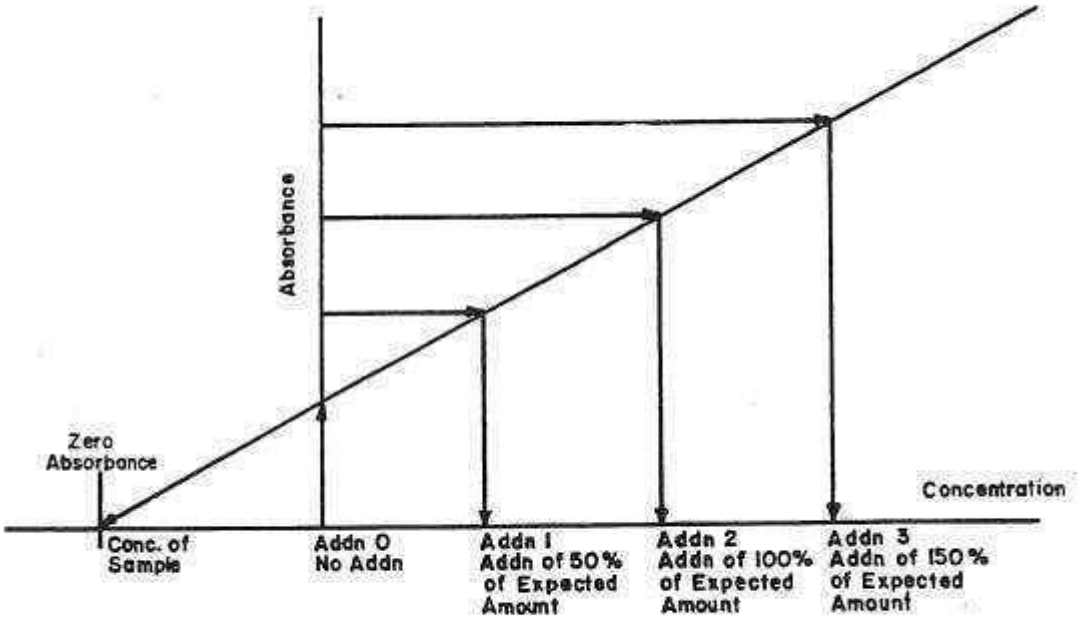
JS 11-13-18

QA-066-Revision 4 - 09/28/2018 Digestion performed by: AMJ On: 11/12/18 Page: LK061 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-625
Revision History
Cover Page
Page 1

TITLE: ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196

Prepared By: George Brewer Date: 08/2002

Approved By:

Group Supervisor: George Brewer Date: 08/29/02

Lab Operations Mgr: John C. Burton Date: 8/29/02

QA Officer: Deborah J. Nadeau Date: 8.29.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	remove AQUEOUS from title. Removed Sect. 4.8 changed block digester to centifuge (sect. 7.11, 7.15, 7.17) Editted and added wording to sect. 8. Table 1, changed flag results to narrate results. Minor changes through out	LAD	031605	031605
02	Added definitions; section 5-edited standard 'A' and "B"; sections 7 and 8-edited to reflect current procedures; sections 4.9 and 7.6-corrected membrane filter size, added Figures 3 and 4.	LAD	04/08	04/08
03	Section 4.12- Changed Spec 401 & 301 to UV/visible spectrophotometer. Sections 4.9, & 7.6 - Changed 0.45 um membrane filters to 0.7 um glass fiber filters Also added 0.7 um filter to Table 2. Method Modifications Added KAS SOP CA-108 reference - subsampling	LAD	04/09	04/09
04	Added definitions to section 1.1. Revised table 1 for compliance with DoD QSM 4.1. Added EHSM, QA-506, Subsampling, DoD, NELAC and CA-101 references.	EN	08/09	08/09
05	Added ICV information throughout. Edited Sect. 7.22.2 for current practice. Added to Table 2 - calibration is not digested. Added DoD QSM QC criteria as Table 2. Renamed Method Modifications as Table 3. Sect. 9 - added MDL, LOD, LOQ info Sect. 10 - Added, edited references updated Figures 1 -> 4.	LAD	04/12	04/12

TITLE: ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect 1 – Change CCB from deionized water to neutralized digestion solution.	LAD	05/13	05/13
07	Sect. 4 and 7 - Added automated pH adjustment procedure. Updated References Updated Figures 1 > 4	LAD	03/19	03/19

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-625-07**, titled **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-625-07**, titled **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196.**

Recipient: _____ Date: _____

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze solid matrix samples for hexavalent chromium analysis in accordance with USEPA SW-846 Methods 3060 and 7196, current revisions.

Method 3060 is an alkaline digestion procedure for extracting hexavalent chromium [Cr(VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. This alkaline digestion is designed to solubilize all forms of Cr(VI) without inducing reduction of native Cr(VI) to Cr(III), and without oxidizing native Cr(III) to Cr(VI). The alkaline conditions of the extraction minimize oxidation and reduction, and Mg^{2+} is added in a phosphate buffer to suppress oxidation. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g.,$K_2Cr_2O_7$ and $PbCrO_4$).

The quantification of Cr(VI) in Method 3060 digests is performed using Method 7196. In this method, Cr(VI) is reacted with diphenylcarbazide to form a red-violet color, the intensity of which is measured by UV-VIS spectrometry.

1.1 Definitions

CCV- Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is analyzed at the beginning of the run, after every ten measurements and at the end of the run..

CCB- Continuing Calibration Blank. The CCB is alkaline digestion solution (section 5.4). One CCB is analyzed after each CCV.

LCS/ICV- Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

LOD – Limit of Detection. The smallest amount or concentration of an analyte that must be present in a sample to be detected at a 99% confidence level. At the LOD, the false negative rate is 1%.

MDL – Method Detection Limit. The minimum amount of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

Method Blank – A deionized water sample that is carried through the entire analytical procedure in the same manner as the sample.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in hexavalent chromium preparation and analysis by USEPA Methods 3060 and 7196, current revisions. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in hexavalent chromium preparation and analysis by USEPA Methods 3060 and 7196 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. Cr(VI) is toxic, and solutions containing it must be handled with care. Lead chromate (PbCrO4) is also very toxic, and care must be taken to avoid spilling or inhaling dust from this reagent while weighing it. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health & Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in hexavalent chromium analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in this determination may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Sample Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

This method uses an alkaline digestion to solubilize both water-insoluble and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during digestion and analysis. Failure to meet the pH specifications will necessitate redigestion of the samples.

The sample is digested using 0.28M Na_2CO_3 /0.5M NaOH solution and heating at 90-95°C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III). Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, are determined colorimetrically by reaction with diphenylcarbazide in acid solution. This reaction is highly selective for Cr(VI). Addition of an excess of diphenylcarbazide yields a red-violet product of unknown composition, and its absorbance is measured photometrically at 540 nm. The reaction is very sensitive, the absorbancy index per gram atom of chromium being about 40,000 at 540 nm.

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

3.0 INTERFERENCES

3.1 The measured Cr(VI) concentration of solid samples, as determined by Methods 3060 and 7196, should be interpreted with an understanding of the reducing/oxidizing tendency of each sample matrix. This is best accomplished by characterization of each sample for additional analytical parameters, such as pH, ferrous iron, sulfides, total organic carbon, and Oxidation Reduction Potential (ORP). Analysis of these additional parameters establishes the tendency of Cr(VI) to exist or not exist in the unspiked sample and assists in the interpretation of QC data for matrix spike recoveries that fall outside method acceptance limits. These additional determinations are done at the request of the client, and some are best performed in the field. For these reasons, interpretation of Cr(VI) results for solid samples must be left to the client.

3.2 For samples containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results obtained using these methods may be biased high due to method-induced oxidation. The addition of Mg^{2+} in a phosphate buffer has been shown to suppress this oxidation.

3.3 The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

3.4 Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

4.0 APPARATUS AND MATERIALS

4.1 250 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning").

4.2 Ribbed watch glasses, 100 mm diameter, pre-cleaned as above.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 10 mL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, griddle, or other heating source - adjustable and capable of maintaining a temperature of 95°C ± 5°C. Hot plates must be numbered for easy identification.

4.6 Disposable 50 mL capacity polyethylene centrifuge tubes with screw caps, graduated in 5 mL increments.

4.7 Graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.8 Vacuum filtration apparatus.

4.9 Glass fiber filter disks, 47 mm diameter, nominal pore size 0.7 um.

4.10 Calibrated pH meter.

4.11 Calibrated balance, capable of weighing to 0.01 g.

4.12 UV/Visible spectrometer for use at 540 nm, 1 cm path length.

4.13 1 cm rectangular cuvette.

4.14 Disposable 0.45 um syringe filters, with cellulose acetate, polycarbonate, or PTFE membranes.

4.15 Disposable polypropylene syringes, 10 mL capacity.

4.16 Mettler-Toledo Autotitrator and Rondo Tower

4.17 Alkalinity Cups

5.0 REAGENTS

5.1 Laboratory reagent grade water – purity >15.6 Megohm, free from interferents.

5.2 Concentrated nitric acid, HNO_3 – trace metals grade.

5.3 Nitric acid, 5.0 M: Add 100 mL of concentrated nitric acid to 100 mL of laboratory reagent grade water in a graduated container, and bring to a final volume of 300 mL with laboratory reagent grade water. Store at 20-25°C in the dark. Do not use

concentrated HNO_3 to make up 5.0 M solution if it has a yellow tinge; this is indicative of photoreduction of NO_3^- to NO_2, a reducing agent for Cr(VI).

5.4 Alkaline Digestion Solution:

5.4.1 Sodium hydroxide: NaOH, analytical reagent grade. Store at 20-25°C in a tightly sealed container.

5.4.2 Sodium carbonate: Na_2CO_3, anhydrous, analytical reagent grade. Store at 20-25°C in a tightly sealed container.

5.4.3 Digestion solution: Dissolve 40.0 ± 0.05 g NaOH and 60.0 ± 0.05 g Na_2CO_3 in laboratory reagent grade water in a two-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20-25°C and prepare fresh monthly. The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater, if not, discard.

5.5 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical reagent grade. A mass of 400 mg $MgCl_2$ is approximately equivalent to 100 mg Mg^{2+}. Store at 20-25°C in a tightly sealed container.

5.6 Phosphate Buffer:

5.6.1 Dibasic potassium phosphate, K_2HPO_4 – analytical reagent grade.

5.6.2 Monobasic potassium phosphate, KH_2PO_4 – analytical reagent grade.

5.6.3 0.5M K_2HPO_4 / 0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09 g K_2HPO_4 and 68.04 g KH_2PO_4 into 700 mL of laboratory reagent grade water. Transfer to a 1L volumetric flask and dilute to volume.

5.7 Lead Chromate ($PbCrO_4$), analytical reagent grade. The insoluble matrix spike is prepared by adding 10-20 mg of $PbCrO_4$ to a separate sample aliquot. Store under dry conditions at 20-25°C in a tightly sealed container.

5.8 Hexavalent Chromium Stock Standard “A”, 1000 ug Cr(VI)/mL – a purchased standard prepared from high purity metals or salts, and supplied by the vendor with certificates of analysis and traceability. Standards must be traceable to NIST standards. This stock standard is the source for calibration standards, CCVs and post digestion matrix spikes.

5.9 Hexavalent Chromium Working Standard “A” – Place approximately 90 mL of laboratory reagent grade water to a graduated snap-lid container. Pipet 2.5 mL of

Hexavalent Chromium Stock Standard "A" into the container, and bring to a final volume of 100 mL. The concentration of this working standard is 25 mg Cr(VI)/L.

5.10 Hexavalent Chromium Stock Standard "B", 1000 ug Cr(VI)/mL – a purchased standard prepared from high purity metals or salts, and supplied to the vendor with certificates of analysis and traceability. Standards must be traceable to NIST standards. This standard is the source for the LCS, ICV and the soluble matrix spike.

5.11 Hexavalent Chromium Working Standard "B" - Place approximately 90 mL of laboratory reagent grade water to a graduated snap-lid container. Pipet 2.5 mL of Hexavalent Chromium Stock Standard "B" into the container, and bring to a final volume of 100 mL. The concentration of this working standard is 25 mg Cr(VI)/L.

5.12 Concentrated sulfuric acid, H_2S0_4 – trace-metals grade.

5.13 Sulfuric acid, 10% (v/v): Add 10 mL of concentrated sulfuric acid to 50 mL of laboratory reagent grade water in a graduated container, and bring to a final volume of 100 mL with laboratory reagent grade water.

5.14 1,5 Diphenylcarbazide, analytical reagent grade.

5.15 Diphenylcarbazide solution: Dissolve 250 mg 1,5-diphenylcarbazide in 50 mL acetone. Store in a brown bottle. Discard when the solution becomes discolored.

5.16 Acetone (analytical reagent grade): Avoid or redistill material that comes in containers with metal or metal-lined caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples should be collected using devices and placed in containers that do not contain stainless steel (e.g., plastic or glass).

6.2 Samples should be stored field-moist at <6°C, without freezing, until analysis.

6.3 Hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection. In addition, Cr(VI) has also been shown to be stable in the alkaline digestate for up to 168 hours after extraction from soil.

6.4 To retard the chemical activity of hexavalent chromium, the alkaline extracts should be stored at $4^{o} \pm 2°$ C until analyzed. The maximum holding time prior to analysis extracts is 24 hr. The 24 hr holding time begins after extraction.

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

7.0 PROCEDURES

ALKALINE DIGESTION

7.1 Adjust the temperature setting the hot plate used in the alkaline digestion by preparing and monitoring a temperature blank [a 250 mL vessel filled with 50 mL alkaline digestion solution (Section 5.4)]. Maintain a digestion solution temperature of 90-95°C as measured with a NIST-traceable thermometer or equivalent.

7.2 Place 2.5 ± 0.10 g of the field-moist sample into a clean and labeled 250 mL digestion vessel. The sample should have been mixed thoroughly before the aliquot is removed. For the specific sample aliquot that is being spiked (Section 8.6), the spike material should be added directly to the sample aliquot at this point. Percent solids determination should be performed on a separate aliquot in order to calculate the final result on a dry-weight basis.

Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on subsampling to ensure reproducibility.

7.3 Add 50 mL ± 1 mL of alkaline digestion solution to each sample using a graduated specimen container, and also add approximately 400 mg of $MgCl_2$ and 0.5 mL of 1.0M phosphate buffer. Cover all samples with watch glasses.

7.4 Mix the samples by swirling the beakers (unheated) for at least five minutes.

7.5 Heat the samples to 90-95°C, then maintain the samples at 90-95°C for at least 60 minutes. Swirl the beakers continuously to mix.

7.6 Gradually cool, with continued agitation, each solution to room temperature. Transfer the contents quantitatively to the filtration apparatus that has been pre-rinsed with 5% Nitric acid; rinsing the digestion vessel with 3 successive portions of laboratory reagent grade water. Transfer the rinsates to the filtration apparatus. Filter through a 0.7 um glass fiber filter that has been pre-rinsed with 5% nitric acid and deionized water.

Rinse the inside of the filter flask and filter pad with laboratory reagent grade water and transfer the filtrate into a graduated polyethylene sample container with attached snap lid.

NOTE: Alkaline digestates of samples that contain significant amounts of organic matter may contain large amounts of extracted humic material. These digestates will be brown in color, and will be difficult to filter. Filtration of all samples may be facilitated by quantitatively transferring the digestate into a disposable centrifuge

tube and centrifuging at high speed for five minutes prior to filtration. Digestate should be transferred in small (5 – 10 mL) aliquots into the filtration apparatus, and the filter should be changed at the earliest signs of clogging. This is necessary to allow quantitative recovery of the filtrate.

7.7 If necessary, the filtered digestate may be stored at 4° C overnight before continuing with pH adjustment and colorimetric analysis.

7.7.1 For automated pH adjustment, refer to section 7.10

7.7.2 For manual pH adjustment refer to section 7.8-7.9

7.8 Place a stirring bar into the digestate container, place the vessel on a stirrer, and, with constant stirring, slowly add 5.0 M nitric acid solution to the beaker dropwise. Monitoring the pH with a pH meter, adjust the pH of the solution to 7.5 ± 0.5. Add the acid very slowly below pH 8.5, allowing the pH to stabilize before each new addition. If the pH of the digest should deviate from the desired range, discard the solution and redigest. If overshooting the desired pH range occurs repeatedly, prepare diluted nitric acid solution and repeat digestion procedure. Carbon dioxide will be evolved during pH adjustment, so this step should be performed with caution. If a flocculent precipitate should form, the sample should be filtered through a 0.45 um membrane filter prior to colorimetric analysis.

7.9 Remove the stirring device and rinse, collecting the rinsate in the beaker. Using the graduations on the sample container, adjust the sample volume to 100 mL with laboratory reagent grade water. Cap the digestate container and shake gently to mix, stopping frequently to vent carbon dioxide from the container.

7.10 Automated pH adjustment is done using the Mettler-Toledo autotitrator and LabX software.

7.10.1 Swap out the titrant currently in use with 5.0 M Nitric Acid.

7.10.2 Prime the pump. Set number of primes to 10. Be sure to empty the cup the probe is resting in prior to priming.

7.10.3 Empty the cup into waste satellite “A” Acid waste and replenish the cup with DI. All rinses will be acidic. Dispose of them in waste satellite “A” Acid waste.

7.10.4 Load samples (keeping them in their 100mL snap-caps with the lids open towards the center of the wheel) in positions 1-18 on the Rondo Tower. Positions 19 & 20 are reserved for pH 7 bath and rinse cup. Do not place samples in positions 19 & 20.

7.10.5 Open LabX Titration software.

7.10.6 Right click Method “00005 CrVI pH Adjustment,” click run.

7.10.7 Set desired number of samples and enter their IDs into the entry windows below. If over 18 samples need adjustment they can be programmed in, but the wheel will continue to run from position 18 back to position 1. Therefore samples 19+ will need to be loaded into positions 1-x as the first samples come off. Alternatively, the program can be run a second time with the rest of the samples.

7.10.8 The program method automatically adjusts pH of samples to a pH of 7.5 ± 0.5 in two stages. The first stage of adjustment slowly brings the pH to 9. The second stage of adjustment brings the sample to a pH of 7.5 ± 0.5 at a slower rate to ensure sample does not reach a pH<7. The program method records the final pH of each sample after an extended stirring period so the analyst can confirm the final pH is in the required range. Probe and components are automatically rinsed with three 5 mL aliquots of DI before moving to the next cup position.

7.10.9 Sample cups must be manually filled to a final volume of 100mL with DI once adjustment has been completed. If a flocculent precipitate should form, the sample should be filtered through a 0.45 um membrane filter prior to colorimetric analysis. Samples are now ready for color development.

7.10.10 Once all samples have been adjusted, the rinse program must be run to remove any traces of Nitric acid from the system and its components. Nitric acid will interfere with alkalinity analysis and pH analysis and could also potentially damage system components if it is not rinsed away thoroughly and in a timely manner. Do not let Nitric titrant sit in the system longer than necessary.

- Remove the line from the 5.0 M Nitric acid titrant.
- Place an empty alkalinity cup in probes resting position.
- Prime the pump to flush out the Nitric acid from the line.
- Place the line in a large container of fresh DI and secure it.
- Place 10 empty alkalinity cups in positions 1-10.
- Right click Method “00004 CrVI Rinse Cycle,” click Run.
- Set number of sample cups to 10.
- Click Run.
- Empty the rinse cups into waste satellite “A” Acid waste.
- Repeat steps 7.10.10.4-7.10.10.8 until a pH of close to 4 is achieved. One repetition should be enough. The pH of the water rinses should

be close to 4 when the Nitric acid is thoroughly rinsed from the system. Reinstall the desired alkalinity titrant.

COLORIMETRIC ANALYSIS – PREPARATION OF CALIBRATION CURVE AND QC SAMPLES

7.11 To match the matrix of the associated samples, the calibration curve and quality control samples are prepared in alkaline digestion solution that has been neutralized to a pH of 7.5 ± 0.5. To prepare the neutralized alkaline digestion solution, put 500 mL of alkaline digestion solution in a bottle containing a stir bar, and stir while measuring the pH with a meter. Titrate to a pH of 7.5 +/- 0.5 with 5 M HNO3. Approximately 70-80 mL of 5 M HNO3 will be required. Add the acid very slowly below pH 8.5, allowing the pH to stabilize before each new addition. Bring to a final volume of 1000 mL with laboratory reagent grade water. Cap the bottle and shake gently to mix, venting the bottle frequently to allow carbon dioxide to escape.

7.12 Pour approximately 15 mL of neutralized alkaline digestion solution into each of 11 disposable centrifuge tubes, which will contain calibration standards and QC samples.

7.13 A fresh calibration curve must be analyzed at least quarterly (every three months), whenever a new operator begins performing the analysis, and whenever the operating conditions of the spectrophotometer change (e.g. after the lamp is replaced, or the instrument is serviced). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 10 uL, 25 uL, 50 uL, 100 uL, 250 uL, 500 uL, 750 uL, and 1000 uL of Hexavalent Chromium Working Standard to separate appropriately-labeled block digester tubes. Using the graduations on the tubes, bring each solution to a final volume of 20 mL with neutralized alkaline digestion solution. The Cr(VI) concentrations of these calibration standards will be, respectively, 0 mg/L (calibration blank), 0.010 mg/L, 0.025 mg/L. 0.050 mg/L, 0.100 mg/L, 0.250 mg/L, 0.500 mg/L, 0.750 mg/L, and 1.000 mg/L.

7.14 Using a calibrated adjustable pipet prepare a Continuing Calibration Verification (CCV) solution by adding 500 uL of Hexavalent Chromium Working Standard “A” to an appropriately labeled block digester tube. Using the graduations on the tube, bring the solution to a final volume of 20 mL with neutralized alkaline digestion solution. The Cr(VI) concentration of this CCV solution will be 0.500 mg/L.

7.15 Prepare a Continuing Calibration Blank by adding neutralized alkaline digestion solution to an appropriately labeled block digester tube to a final volume of 20 mL.

7.16 Using a calibrated adjustable pipet prepare an Initial Calibration Verification (ICV) solution by adding 500 uL of Hexavalent Chromium Working Standard “B” to an appropriately labeled block digester tube. Using the graduations on the tube, bring

the solution to a final volume of 20 mL with neutralized alkaline digestion solution. The Cr(VI) concentration of this ICV solution will be 0.500 mg/L.

PREPARATION OF DIGESTED SAMPLES

7.17 Place an appropriate volume of the neutralized alkaline digestate of sample into each of two appropriately labeled centifuge tubes (if the pH-adjusted digestate contains a flocculent precipitate, filter it through a 0.45 um syringe filter using a 10 mL disposable plastic syringe prior to placing the aliquots in the block digester tubes). One of these aliquots will serve as a matrix blank, and the other will be reacted with diphenylcarbazide for analysis. For clear or lightly colored digestates, the appropriate volume is 20 mL. For highly colored samples, smaller aliquots should be pipetted into the tube and diluted to 20 mL with neutralized alkaline digestion solution. To avoid matrix interferences, the color of the unreacted analysis aliquot should be no darker than pale yellow

7.18 Prepare analytical spike samples (post-digestion spikes) by placing an appropriate volume of sample into an appropriately labeled block digester tube, adding 500 uL of Hexavalent Chromium Working Standard, and diluting (if necessary) to a final volume of 20 mL with neutralized alkaline digestion solution. The volume of digestate that is used for the analytical spike should be the same as that prepared for the unspiked sample.

COLOR DEVELOPMENT AND SPECTROPHOTOMETRIC ANALYSIS

7.19 At this point, each centifuge tube should contain 20 mL of sample or standard. Add 0.50 mL of diphenylcarbazide solution to each tube containing a standard, an analytical spike sample, or a sample requiring color development. Don't add diphenylcarbazide to the matrix blanks. Instead, add 0.50 mL of acetone to each matrix blank. The samples will not become colored when the diphenylcarbazide is added, because the pH (approx. 7.5) is too high.

7.20 Quickly add 1.5 mL of 10% H_2SO_4 to each sample and matrix blank, starting with the CCV solution. This will adjust the pH of each sample to 2.0 +/- 0.5. The samples will foam slightly due to generation of carbon dioxide, and samples containing Cr(VI) will begin to color. Record the time at which the addition of H_2SO_4 was started.

7.21 Quickly add 3.0 mL of laboratory reagent grade water to each sample and matrix blank to bring to a final volume of 25 mL. (The volume will actually be slightly less than 25 mL, due to the miscibility of acetone and water. This will not matter, however, because calibration standards are processed the same way.)

7.22 Cap each tube and invert several times to mix. Allow the color to develop for 10 minutes (from the time that the H_2SO_4 addition was started).

7.23 Begin measuring the absorbances of the standards and samples at 540 nm with the UV-VIS spectrophotometer, using laboratory reagent grade water to zero the spectrophotometer prior to beginning analysis. Record the absorbances and the analysis times in the Hexavalent Chromium Instrument Runlog (see Figure 1) . Rinse the sample cuvette with laboratory reagent grade water before adding each sample. Then gently tap the plastic block digester tube containing the sample on the counter to release any gas bubbles before pouring the sample into the cuvette. Rinse the cuvette once with sample before filling it with sample and placing it in the spectrophotometer. Analyze the samples in the order that they were prepared, so that the color development time for all samples will be approximately the same. For each digestate, analyze the matrix blank first, and then analyze the reacted sample. The CCV and CCB must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to ensure continuing calibration accuracy. The developed color will fade over time, so all samples should be analyzed within about 30 minutes from the time that H_2SO_4 was added to the first sample. To ensure that all samples have been analyzed within an appropriate period of time, add H_2SO_4 to the CCV first. Because the CCV is analyzed periodically throughout the run and at the end of the run, a low CCV recovery will indicate that too much time has elapsed between color development and analysis.

7.24 All samples that exceed the calibration range must be diluted and reanalyzed. Perform dilutions prior to color development, using neutralized alkaline digestion solution as the diluent and preparing a matrix blank for each diluted sample. Perform color development and analysis of the diluted samples as described in Steps 7.18 – 7.22. Prepare fresh CCV and CCB solutions with any diluted samples, and analyze all diluted samples in a separate analytical sequence, containing QC samples at the appropriate frequency.

Quantitate all samples by entering the absorbance data into the Excel spreadsheet entitled “Hexavalent Chromium in Soils”. The calibration standards are analyzed by linear regression of concentration versus absorbance, and the calibration must have a correlation coefficient of at least 0.995. If the correlation coefficient is less than 0.995, the calibration is not valid, and results may not be reported. For each sample, the solution concentration is calculated by mathematically comparing the corrected absorbance (developed sample absorbance minus matrix blank absorbance) to the absorbances of the standards. The final hexavalent chromium concentrations of the samples are calculated by entering the initial and final digestate volumes, digested wet sample weight, any analytical dilution factor, and total solids for each sample into the spreadsheet. The calculation is performed as follows:

7.24.1 Sample Concentration:

$$\text{Concentration} = \frac{A \times D \times E}{B \times C}$$

where:

A	=	Concentration observed in the digest (mg/mL)
B	=	Initial moist sample weight (g)
C	=	% Solids/100
D	=	Dilution Factor
E	=	Final digestate volume (mL)

7.24.2 All Batch sheets, raw data, and supporting documents are scanned after final review and the resulting images are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3060 and 7196 require the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but samples may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC SAMPLES

8.2 A Continuing Calibration Verification (CCV) solution is analyzed at the beginning of the run, after every ten samples, and at the end of the analytical run. The CCV solution is prepared from the same source as the calibration standards, and at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.3 An Initial Calibration Verification (ICV) solution is analyzed at the beginning of the run before samples. The ICV solution is prepared from a separate source than the calibration standards, and at a concentration near the mid-point of the calibration curve. The result of the ICV must fall within 90% to 110% of the expected values. If an ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.4 A Continuing Calibration Blank is analyzed after each CCV. The absolute values of results of CCBs must be less than the Practical Quantitation Level (PQL). If a CCB fails, results may not be reported from the run until the problem is corrected and a passing CCB has been analyzed.

PREPARATION BATCH QC SAMPLES

8.5 A preparation blank (PBS), consisting of laboratory reagent grade water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL). If a preparation blank fails, results may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL, associated sample results that are greater than or equal to ten times the measured preparation blank concentration may be reported with "B" notation.

8.6 A laboratory control sample (LCS), consisting of spiked laboratory reagent grade water, is prepared with each digestion batch of twenty or fewer samples. The LCS is prepared by adding 100 uL of Hexavalent Chromium Stock Standard "B" to 50 mL of alkaline digestion solution, and carrying this solution through the digestion and analysis process. Results for laboratory control samples must fall within 80% to 120% of the expected value. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.7 Pre-Digestion Matrix Spikes: Both water-insoluble and water-soluble matrix spikes must be analyzed at a frequency of one per digestion batch of 20 or fewer samples. Identification of specific samples for matrix spike analysis is left to the discretion of the client. The soluble spike is prepared by adding 100 uL of Hexavalent Chromium Stock Standard "B" (1000 ug Cr(VI)/mL) to an aliquot of wet soil prior to addition of alkaline digestion solution. The insoluble spike is prepared by adding 10 – 20 mg of lead chromate ($PbCrO_4$) to an aliquot of wet soil prior to addition of alkaline digestion solution. The insoluble spike is used to evaluate dissolution during the digestion process. Both spikes are then carried through the digestion process. Spike recoveries are calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where:

S = Measured concentration of spiked aliquot (mg/kg)
U = Measured concentration of unspiked aliquot (mg/kg)
SA = Amount of spike added (mg/kg)

The acceptance range for pre-digestion matrix spike recoveries is 75% - 125%, if the native sample concentration is less than four times the amount of spike added. If the pre-digestion matrix spike recoveries are not within these limits, and the LCS recovery is within 80% - 120%, a reducing condition within the sample may be indicated. Matrix spike recoveries should be evaluated by the client in light of the oxidizing/reducing tendency of the soil, as indicated by ORP, pH, TOC, and other available measurements.

8.8 Post-Digestion Matrix Spikes: One post-digestion matrix spike (also known as an analytical spike) must be analyzed in each batch of 20 or fewer samples. The post-digestion matrix spike is prepared by adding 500 uL of Hexavalent Chromium Working Standard to the analysis aliquot prior to color development (see Section 7.16). The acceptance range for post-digestion matrix spike recoveries is 85% - 115%, if the native concentration is less than four times the amount of spike added.

If the post-digestion matrix spike recovery is not within these limits, a reducing condition or a chemical interference in the digestate should be suspected.

8.9 Duplicates: One duplicate sample must be analyzed in each batch of 20 or fewer samples. The relative percent difference between duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D_1 = Sample result
D_2= Duplicate sample result

A control limit of 20% RPD is applied to duplicate sample analysis.

8.10 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Methods 3060 and 7196 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Methods 3060A and 7196A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS).

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

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TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Hexavalent Chromium in Soils by USEPA Methods 3060A / 7196A	Initial Calibration, minimum 5 points plus a calibration blank.	Quarterly, prior to sample analysis. (For DoD QSM, daily prior to sample analysis)	Correlation coefficient ≥ 0.995.	Investigate and correct problem, then prepare and analyze a new calibration curve.
	Initial Calibration Verification (ICV, and independently prepared check standard)	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and reanalyze.
	Continuing Calibration Verification (CCV), prepared from the same source as the calibration standards.	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	Following each CCV (at beginning of run, after every 10 samples, and at end of run).	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL. 2) Investigate and correct problem.
	Method Blank (MB) or Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	No analyte detected >PQL (For DoD QSM, no analyte detected > ½ PQL and > 1/10 the amount measured in any sample).	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank conc. 3) For DoD QSM, problem must be corrected and samples reanalyzed. Flagging is only appropriate if samples cannot be reanalyzed.

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

TABLE 1

QC REQUIREMENTS (Continued)

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Hexavalent Chromium in Soils by USEPA Methods 3060A / 7196A	Laboratory Control Sample (LCS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) For DoD QSM, problem must be corrected and samples reanalyzed. Flagging is only appropriate if samples cannot be reanalyzed.
	Soluble and Insoluble Pre-Digestion Matrix Spikes	One of each per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	Correct problem and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.
	Duplicate Sample (D)	One per digestion batch of 20 or fewer samples.	RPD ≤ 20%, if both the sample and duplicate are ≥ four times the PQL.	Flag results
	Post-Digestion Matrix Spike (A)	One per digestion batch of 20 or fewer samples.	Recovery ± 15% of true value, if sample < 4x spike added.	If check indicates interference, dilute and reanalyze sample; persistent interference may indicate the presence of reducing conditions.
	Method Detection Limit (MDL), LOD, and LOQ Studies	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published in method; otherwise QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for the analyte that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Reference blank (reagent water)	Before beginning standards or sample analysis.	NA.	NA.	NA.	Used for blank subtraction of standards, field and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank (using a sample aliquot prepped in accordance with Method 7196A (Section 7.1)).
Initial calibration (ICAL) (minimum three standards and a calibration blank)	Daily ICAL prior to sample analysis.	$r \geq 0.995$.	Correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.

TABLE 2 (cont)

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV) (also known as independently prepared check standard)	Before beginning a sample run.	Value of second source within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 15 field samples and at the end of the analysis sequence.	Value of CCV within ± 10% of true value.	Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Problem must be corrected. No samples may be run until calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. Contact Client if samples cannot be reprepped within hold time.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

TABLE 2 (cont)

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS	One per preparatory batch.	QC acceptance criteria specified by DoD; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Sample matrix verification (also known as matrix spike)	Once for every sample matrix analyzed.	Spike recovery within 85–115%.	If check indicates interference, dilute and reanalyze sample; persistent interference indicates the need to use alternative method or analytical conditions, or to use method of standard additions.	Flagging criteria are not appropriate.	Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in Method 7196A (Sections 7.4 and 7.5).
Matrix spike duplicate (MSD) or sample duplicate	Aqueous matrix: One per every 10 project samples per matrix. Solid matrix: One per preparatory batch per matrix.	Aqueous matrix: RPD ≤ 20% (between MS and MSD or sample and sample duplicate). Solid matrix: RPD ≤ 30%.	Examine project-specific DQOs. Contact the client as to additional measures to be taken.	Flagging criteria are not appropriate.	Refer to sample matrix verification sample for MS data evaluation.
Pre-digestion matrix spikes (solid matrix samples only, Method 3060)	One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis.	MS recoveries within 75–125%.	Correct problem and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.	If corrective action fails, narrate	

TABLE 2 (cont)

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Post-digestion matrix spike	NA.	One per preparatory batch.	Recovery between 85–115%.	Correct problem and rehomogenize, redigest, and reanalyze samples. Persistent interference indicates the need to use an alternative method or analytical conditions, or to use method of standard additions.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-625-07	USEPA Methods 3060A and 7196A, current revisions
Apparatus/Materials	Alkaline digestates filtered through 0.7 um glass fiber filters, because of the difficulty of filtering through 0.45 um membrane filters.	Alkaline digestates filtered through 0.45 um membrane filters.
Reagents	Purchased primary standards are used to prepare calibration standards, QC samples, and matrix spikes.	Standards are prepared from potassium dichromate by the laboratory.
Sample preservation/ handling		
Procedures	1) During digestion, samples are mixed by continuously swirling beakers. 2) Colorimetric analysis performed on 20 mL aliquot, with reagent volumes adjusted accordingly. 3) Curve is not digested	1) Samples are mixed using stirring hotplates. 2) Colorimetric analysis performed on 95 mL aliquot of digestate. 3) Curve is treated same as samples (7196A)
QC - Spikes	1) Failure of matrix spikes requires flagging of associated sample results. 2) Evaluation of low matrix spike recoveries is left to the client.	1) Failure of matrix spikes requires redigestion of samples. 2) Measure pH, ORP, $Fe^{2+,}$ and sulfides to evaluate low spike recoveries.
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks		

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

FIGURE 1

EXAMPLE PAGE FROM HEXAVALENT CHROMIUM INSTRUMENT RUNLOG

KATAHDIN ANALYTICAL SERVICES WG344660 R501415

METHOD SW846 7196A

MATRIX: SOIL / SEDIMENT / SOLID — PQL: 0.5mg/Kg (assumes 2.5g dry weight to 100 mL)

Calibration / Quality Control Checks:

Cal Std. Source ID (K2Cr2O7): WI6867 25 ug/mL — CCV ID: WI7296 25. ug/mL

LCS Stock ID: SWL4338 1000 ug/mL — ICV ID: WI7297 25. ug/mL

Reagents: — Phosphate Buffer ID: WI4990

Diphenylcarbazide Solution ID: WI7163 — 10% H2SO4 ID: WI7318

Alkaline Digestion Reagent ID: ~~13.5~~ WI7159 — Alkaline Digestion Reagent pH: 13.5 AP 2/14/19

SW 3060A Leachate Generation Date: 02/07/19 — Filtration End Time: 18:00

Sample ID	Analysis Time	Dilution	pH adj'd 7.5 +/- 0.5	pH adj'd 2.0 +/- 0.5	Sample to Color Vol. (mL)	Final Color Vol. (mL)	Sample Color/Turb Abs @ 540 nm	Developed Sample Abs @ 540 nm
ICV	~~17~~ 17:53		✓	✓	20.0	25.0		0.373
CCV	17:54		✓	✓				0.386
CCB	55		✓	✓				0.002
Prep Blank	56		✓	✓				0.003
LCS	57		✓	✓				0.586
LOD	58		✓	✓				0.007
LOQ	59		✓	✓	✓			0.015
SM0546-7	18:00		✓	✓	~~20~~ 20		0.000	0.236
SM1015-1	01		✓	✓	15.0		0.005	0.012
-2	02		✓	✓			0.007	0.012
-3	03		✓	✓			0.007	0.017
-4	04		✓	✓			0.013	0.016
-5	05		✓	✓	↓		0.005	0.010
CCV	06		✓	✓	20.0			0.381
CCB	07		✓	✓	↓			0.002
SM1015-6	08		✓	✓	~~15.0~~ 20.0		~~0.003~~ 0.012	~~0.003~~ 0.012
-7	09		✓	✓	15.0		0.005	0.012
-8	10		✓	✓	↓		0.004	0.011
-9	11		✓	✓	5.0		0.023	0.025
-9serDil	12		✓	✓	1.0		0.005	0.006
-9PDS	18:13		✓	✓	5.0	↓	0.023	0.398

Notes: Used reduced initial volumes for several samples to reduce interference. SM1015-9 was very dark in color, and required the largest dilution.

Analyst: — Date: 02/08/19

Checked By: — Date: 02/16/19

WL-038 - Revision 1 - 10/27/2011 QAWL877 0000044

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

FIGURE 2

EXAMPLE SPREADSHEET FOR CALCULATION OF HEXAVALENT CHROMIUM IN SOILS

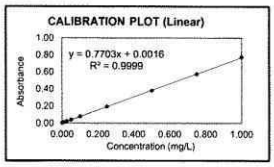
HEXAVALENT CHROMIUM IN SOILS
Preparation Method: 3060A (Alkaline Digestion) Analysis Method: 7196A (Colorimetric)

CALIBRATION INFORMATION

Calibration Analysis Date:	11/15/2018	KCrO4 Stock Standard ID:	W16867
Calibration Analyst:	AP	KCrO4 Stock Standard Conc (mg/L):	25

Standard Name	Vol. Of Stock Std. Added (ml)	Final Volume (mL)	Std. Conc. (mg/L)	Meas. Abs.	Response Factor
Calib. Blank	0.000	25	0.000	0.001	
Std. 1	0.010	25	0.010	0.010	1.000
Std. 2	0.025	25	0.025	0.022	1.136
Std. 3	0.050	25	0.050	0.041	1.220
Std. 4	0.100	25	0.100	0.080	1.250
Std. 5	0.250	25	0.250	0.195	1.282
Std. 6	0.500	25	0.500	0.381	1.312
Std. 7	0.750	25	0.750	0.576	1.302
Std. 8	1.000	25	1.000	0.777	1.287

No. of data points:	9
Corr. Coeff. (R):	0.999941414
Slope (m):	0.770347376
Intercept (b):	0.001624144



SAMPLE ANALYSIS

CCV Source Standard ID:	W17296	Spreadsheet File Name:	020819.xlo
CCV Source Std. Conc. (mg/L):	25	Sample Anal. Date:	2/8/2019
		Sample Analyst:	GFB

	ANALYSIS INFORMATION								DIGESTION INFORMATION				CALCULATIONS		
Katahdin Sample No.	Initial Volume, IV (mL)	Final Volume, FV (mL)	Dil. Fctr.	Sample Absorbance	Matrix Blk. Absorbance	Corrected Absorbance	Calc. Cr+6 in FV (mg/L)	Calc. Cr+6 in IV (mg/L)	Digested Wet Wt. (g)	Digestate Final Vol. (L)	Total Solids (%)	Cr+6 Spike Added (mg)	Calc. Cr+6 in Sample (mg/kgdrywt)	Calc. Cr+6 Spike Added (mg/kgdrywt)	CCV / Spike Recovery
ICV	25.0	25.0	1.000	0.373		0.373	0.482	0.482							96.4%
CCV	25.0	25.0	1.000	0.386		0.386	0.499	0.499							99.8%
CCB	25.0	25.0	1.000	0.002		0.002	0.000	0.000							
Prep Blank	20.0	25.0	1.250	0.003		0.003	0.002	0.002	2.5000	0.100	100.000%		0.09		
LCS	20.0	25.0	1.250	0.586		0.586	0.759	0.948	2.5000	0.100	100.000%	0.100	37.93	40.00	94.8%
LOD	20.0	25.0	1.250	0.007		0.007	0.007	0.009	2.5000	0.100	100.000%	0.000625	0.35	0.25	140.0%
LOQ	20.0	25.0	1.250	0.015		0.015	0.017	0.022	2.5000	0.100	100.000%	0.002	0.87	0.80	108.8%
SM0546-7	2.0	25.0	12.500	0.236	0.000	0.236	0.304	3.803	2.4351	0.100	100.000%		156.18		
SM1015-1	15.0	25.0	1.667	0.012	0.005	0.007	0.007	0.012	2.5211	0.100	87.156%		0.53		
SM1015-2	15.0	25.0	1.667	0.012	0.007	0.005	0.004	0.007	2.4677	0.100	85.446%		0.35		
SM1015-3	15.0	25.0	1.667	0.017	0.007	0.010	0.011	0.018	2.5577	0.100	84.078%		0.84		
SM1015-4	15.0	25.0	1.667	0.016	0.013	0.003	0.002	0.003	2.4166	0.100	83.506%		0.15		
SM1015-5	15.0	25.0	1.667	0.010	0.005	0.005	0.004	0.007	2.4229	0.100	85.198%		0.35		
CCV	25.0	25.0	1.000	0.381		0.381	0.492	0.492							98.5%
CCB	25.0	25.0	1.000	0.002		0.002	0.000	0.000							
SM1015-6	20.0	25.0	1.250	0.012	0.003	0.009	0.010	0.012	2.4220	0.100	90.536%		0.55		
SM1015-7	15.0	25.0	1.667	0.012	0.005	0.007	0.007	0.012	2.4005	0.100	89.381%		0.54		
SM1015-8	15.0	25.0	1.667	0.011	0.004	0.007	0.007	0.012	2.4716	0.100	89.151%		0.53		
SM1015-9	5.0	25.0	5.000	0.025	0.023	0.002	0.000	0.002	2.4571	0.100	76.582%		0.13		
SM1015-9 Serial Diln.	1.0	25.0	25.000	0.006	0.005	0.001	-0.001	-0.020	2.4571	0.100	76.582%		-1.08		
SM1015-9 Post-Dig MS	5.0	25.0	5.000	0.398	0.023	0.375	0.485								97.0%
SM1015-9 Soluble MS	5.0	25.0	5.000	0.027	0.017	0.010	0.011	0.054	2.5114	0.100	76.582%	0.100	2.83	51.99	5.2%
SM1015-9 Insoluble MS	1.0	25.0	25.000	0.505	0.005	0.500	0.647	16.174	2.4182	0.100	76.582%	2.880	873.36	1555.10	56.2%
SM1015-10	15.0	25.0	1.667	0.010	0.005	0.005	0.004	0.007	2.4038	0.100	87.961%		0.35		
CCV	25.0	25.0	1.000	0.381		0.381	0.492	0.492							98.5%
CCB	25.0	25.0	1.000	0.002		0.002	0.000	0.000							
SM0546-7	5.0	25.0	5.000	0.573	0.001	0.572	0.740	3.702	2.4351	0.100	100.000%		152.03		
CCV	25.0	25.0	1.000	0.385		0.385	0.498	0.498							99.5%
CCB	25.0	25.0	1.000	0.002		0.002	0.000	0.000							

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

FIGURE 3

EXAMPLE OF METALS PREPARATION BENCHSHEET

Katahdin Analytical Services, Inc. **Metals Preparation Benchsheet**

Reagent Information:

HNO3:_____ **HCL:**_____ **H2O2:**_____ **Method:** 3060

LCS / Spike **LCS/Spiking Information:**

☐ ☐ **CLPP-SPK-1 (ID/Vol):**_____/_____mL **Hot Plate/Block ID :** 0 **Filter Paper:**_____

☐ ☐ **CLPP-SPK-INT1 (ID/Vol):**_____/_____mL **Start Time/Temp.:** 1015 / 90.0 °C

☐ ☐ **CLPP-SPK-INT2 (ID/Vol):**_____/_____mL **End Time/Temp. :** 1115 / 91.3 °C

☐ ☐ _____ **Spike (ID/Vol):**_____/_____mL **Thermometer ID/Pos.:** Dig 61 /_____

☐ ☐ **CLPP-SPK-4 (ID/Vol):**_____/_____mL

LCSS :_____ **Balance ID:** 1

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
BLANK	MB07CRS1	2.5	g	0.10	L	SL	CR	AP	02/07/2019	N/A	N/A	N/A	N/A		
LCS	MB07CRS1		g		L	SL	CR	AP	02/07/2019	N/A	N/A	N/A	N/A		
LOD	MB07CRS1		g		L	SL	CR	AP	02/07/2019						
LOQ	MB07CRS1		g		L	SL	CR	AP	02/07/2019						
SM0546-7	MB07CRS1	2.4351	g		L	SL	CR	AP	02/07/2019						
SM1015-1	MB07CRS1	2.5211	g		L	SL	CR	AP	02/07/2019						
SM1015-10	MB07CRS1	2.4038	g		L	SL	CR	AP	02/07/2019						
SM1015-2	MB07CRS1	2.4677	g		L	SL	CR	AP	02/07/2019						
SM1015-3	MB07CRS1	2.5577	g		L	SL	CR	AP	02/07/2019						
SM1015-4	MB07CRS1	2.4166	g		L	SL	CR	AP	02/07/2019						
SM1015-5	MB07CRS1	2.4229	g		L	SL	CR	AP	02/07/2019						
SM1015-6	MB07CRS1	2.4220	g		L	SL	CR	AP	02/07/2019						
SM1015-7	MB07CRS1	2.4005	g		L	SL	CR	AP	02/07/2019						
SM1015-8	MB07CRS1	2.4716	g		L	SL	CR	AP	02/07/2019						
SM1015-9	MB07CRS1	2.4571	g		L	SL	CR	AP	02/07/2019						
SM1015-9 INSOL MS	MB07CRS1	2.4182	g		L	SL	CR	AP	02/07/2019						
SM1015-9 SOL MS	MB07CRS1	2.5114	g		L	SL	CR	AP	02/07/2019						

pipettes: W3, W7

Insoluble MS spiked w/ 0.0174 g PbCrO4 (SWL3760)
Soluble MS & LCS spiked w/ 0.10 mL Cr6+ Std (1000 ppm) SWL 4338
LOD spiked w/ 0.025 mL 25ppm Cr6+ LCS Int. W17297
LOQ " "0.080 "

Alkalinedig. Sol'n 13.550 pH
(W17159)
Phos Buffer: W14990
MgCl: SWL 4332

Reviewed 02/16/19

QA-065-Revision 1 - 09/23/2010 **Digestion performed by:** AP **On:** 2/7/19 **Page:** MB020 **Revision:** 00

TITLE: **ALKALINE DIGESTION AND SUBSEQUENT DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID SAMPLES USING EPA SW846 METHODS 3060 AND 7196**

FIGURE 4

EXAMPLE OF BATCH SHEET FOR HEXAVALENT CHROMIUM

WET CHEMISTRY BATCH REPORT
Feb 16 2019, 11:50 am
Batch: WG246660

Parameter: Chromium, Hexavalent
Prep Date: 07-FEB-19

Date Analyzed: 08-FEB-19
Prep Method: SW846 3060A

Analyst Initials: AP
Prep Chemist: AP

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SM0546-7	SAMP	SW846 7196A	5.0000mL	25.000mL	5	152.03	152. mg/Kgdrywt	100	.4	0.62	2.0		
SM1015-1	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.53	J0.53 mg/Kgdrywt	87.	.4	0.23	0.77		
SM1015-10	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	520.35	J0.35 mg/Kgdrywt	88.	.4	0.23	0.77		
SM1015-2	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.35	J0.35 mg/Kgdrywt	85.	.4	0.24	0.81		
SM1015-3	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.84	0.84 mg/Kgdrywt	84.	.4	0.24	0.79		
SM1015-4	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.15	U0.50 mg/Kgdrywt	84.	.4	0.25	0.84		
SM1015-5	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.35	J0.35 mg/Kgdrywt	85.	.4	0.25	0.82		
SM1015-6	SAMP	SW846 7196A	20.000mL	25.000mL	1.2	.55	J0.55 mg/Kgdrywt	90.	.4	0.16	0.55		
SM1015-7	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.54	J0.54 mg/Kgdrywt	89.	.4	0.24	0.79		
SM1015-8	SAMP	SW846 7196A	15.000mL	25.000mL	1.7	.53	J0.53 mg/Kgdrywt	89.	.4	0.23	0.77		
SM1015-9	SAMP	SW846 7196A	5.0000mL	25.000mL	5	.13	U1.6 mg/Kgdrywt	76.	.4	0.80	2.6		
WG246660-1	MBLANK	SW846 7196A	25.000mL	25.000mL	1	.09	U0.24 mg/Kgdrywt	NA	.4	0.12	0.40		
WG246660-2	LCS	SW846 7196A	20.000mL	25.000mL	1.2	37.93	38. mg/Kgdrywt	NA	.4	0.14	0.48		95
WG246660-3	MS	SW846 7196A	5.0000mL	25.000mL	5	2.83	2.8 mg/Kgdrywt	NA	.4	0.78	2.6		5
WG246660-4	MS	SW846 7196A	1.0000mL	25.000mL	25	873.36	870 mg/Kgdrywt	NA	.4	4.0	13.		56

Comments:

SM1015-9	MS/MSD
WG246660-1	SM1015-9
WG246660-2	SM1015-9
WG246660-3	SM1015-9
WG246660-4	SM1015-9

Entered by: jm AP Date: 02/16/19 Accepted by: Date: 02/16/19

TITLE: **TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES**

Prepared By: Wet Chemistry Date: 12/97

Approved By:

Group Supervisor: Keith Tanguay Date: 02/08/01

Operations Manager: John C Dinton Date: 2/8/01

QA Officer: Deborah J. Nadeau Date: 2.8.01

General Manager: Dennis C Hufnagle Date: 2/08/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention and standard method reference. Other minor changes to sections 7, 8 and QA Table.	DN	2.8.01	2.8.01
02	Added ASTM D2216 reference to the SOP.	DN	5.22.02	5.22.02
03	Revised section 7.3.15 to refer to KIMS. Removed references to supervisor - changed to Department Manager. Replaced Figures 1 and 2 with updated ones.	HRC	05.04.04	05.04.04
04	Added wording to Section 8, added laboratory reagent grade water, changed formatting.	LAD	09.07	09.07
05	Corrected formulas in Sect.s 7.3.15 and 7.4.5.1.	LAD	06/08	06/08

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added the requirement to record the oven ID and thermometer ID in logbook. Added references to section 10.	EN	08/09	08/09
07	Updated and/or added references to sections 7, 9 and 10. updated Logbook page.	LAD	06/10	06/10
				LAD 05/12
08	Sect 7 - Edited for new variance with direct upload to KIMS. Added scanning and archival of raw data. Added Blank prep. Other minor changes to reflect current practice and for clarity. Sect. 8 - Added calculation acceptance criteria and blank prep. Sect. 9 - Added MDL, LOD and LOQ info. Sect. 10 - Added and edited references. Table 2 - Added only 2 weighings are performed. Updated Fig. 1 & 2.	LAD	05/12	05/12
09	Throughout - changed KAS, INC to KAS. Sect. 7 - changed 0.0019 to 0.0001g, changed approx. 5g to 25-50g, added sections to describe additional manual weighings; Sect. 8 - Changed duplicate RPD from < 10 to < 5. Sect. 10 - updated references. Table 2 - Removed modification for multiple weighings. Updated Fig.s 1 and 2.	LAD	05/15	05/15
10	Sect 6 - changed 11 T. to 7 days. changed 500 +/- 50°C to 550°C throughout SOP. Updated figures 1 and 2.	LAD	12/17	12/17
11	Added TS-ME reference and changed drying time to overnight throughout SOP. Sect. 1 added method numbers and waste disposal instructions. Sect. 4 - added wooden tongue depressors. Sect. 7 - Many changes to reflect current practices. Sect. 8 & Table 1 - Updated method blank criteria. Added	LAD Figures 2→13	02/19	02/19

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **CA-717-11**, titled **Total Solids/Total Volatile Solids Determination in Solid Matrices.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **CA-717-11**, titled **Total Solids/Total Volatile Solids Determination in Solid Matrices.**

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel to determine the Total Solids (percent solids) and Total Volatile Solids in soil, sediment, sludges and solid matrices in accordance with Standard Methods 2540 G or SW846 3540C (Total Solids only).

1.1 Definitions

Total Solids - The term applied to the "as received" sample where material residue left in the vessel after drying in an oven at 103-105°C.

Decanted total Solids - The term applied to the "decanted" sample where material residue left in the vessel after drying in an oven at 103-105°C.

TS - Total Solids (SW846 3540C)

TS-ME – Total Solids (SM2540G)

Fixed Solids - The residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature.

Volatile Solids - The weight loss on ignition of fixed solids.

Total Volatile Solids - The weight loss on ignition of total solids.

TVS - Total Volatile Solids

Batch - Twenty or fewer samples prepared and analyzed together with handling that is similar to all.

LCS - Laboratory Control Sample

PQL - Practical Quantitation Limit

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the determination Total Solids and Total Volatile Solids. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the determination of Total Solids and Total Volatile Solids to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in

the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data. It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to perform periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Wastes generated by this method can be disposed of using general incineration practices, unless knowledge of the sample matrix determines it to be hazardous waste.

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

2.0 SUMMARY OF METHOD

A quantity of soil, sediment, or solid matrix sample is weighed out, dried at 103-105°C overnight and then re-weighed. The difference in the weights before and after drying represents the "total solids" portion of the sample as defined by this procedure. The percent solids determined by this method are further used in the final calculations for other analyses to convert the analytical value of the sample to a "dry weight basis". The PQL for this procedure is 0.1 wt% for an initial weight of approximately 25-50g.

To determine Total Volatile Solids, the total fixed solids after drying at 103-105°C are ignited at 550 °C. The residue is brought to constant weight and weighed. The loss on ignition between 103-105°C to 550 °C constitutes the measurement of Total Volatile Solids.

3.0 INTERFERENCES

3.1 Total Solids

Residues dried at 103-105°C may retain water of crystallization as well as some mechanically occluded water. Because removal of occluded water is marginal at this temperature, attainment of constant weight may be very slow. The time of drying will be maintained consistently overnight. Loss of CO_2 will result in the conversion of bicarbonate to carbonate. Loss of organic material at this temperature by volatilization may be very slight, however, this is dependent upon the material. The loss of ammonium carbonate and volatile organics may be significant for sediments, sludges, and sludge cakes. After drying some residues are often hygroscopic and rapidly absorb moisture from the air.

3.2 Total Volatile Solids

Negative errors may be present due to loss of volatile constituents at the initial temperature of drying, 103-105°C. The mass of recovered organic matter from sludges and sediments may require a longer ignition time than that for wastewaters. Determination of low concentrations of volatile solids in the presence of high fixed solids may be subject to considerable error. In such instances where volatile constituents are suspected and analytes of interest alternative techniques should be sought for their determination. After drying some residues are often hygroscopic and rapidly absorb moisture from the air.

4.0 APPARATUS AND MATERIALS

4.1 Drying oven - capable of maintaining drying temperature of 103-105°C

4.2 Muffle Furnace for operation at 550 ° C

4.3 Aluminum tins, porcelain crucibles or evaporating dishes

4.4 Analytical Balance accurate to 0.0001g

4.5 Scoopulas or wooden tongue depressors

4.6 Dessicator provided with a desiccant containing a colored indicator of moisture content - with conditioned desiccant; condition by drying at 210°C for one hour (per the manufacturer's directions).

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Reagent Grade Water

5.2 Sand: Muffled at 550°C for 1 hour

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are maintained under storage conditions dictated by analytes of interest. This normally constitutes storage at 4°C. The analytical holding time for Total Solids by Method SM2540G is 7 days from sample collection. SW-846 3540C does not specify a holding time; Katahdin applies14 days as a conservative limit.

The laboratory initiates sample analysis as quickly as possible for both methods to facilitate data availability for use in final calculation of other analytical parameters.

7.0 PROCEDURES

7.1 In accordance with the current revision of Katahdin SOP CA-102, "Balance Calibration", verify balance calibration at the beginning of the day or prior to beginning analysis. Refer to Section 8.0 for acceptance criteria.

7.2 The oven ID must be recorded in the Total Solids/Total Volatile Solids logbook (or on the Work group Report). When preparing for TS exclusively, a Work Group Report may be used in place of a logbook and data may be entered directly using KIMS. See section 7.4.15 through 7.4.41 for instructions to generate a Work Group Report and export data.

7.3 PREPARATION OF CRUCIBLES, EVAPORATING DISHES, OR TINS FOR TVS

7.3.1 Verify that crucible/dish/tin IDs are unique and legible. IDs may be applied to crucibles using porcelain ink or china marker. Tin ID's should be visibly etched

into the tin. Freshly labeled crucibles/dishes should be ignited at 550°C prior to use.

7.3.2 If *total volatile solids* are to be measured, ignite crucible/evaporating dishes in muffle furnace at 550°C for 1 hour. Tins do not need to be muffled.

7.3.3 Check desiccant to assure that it has sufficient blue color. Replace with fresh material if needed prior to removing crucible/evaporating dishes from the oven or muffle.

7.3.4 Place in desiccator with desiccant that has indicator beads showing a blue color. Cool for a minimum of 1 hr. Clean dried/ignited crucible/evaporating dishes may be maintained in a desiccator prior to use. Weigh to nearest 0.0001 g and record dish ID and weight in the solids logbook (Figure 1).

7.4 TOTAL SOLIDS (TS) ANALYSIS

7.4.1 Label and weigh each tin/crucible/evaporating dish before use and record initial (tare) weight in Total Solids logbook (Figure 1). When using tins for weighing total solids, the average weight of the tins may be used for all samples. To obtain an accurate average weight of tins, at least one tin from each box per case of tins must be weighed and the average calculated using a spreadsheet (found on Server A: \\SERVER_A\wetchem\Spreadsheets\Total Solids\TIN TARE WEIGHT AVG\NEW DISH LOT AVG) (Figure 2). Once the average is calculated, a Wet Chem Lab supervisor must be notified to enter the average tare amount in KIMS.

NOTE: It is necessary to obtain a new average tin weight for each new case of total solids tins received, the lot number of the tins should be recorded with the spreadsheet.

7.4.2 Samples will be analyzed under this procedure as "total solids" or "decanted total solids". "Total Solids" will be performed on a well-homogenized sample as received where any aqueous phase is incorporated into the solid matrix prior to aliquotting. "Decanted total Solids" will be performed on sample aliquots designated for extractable organic parameters where it has been determined that the standing aqueous layer must be decanted prior to aliquotting for the organic extractable preparation. Sample login decants the samples during the login process. Decanted samples will be marked with a "DEC" on the cap. The decanted sample aliquot will constitute the "original" sample for total solids determination.

7.4.3 Whether the sample has been decanted or not, remove any obvious non-representative material such as large sticks and rocks. Thoroughly mix the sample with a scoopula (or wooden tongue depressor). Weigh 25-50 g of a

representative sample aliquot into a labeled weighing dish. For samples that are analyzed "as received" and contain a significant standing aqueous layer, a larger aliquot may be used. In order to facilitate drying and minimize moisture entrainment, the sample may be spread out across the surface of the pan/dish/crucible. Please refer to Katahdin SOP, CA-108, Basic Laboratory Technique, current revision, for more information on subsampling.

NOTE: For samples with a low density (i.e., ash), the minimum weight of 25 g may not be attainable. Also, for samples with limited volume available, it may be necessary to communicate with lab supervisors, project managers, and clients to determine how much volume should be used for TS.

7.4.4 Record the weight (to the nearest 0.0001g) of the sample plus dish by uploading the weight directly into KIMS. Please refer to sections 7.4.15 through 7.4.41 for instructions.

7.4.5 A Method Blank and LCS are prepared at a minimum frequency of 1/20 field samples and a Duplicate is prepared at a minimum frequency of 1/10 field samples or per preparation batch if fewer samples. When the batch contains TVS samples the duplicate for TS should be selected from a sample requesting TVS.

7.4.6 A Method Blank consists of a weighing tin/dish/crucible containing about 4.5 grams of muffled dry sand carried through the entire analytical process. The Method Blank result must be 100% TS +/-0.5%. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

7.4.7 The LCS is prepared by spiking muffled dry sand with a known amount of laboratory reagent grade water. Weigh out 4.5 g of sand and 0.50 mL (0.50 g) of laboratory reagent grade water and carry LCS through entire analytical process. Recovery for the LCS must be within 90-110% of the expected true value of 90% solids. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

7.4.8 A Duplicate is prepared by taking a duplicate sample aliquot, 25-50 g, through the procedure as described for the sample. NOTE: The duplicate should be aliquotted at the same time as the original sample.

7.4.9 Transfer samples to drying oven maintained at 103-105°C. Record "date in", "time in", and "temperature in" in appropriate logbook or on the Work Group Report.

7.4.10 Dry samples for overnight. Remove the samples from the oven and cool to ambient temperature in a desiccator. Record "date out", "time out", and "temp out" in logbook or on the Work Group Report.

7.4.11 Weigh the final sample plus dish weights to the nearest 0.0001g.

7.4.12 Record the final weighing in the solids logbook, or export it directly to KIMS.

7.4.13 Repeat the drying (1 hour), cooling, desiccating, and weighing until the weight change is less than 4% or 50 mg, whichever is less.

7.4.14 The raw data (batch number, sample number, pan weight, original sample + pan, and final sample + pan weights) are entered into KIMS for calculation. The calculation performed is as follows:

$$\% \text{ Total Solids} = \frac{(\text{B} - \text{pan, g})}{(\text{A} - \text{pan, g})} \times 100$$

Where: *A* = weight of original sample+ pan, g, and
B = weight of dried sample + pan, g

EXPORTING TOTAL SOLIDS DATA INTO KIMS

CREATING A TS WORK GROUP AND GENERATING A WORK GROUP REPORT

7.4.15 Samples must be work grouped prior to analysis in order to use a Work Group Report instead of a logbook. To work group total solids, one must go to Sample Management on the menu and then to Workstat. A new window opens up and you must make sure the mode is set to Schedule Work (Figure 3).

7.4.16 In the Work Group for Scheduling section, the Work Profile needs to be set to SOLIDS. Click on the new button to get a new work group number the system automatically generates (Figure 3). Click the SAVE and then OK buttons. It may be necessary to record the generated work group number on paper for reference.

7.4.17 In the Query Criteria section, the department number (100), new status section (WIP), product (TS%), matrix (SL), and QC profile (TOTAL SOLIDS) will be already filled in. The OK button may now be selected to show all available samples. To select the samples for the batch, in the available space check with an 'x' (Figure 4). Click save to put them in the work group.

7.4.18 Once all the samples are selected, the add QC button must be hit to add all the pertinent QC for the batch. In the reference column the sample that the QC is associated with must be entered (Figure 5). Click save to save changes. After all this is done, all the windows may be closed.

7.4.19 To print out a Work Group Report, go to Reports on the menu, then Worklist and then Print Work group. A window opens and you must enter the work

group number (unless it automatically generates, this happens when the work group is printed IMMEDIATELY after work grouping samples), the department number the work group is associated with should automatically fill in (Figure 6). Press the OK button and the Work Group Report will generate. Double check the work group to make sure all QC and samples are in the batch, and print.

7.4.20 On the left side of the Work Group Report, there is a column labeled 'Tin' next to 'Sample'. Label the corresponding tins and samples before weighing them out (Figure 7).

COLLECTING TARE WEIGHTS

7.4.21 Total Solid samples to be electronically entered into KIMS must be weighed at all steps on a balance connected to a computer with the WinWedge program (currently serial number 1124016031) and have a connection to KIMS.

7.4.22 From the desktop of the associated computer open up the WinWedge program and the file entitled Ohaus_discovery, in that order. Proceed to open KIMS

7.4.23 Log into KIMS. Work group samples. From the pull-down menu, select Data ► Enter Data.

7.4.24 Enter TARE in the list pointer Field. Select Query by work group and enter the work group number. Under the Run ID section, click New. Record the generated Run ID on either the logbook page or the Work Group Report. Enter the instrument ID (NONE) and analyst. (Figure 8). Click save. Close out the window.

7.4.25 Click OK. Select Modes► All Samples. Click Close. Click the Header button. Enter in the prep date and time in Prep Date 1 and the operator's initials in Comments. Check the box titled Save Measure Date. (Figure 9). Click Close. Click OK to start entering data.

7.4.26 Enter in prep chemist initials in the appropriate column. Record the start of prep time on the logbook page or on the Work Group Report. If the average tin weights are used (see section 7.4.1), the weights should automatically be filled-in in KIMS (Figure 10). If not, take the appropriately labeled empty weighing dish and place it on the balance. Once the weight has settled click the Print key located on the balance. The weight data should be entered in the selected box. Check to make sure both the weight in KIMS and weight displayed on the balance are the same. This is the initial weight of the weighing dish. Do this for all samples in the work group. Click Save. Close all Windows.

COLLECTING WET WEIGHTS

7.4.27 From the pull down menu, select Data ► Enter Data.

7.4.28 Enter WET-WT in the list pointer field. Select Query by work group and enter the work group number. Enter in the Run ID previously assigned to the work group. Click the ok button

7.4.29 Click the Header button. Enter in the prep date in Prep Date 1 and the operator's initials in comments. Check the box titled Save Measure Date. Click Close. Click OK.

7.4.30 Take the appropriately labeled weighing dish filled with sample and place it on the balance. Once the weight has settled click the Print key located on the balance. The weight data should be entered in the selected box. Check to make sure both the weight in KIMS and weight displayed on the balance are the same. Do this for all samples in the work group. Click save. Close all Windows.

COLLECTING DRY WEIGHTS

7.4.31 Heat and cool the samples for the appropriate amount of time.

7.4.32 From the pull down menu, select Data ► Enter Data.

7.4.33 Samples logged in for TS-ME require two dry weight measurements. To collect the first dry weight: Enter TS-FINAL in the list pointer field. Select Query by work group and enter the work group number. Enter in the Run ID previously assigned to the work group. Click the ok button.

7.4.34 Click the header button. Enter in the prep date in Prep Date 1 and the operator's initials in comments. Check the box titled Save Measure Date. Click Close. Click OK.

7.4.35 For all samples, to collect the final dry weight: Enter DRY-WT in the list pointer field. Select Query by work group and enter the work group number. Enter in the Run ID previously assigned to the work group. Click the ok button.

7.4.36 Click the header button. Enter in the prep date in Prep Date 1 and the operator's initials in comments. Check the box titled Save Measure Date. Click Close. Click OK.

7.4.37 Take the appropriately labeled weighing dish filled with dried sample and place it on the balance. Once the weight has settled click the Print key located on the balance. The weight data should be entered in the selected

box. Check to make sure both the weight in KIMS and weight displayed on the balance are the same. Do this for all samples in the work group. Click save. Close all Windows.

DATA APPROVAL

7.4.38 Once the analytical data has been sent to KIMS, the analyst must review the calculated results for completeness and accuracy. In order to do this, the analyst must be logged into KIMS

7.4.39 From the pull-down menu, select Sample Data ▶ Approval. The process ID is WETREV. Click OK. Enter in the work group number. Click OK. (Figure 11)

7.4.40 When approving, if a result is not going to be reported, the analyst must make sure the report flag is set to N.

7.4.41 Once all data is approved a batch sheet is automatically printed which contains all QC and samples within the work group. The results, recoveries, and RPD's are printed on the batch sheet. The analyst must sign and date the batch sheet (Figure 12). Before turning in the data, the analyst must make a copy of the Work Group Report (unless a logbook was used), and put the copied report in to the Total Solids 3 ring binder kept in the Wet Chemistry Lab. Use a sharpie marker to black out client names on the Work List Report if it contains multiple client names when turning in data.

7.5 TOTAL VOLATILE SOLIDS (TVS) ANALYSIS

7.5.1 A Method Blank is prepared at a minimum frequency of 1/20 field samples and a Duplicate is prepared at a minimum frequency of 1/10 field samples or per preparation batch if fewer samples utilizing the aliquots prepared for the TS determination.

7.5.2 For the determination of TVS ignite the residue in the TS crucible/dish/tin in a muffle furnace at 550°C for 1 hour. Start the muffle oven temperature at 200°C, and increase the temp by 50°C every 15-30 minutes, checking to make sure that the sample does not start to smolder/burn. If this happens, reduce the temp increase and change temp at a slower rate. Once the muffle oven temp reaches 550°C, start a 1 hour timer. At the end of one hour, reduce the oven temp and leave the door partly ajar to cool samples before removing them from the oven.

7.5.3 Transfer the dish to a desiccator, cool to room temperature and weigh.

7.5.4 Record the final weighing in the solids logbook.

7.5.5 Total Volatile Solids Calculation

The raw data (batch number, sample number, dish/crucible weight, dried 103-105 °C sample + dish/crucible, and ignited sample + dish/crucible weights) are entered into KIMS (see Current version of Katahdin SOP CA-762, Wet Chemistry Data Entry And Review Using Katahdin Information Management System (KIMS)) for calculation. The calculation performed is as follows:

$$TVS(\%) = \frac{(A - dish, g) - (B - dish, g)}{(A - dish, g)} \times 100$$

Where: A = weight of residue + dish before ignition, g, and
B = weight of residue + dish after ignition, g

7.6 Data Archival

All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of

the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 The analytical balance used must be calibrated using ASTM Class 1 weights (1.0g, 10.0g, and 50.0g) prior to beginning analysis. Acceptance criteria are as follows:

ASTM Class 1 Weight	Acceptance Range
1.0g	0.9995-1.0005g
10.0g	9.9995g-10.0005g
50.0g	49.9995g-50.0005g

Refer to TABLE 1 for summary of QC requirements, frequency, and acceptance criteria.

8.2 One method blank must be analyzed for every batch of twenty or fewer samples. A method blank consists of a weighing dish containing about 4.5 grams of muffled dry sand carried through the entire analytical process. The method blank result must be 100% TS +/-0.5%. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

Refer to TABLE 1 for summary of QC requirements, frequency, and acceptance criteria.

8.3 One laboratory control sample (LCS) must be prepared and analyzed for every batch of twenty or fewer samples. The LCS is prepared by spiking muffled dry sand with a known amount of laboratory reagent grade water. Weigh out 4.5g of sand and 0.5 mL of laboratory reagent grade water and carry LCS through entire analytical process. Recovery for the LCS must be within 90-110% of the expected true value. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

8.4 One in every ten (10) samples is analyzed in duplicate. The precision of duplicate analyses, as measured by Relative Percent Difference (RPD), must be less than 5%. If acceptance criteria are not met, examine sample for physical characteristics that may lead to lack of homogeneity or other matrix related interferences. If no matrix related issues are apparent all associated samples should be reanalyzed if possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of Standard Method 2540 G for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

American Public Health Association (APHA) (2012) Standard method for examination of water and wastewater, 22nd edn. APHA, AWWA, WPCF, Washington, Method 2540G.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, 1979, Method 160.4

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)

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TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

TABLE 1

QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SM 2540G / ASTM D2216-98	Total Solids Determination	Method blank	One per batch of 20 or fewer	Blank result is 100% TS +/- 0.5%	(1) Investigate source of contamination (2) Reprep method blank and reanalyze along with all associated samples
		LCS	One LCS per batch of 20	90-110%	Reprep and reanalyze all affected QC and field samples
		Sample Duplicate	One sample duplicate per ten samples	RPD ≤ 5%	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Or else, reanalyze
		Demonstration of analyst proficiency – 4 replicates	Initially and once annually thereafter	P&A meet method criteria	Repeat P&A study
		MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-717-11	METHOD 2540G current revision or ASTM D2216-98, current revision
Apparatus/Materials		
Reagents	1) Muffled Sand for Blank and LCS	1) No LCS listed
Sample preservation/ handling		
Procedures	Use Tins for TVS	Crucible and Glass used for TVS
QC - Spikes		
QC - LCS	1) Muffled Sand spiked with 0.5 g laboratory reagent grade water for LCS	1) No LCS listed
QC - Accuracy/Precision	1) Duplicate Frequency 1/10	1) Duplicate Frequency 1/20
QC - MDL		

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 1

EXAMPLE OF TOTAL SOLIDS LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

WG213301 R437786

Total Solids: SM2540 G / ASTM D2216			PQL: 0.10%	Total Volatile Solids: SM2540 G / E160.4			PQL: 0.10%	Balance Calibrated?
TS/TVS	In 1	In 2	TS Only	Out 1	Out 2	TVS Only		Y / N
ANALYST IN:	[illegible]	[illegible]	ANALYST OUT:	[illegible]	[illegible]	ANALYST OUT:	[illegible]	In: Y
DATE IN:	9-7-17	9-8-17	DATE OUT:	9-8-17	9-8-17	DATE OUT:	9-11-17	Out (TS): Y
TIME IN:	16:08	12:15	TIME OUT:	10:00	13:47	TIME OUT:	15:50	Out (TVS): Y
TEMP IN:	104°C	104°C	TEMP OUT:	104°C	104°C	TEMP OUT:	550°C	Balance ID:
Oven ID: 107N0042			Muffle Oven ID: 030607 (assigned)					S/N 1124016031

SAMPLE ID	DISH ID	DISH WT (g)	DISH WET WT (g)	1st DISH DRY WT (g)	2nd DISH DRY WT (g)	DISH/ASH WT (g)	TIME
SK7950-1	20	1.3251	29.9835	23.9650	23.9644	~~27.7780~~ 28.0437	9-13-17 10:00
Blank	21	1.2649	5.8652	5.8628	5.8632	5.8559	
SK7950-1 DUP	24	1.3068	36.4084	28.8677	28.8413	27.7880	

9-13-17

WL-009 – Revision 5 – 04/29/2016

Reviewed By: QAWL820

Reviewed Date: 09/13/17

0000089

FIGURE 2

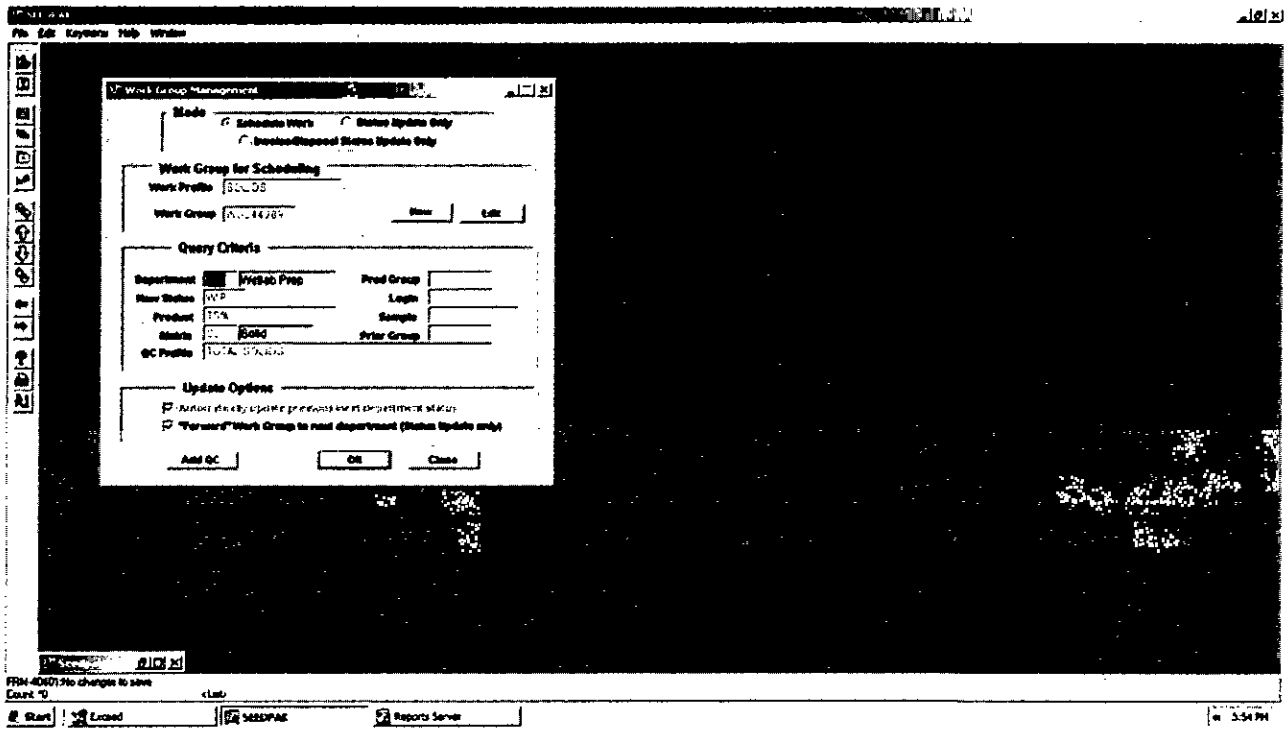
EXAMPLE OF A TIN TARE WEIGHT / NEW DISH LOT AVERAGE SPREADSHEET

Sample	Wt. (mg)	Tin ID
	1.0464	
	1.0049	
	1.0244	
	1.0437	
	1.0037	
	1.0206	
	1.0146	
	1.0134	
	1.0295	
	1.0469	
AVERAGE WT.	1.02481	

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 3

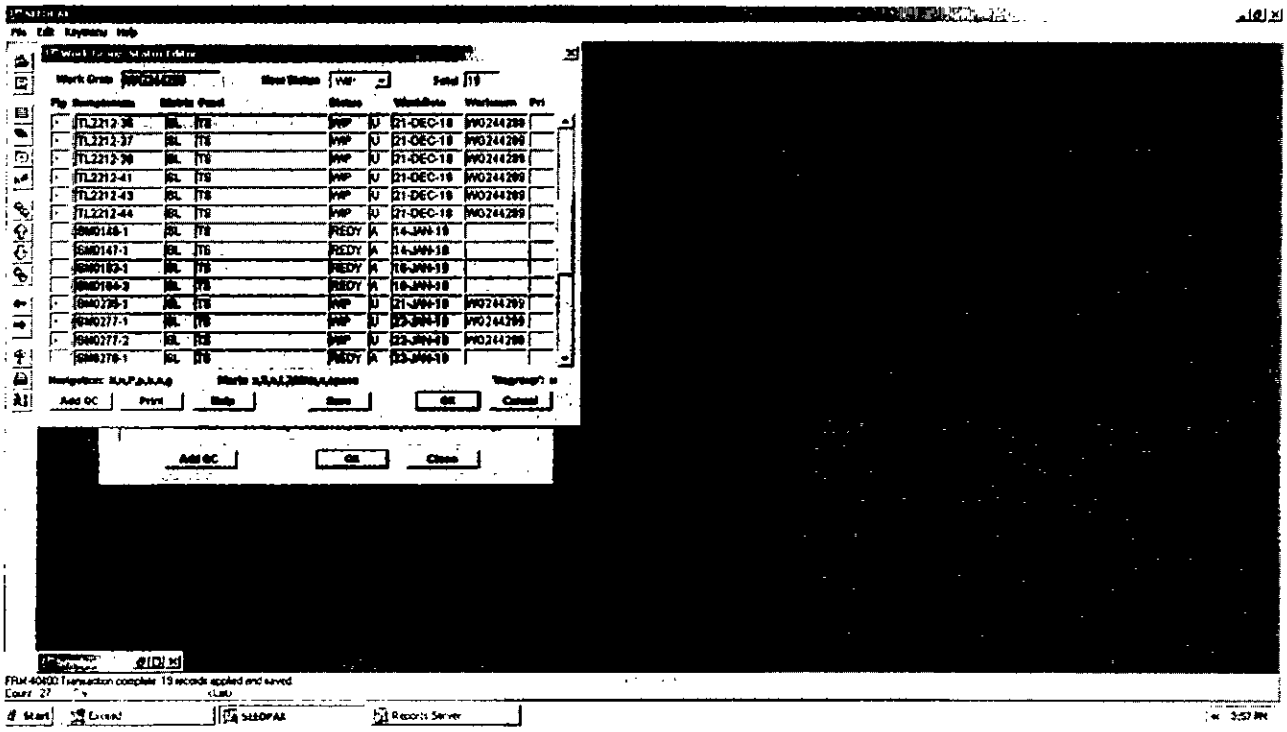
WORK GROUPING TOTAL SOLIDS IN KIMS



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 4

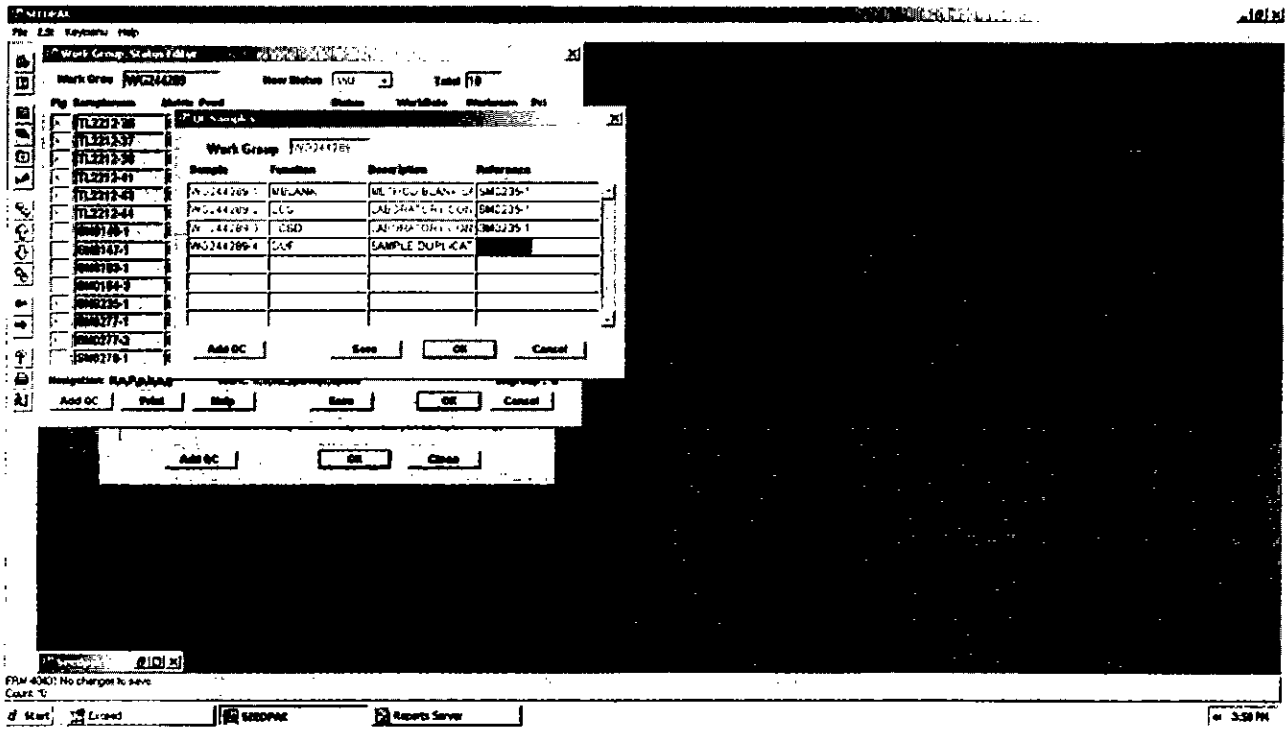
ADDING SAMPLES TO A WORKGROUP



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 5

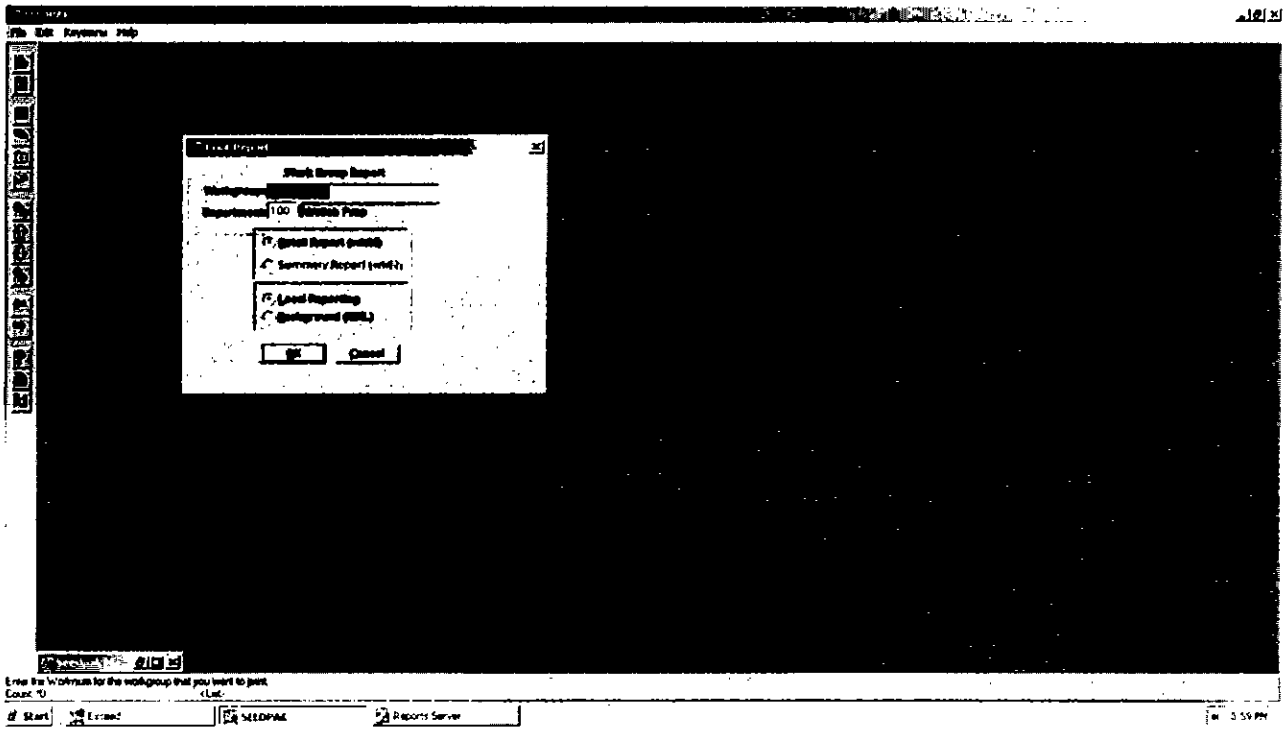
ADDING QC TO A WORKGROUP



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 6

PRINTING A WORKGROUP REPORT



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 7

EXAMPLE OF A WORK GROUP REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Work Group Report (wk02)
24-DEC-18 11:28 AM

Page 1 of 1

Work Group: WG243473 R497188 **Created:** 24-DEC-18
Department: 100 Wetlab Prep **Operator:** **Due:**

Tin	Sample	Account Name	Product	Matrix	Status	UA	Workdate	PR Location
1	TL2535-1	ecoMaine	TS-ME	Solid	WIP	U	30-DEC-18	
2	TL2560-1	Sanford Sewer District	TS-ME	Solid	WIP	U	31-DEC-18	
3	TL2698-1	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
4	TL2698-2	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
5	TL2698-3	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
6	TL2698-4	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
7	TL2698-5	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
8	TL2698-6	Clean Harbors	TS-ME	Solid	WIP	U	28-DEC-18	
9	TL2707-1	Twin Rivers Paper Compan	TS-ME	Solid	WIP	U	02-JAN-19	1L N-Amber Glass
10	WG243473-1	MBLANK	TS-ME	Solid	WIP	U	24-DEC-18	
11	WG243473-2	LCS	TS-ME	Solid	WIP	U	24-DEC-18	
12	WG243473-3	DUP	TS-ME	Solid	WIP	U	24-DEC-18	

Comments:
WG243473-1 TL2707-1
WG243473-2 TL2707-1
WG243473-3 TL2707-1

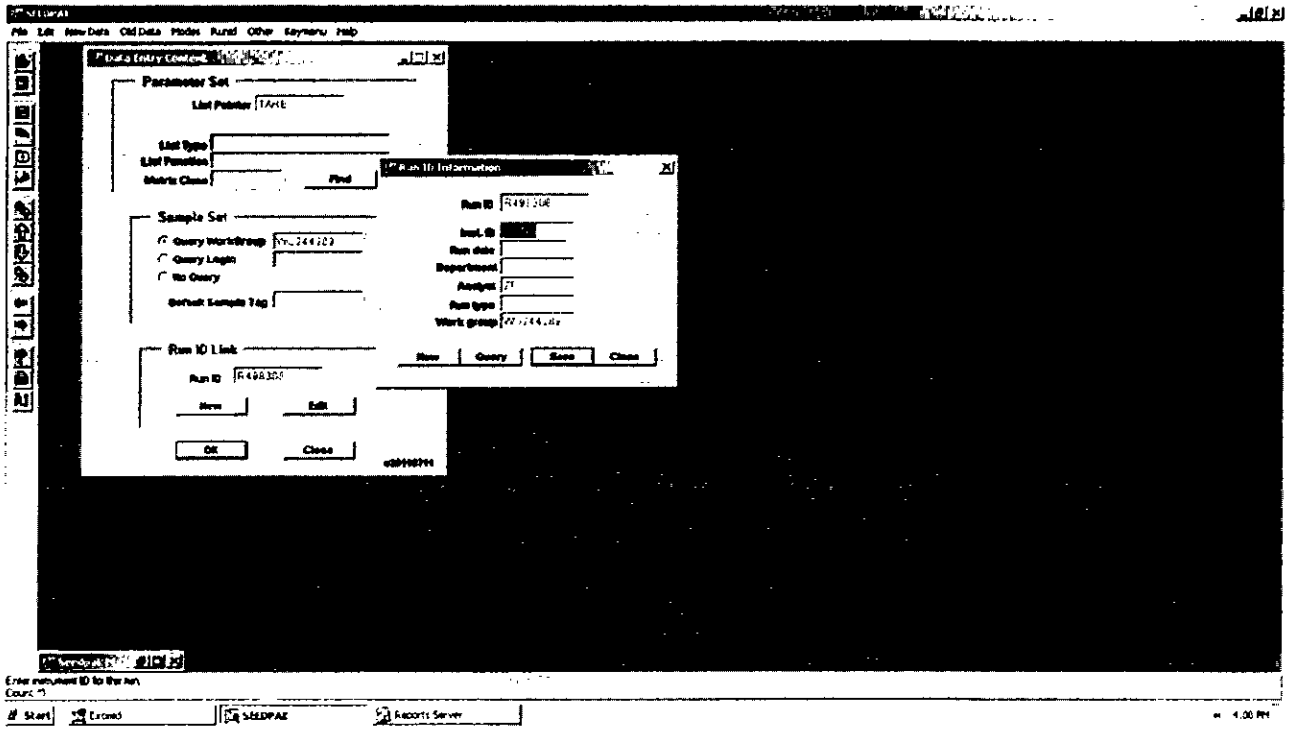
Prep Time: 11:31

Total Solids: SM2540 G / ASTM D2216			PQL: 0.10%	Oven ID: 107N0042		Balance Calibrated?
TS	In 1	In 2	TS	Out 1	Out 2	(Y) N
ANALYST IN:	ZF	ZF	ANALYST OUT:	BP	ZF	In: ✓
DATE IN:	12/24/18	12/26/18	DATE OUT:	12/26/18	12/26/18	Out (TS): ✓
TIME IN:	11:45	10:42	TIME OUT:	0855	11:52	Balance ID: Bal 01
TEMP IN:	104°C	104°C	TEMP OUT:	104	104°C	S/N 1124016031

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 8

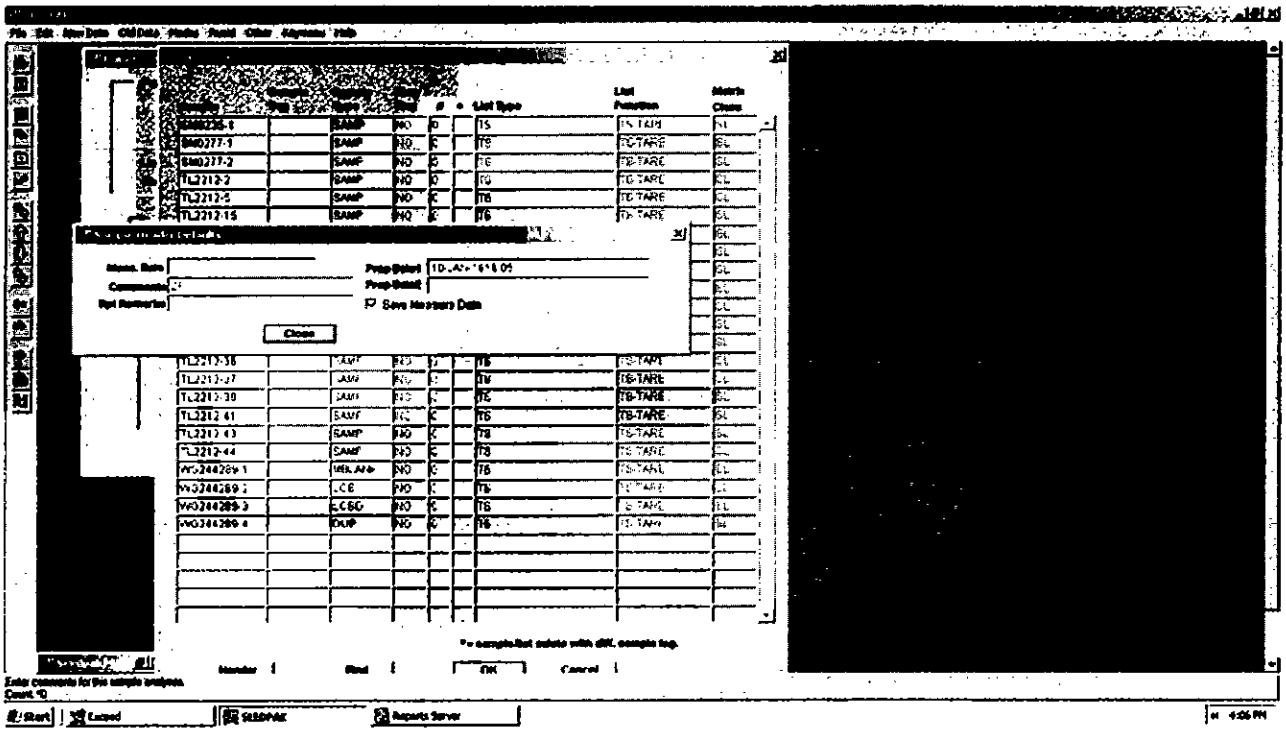
GENERATING A RUN ID



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 9

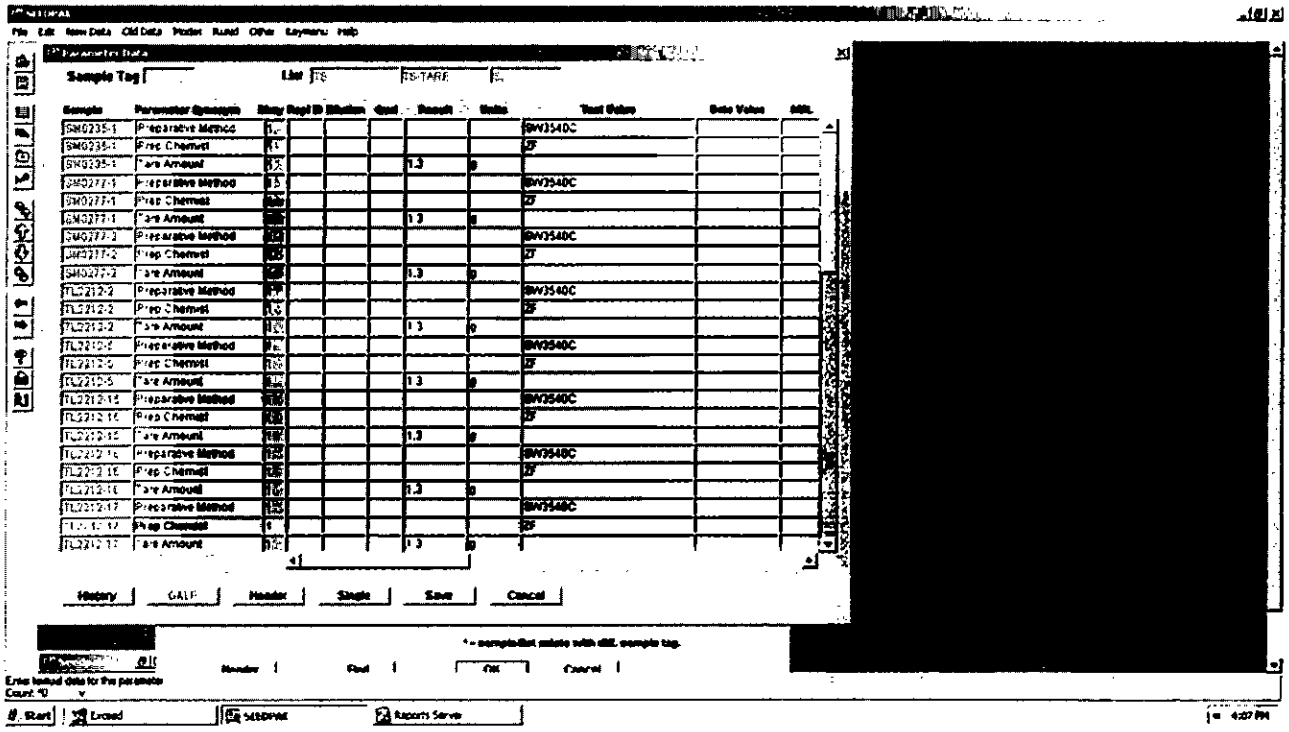
ENTERING HEADER INFORMATION



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 10

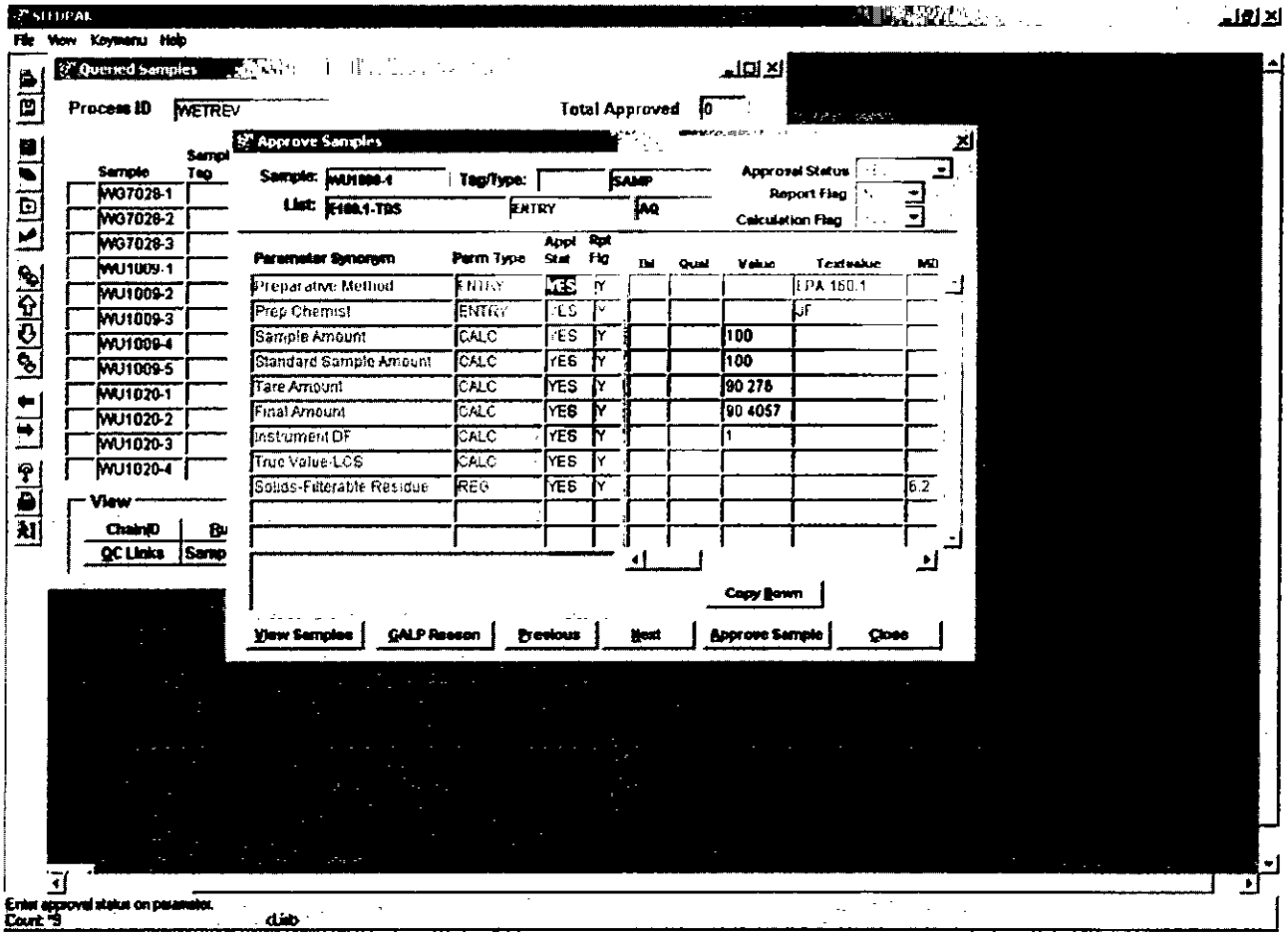
ENTERING TARE WEIGHTS



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 11

DATA APPROVAL SCREEN



TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 12

EXAMPLE OF A TOTAL VOLATILE SOLIDS BATCH REPORT

WET CHEMISTRY BATCH REPORT
Sep 13 2017, 10:48 am
Batch: WG213301

Parameter: Solids-Loss On Ignition
Prep Date: 07 SEP-17

Date Analyzed: 12-SEP-17
Prep Method: SM 2540G

Analyst Initials: AZ
Prep Chemist: AZ

Sample	Samp Type	Method	Tare Amt.	Dry Wt.	Ash Wt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SK7950-1	SAMP	STDM 2540	1.325100g	23.96440g	23.04370g	1	4.06682	4.1 wt %	79.	.1	.1	.1		
WG213301-1	MBLANK	STDM 2540	1.264900g	5.863200g	5.855900g	1	.15875	0.16 wt %	NA	.1	.1	.1		
WG213301-2	DUP	STDM 2540	1.306800g	28.84130g	27.78800g	1	3.82538	3.8 wt %	NA	.1	.1	.1	6	

Comments:

WG213301-1 SK7950-1
WG213301-2 SK7950-1

Entered By: _____ Date: 9-13-17 Accepted By: _____ Date: 09/13/17 ✓

TITLE: TOTAL SOLIDS/TOTAL VOLATILE SOLIDS DETERMINATION IN SOLID MATRICES

FIGURE 13

EXAMPLE OF A TOTAL SOLIDS BATCH REPORT

TOTAL SOLIDS BATCH REPORT
Jan 11 2019, 03:47 pm
Batch: MS244289

Sample	Matrix	Type	Prep Date	Tare	Initial	Final1	Final	by	Date	Raw TS	Rep TS	Recovery	RPD
SM0235-1	SL	SAMP	10-JAN-18	1.3 g	25.7992 g	24.[illegible]957 g	24.[illegible]957 g	ZF	11-JAN-19	94.2710	94. %		
SM0237-1	SL	SAMP	10-JAN-18	1.3 g	29.6725 g	28.5662 g	28.5602 g	ZF	11-JAN-19	94.[illegible]	95. %		
SM0237-2	SL	SAMP	10-JAN-18	1.3 g	26.3375 g	24.4557 g	24.4557 g	ZF	11-JAN-19	92.4820	92. %		
TL2212-35	SL	SAMP	10-JAN-18	1.3 g	30.1565 g	24.6185 g	24.6185 g	ZF	11-JAN-19	87.7392	88 %		
TL2212-36	SL	SAMP	10-JAN-18	1.3 g	24.7298 g	22.2857 g	22.2857 g	ZF	11-JAN-19	89.5680	90 %		
TL2212-37	SL	SAMP	10-JAN-18	1.3 g	26.6185 g	24.0331 g	24.0331 g	ZF	11-JAN-19	89.6030	90 %		
TL2212-2	SL	SAMP	10-JAN-18	1.3 g	22.4974 g	21.2688 g	21.2688 g	ZF	11-JAN-19	94.2047	94 %		
TL2212-26	SL	SAMP	10-JAN-18	1.3 g	26.9908 g	23.4604 g	23.4604 g	ZF	11-JAN-19	8[illegible]	87 %		
TL2212-27	SL	SAMP	10-JAN-18	1.3 g	29.457 g	25.3544 g	25.3544 g	ZF	11-JAN-19	85.4307	85. %		
TL2212-28	SL	SAMP	10-JAN-18	1.3 g	27.6814 g	24.3839 g	24.3839 g	ZF	11-JAN-19	87.5015	88. %		
TL2212-31	SL	SAMP	10-JAN-18	1.3 g	33.2882 g	26.8459 g	26.8459 g	ZF	11-JAN-19	80.1740	80 %		
TL2212-35	SL	SAMP	10-JAN-18	1.3 g	27.0438 g	25.0424 g	25.0424 g	ZF	11-JAN-19	92.2260	92 %		
TL2212-36	SL	SAMP	10-JAN-18	1.3 g	23.4031 g	19.9743 g	19.9743 g	ZF	11-JAN-19	84.4870	84 %		
TL2212-37	SL	SAMP	10-JAN-18	1.3 g	28.2184 g	25.0915 g	25.0915 g	ZF	11-JAN-19	88.3470	88 %		
TL2212-38	SL	SAMP	10-JAN-18	1.3 g	28.1898 g	23.7958 g	23.7958 g	ZF	11-JAN-19	83.6[illegible]	84. %		
TL2212-41	SL	SAMP	10-JAN-18	1.3 g	22.3134 g	18.7359 g	18.7359 g	ZF	11-JAN-19	82.9750	83 %		
TL2212-43	SL	SAMP	10-JAN-18	1.3 g	24.8061 g	21.4508 g	21.4508 g	ZF	11-JAN-19	85.7200	86 %		
TL2212-44	SL	SAMP	10-JAN-18	1.3 g	23.5399 g	19.7049 g	19.7049 g	ZF	11-JAN-19	82.7690	83. %		
TL2212-5	SL	SAMP	10-JAN-18	1.3 g	26.5824 g	22.0425 g	22.0425 g	ZF	11-JAN-19	8[illegible]	8[illegible] %		
MS244289-1	SL	MBLANK	10-JAN-18	1.3 g	5.6432 g	5.6424 g	5.6424 g	ZF	11-JAN-19	99.9820	100 %		
MS244289-2	SL	LCS	10-JAN-18	1.3 g	6.1363 g	5.6327 g	5.6327 g	ZF	11-JAN-19	85.6060	90 %	100	
MS244289-3	SL	LCSD	10-JAN-18	1.3 g	6.1915 g	5.6883 g	5.6883 g	ZF	11-JAN-19	85.7[illegible]	[illegible] %	100	0
MS244289-4	SL	DOD	10-JAN-18	1.3 g	31.5352 g	29.8459 g	29.8459 g	ZF	11-JAN-19	94.4130	94 %		0

Comments:

TL2212-41	MS/Int DOD
MS244289-1	SM0235-1
MS244289-2	SM0235-1
MS244289-3	SM0235-1
MS244289-4	SM0235-1

Entered by:_____ Date:_____ Accepted by:_____ Date:_____

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

Prepared By: Greg Lull Date: 7/2002

Approved By:

Group Supervisor: Keith Tanguay Date: 091102

Lab Operations Mgr: C. Burton Date: 9/11/02

QA Officer: Deborah J. Nadeau Date: 9.11.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1-clarified CCB, Sect. 2-clarified phosphoric acid conc. Sect. 7- reworded software and instr. start up, added TS to calc., removed steps not currently in practice. Sect. 8 - updated LCS and CCV information. Updated Table 1	LAD	01/07	01/07
02	Added method SW846 9060 to title and Table 2. Added 9060 quadruplicate Sample analysis to sect. 7 and Table 1. Removed D.I. water from MB definition 5.3-added TIC std. info. Sect. 7.0 - reworked for clarity on instrument and software instructions. Fixed typos- sect. 2.	LAD	06/08	06/08
03	Minor changes to reflect current equipment, practices and techniques.	LAD	08/09	08/09
04	Added 9060 QC requirements to Table 1. Added method modifications to Table 2. Minor edits to Section 7 to reflect current practice, remove redundancy and for clarification. Updated and/or added references to Section 7.9 and 10.	LAD	06/10	06/10
05	Removed 1:1 phosphoric acid and added 1:1 hydrochloric acid. Added MDL, LOD and LOQ information. Updated Figures 1 → 4.	LAD	02/13	02/13

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 5 – Updated for current CCV and LCS source and preparation.	LAD	05/16	05/16
07	Added Particulate Organic Carbon to Sect. 1&2. Sect. 8 - Added contingency plan. Added Attachment A - preparation and calculation of particulate organic carbon. (POC)	LAD	05/18	05/18
08	Sect. 1 - Added muffled sand to M. Blk and CCB definitions Sect 6 - Added 0.5M HCl to TOC calibration std. prep., Added muffled sand and the preparation. Sect. 7 - Added requirement to dry M. Blk, LCS and CCVs. Updated Logbook Ex.	LAD	01/19	01/19

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP **CA-741-08**, titled **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP **CA-741-08**

, titled **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.**

Recipient: _____ Date: _____

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services technical personnel to determine Total Organic Carbon (TOC) in solids and Particulate Organic Carbon (POC) in aqueous matrices in accordance with EPA Region II Lloyd Kahn method and SW846 9060 Mod.

This method is applicable to sediment, sludges, and soil samples. The detection limit for this method is 100 µg C and a method PQL of 400 µg/g.

This method may also be used to determine the Particulate Organic Carbon (POC) associated with aqueous samples. The preparation and calculation of POC is detailed in Attachment A of this SOP.

1.1 Definitions/Acronyms

TC – Total carbon

IC – Inorganic Carbon

TOC – Total Organic Carbon

POC - Particulate Organic Carbon

Method Blank – A sample boat filled with approximately halfway with muffled sand, no reagents are added and is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is a sample boat filled approximately half way with muffled sand, no reagents are added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of TOC in solids by the Lloyd Kahn Method. Each analyst must demonstrate and document their ability to generate acceptable results with this

method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of TOC in solids by the Lloyd Kahn method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

2.0 SUMMARY OF METHOD

Total Carbon (TC) is measured utilizing a carbonaceous analyzer with a boat sampling module and 900°C furnace attached. The resulting combustion converts carbon-to-carbon dioxide (CO_2) in the presence of oxygen. The amount of CO_2 derived from a sample is directly proportional to the concentration of carbonaceous material in the sample and is then measured by a non-dispersive infrared detector (NDIR).

To determine **Total Organic Carbon** (TOC), however, carbonate and bicarbonate ions contributing to the TC result must be accounted for. This is achieved by adding 1:1 hydrochloric acid to the sample and combusting it at 103° C for 10 minutes to remove any **Inorganic Carbon** (IC) before analyzing the sample. The **Total Carbon** result then equals the **Total Organic Carbon.**

To determine Particulate Organic Carbon (POC), an aqueous sample is filtered through a pre-combusted / pre-cleaned glass fiber filter to obtain the particulates on the filter. The filter + particulates sample is then dried. The dried filter + particulates are then milled, ground, and homogenized prior to removal of an aliquot to place in the sampling boat, treated with HCl to remove IC (using the same procedure as for TOC) and placed into the carbonaceous analyzer for combustion at 900°C. Procedures for POC are detailed in Attachment A of this TOC SOP.

3.0 INTERFERENCES

Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu model TOC-Vcph with NDIR.

4.2 SSM-5000A 900°C furnace with boat sampling module.

4.3 Mettler AE 100 balance (accurate to 0.1 mg) or equivalent.

4.4 Ceramic boats.

4.5 Drying oven capable of maintaining 103-105°C

4.6 Oxygen gas

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

5.0 REAGENTS AND STANDARDS

5.1 TOC Calibration / CCV Standard: D(+) Glucose Reagent ACS Anhydrous, ACROS

5.1.1 Calibration / CCV Solution – Prepare using 25.0 g and bring up to 250 mL with DI water. Before reaching the 250 mL fill line, add 0.5 mL HCl for preservation. Stable for 3 months.

5.1.1.1 Using the table below, each calibration level is prepared by pipetting the appropriate amount of Calibration / CCV Solution into a ceramic boat filled with approximately 0.5 g of sand.

Calibration Level ug C	Weight (mg)	mL of stock
Blank	0	0
200	0.5	0.005
400	1.0	0.01
2000	5.0	0.05
4000	10	0.1
16000	40	0.4
24000	60	0.6

(Upper range limit for TOC is 24,000ugC)

5.1.1.2 The 16000 ug C level is also used as the CCV

5.2 LCS Stock Standard – Dextrose Anhydrous Powder – Prepare by weighing 5.0 and bringing to 50 mL with D.I. Water. Stable for three months.

5.3 TIC Calibration / CCV Standard Sodium Carbonate, anhydrous (11.3% Carbon by weight = 113,000 ug/gC)

Calibration Standards: weigh into ceramic boat.

0.0 mg - Calibration Blank
3.5 mg - 400ugC
17.7 mg - 2000ugC
35.3 mg - 4000ugC
70.7 mg - 8000ugC
212.0 mg - 24,000ugC

(Upper range limit for TIC is 25,000 ugC)

5.4 1:1 Hydrochloric acid / DI water solution

5.5 Sodium Hydrogen Carbonate (14.28% Carbon by weight = 142,857ug/gC) used as LCS run at 4000ugC level (28 mg).

5.6 Muffled Sand – Play sand muffled at 900 ºC for at least 4 hours.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect sediments in a glass jar with Teflon or aluminum foil. Cool and maintain at 4° (±2) C. Analyze within 14 days for Lloyd Kahn and 28 days for SW846 9060.

7.0 PROCEDURES

SET UP AND CALIBRATION

7.1. Turn on TOC-Vcph analyzer, SSM-5000Afurnace, and oxygen supply and connect to the TOC software program.

7.2. Start the TOC-V software program by double clicking the TOC-V icon on the desktop and selecting sample table editor. Enter user name, passwords and press **OK**. Click the “new” icon followed by “sample run”. Choose SSM-5000A from the pull down menu. This activates a sample field spreadsheet in which calibrations, controls, and samples can be inserted and run. Click the “connect” icon and choose “Settings on PC”. This will start the gas flows through both units and will activate the NDIR. Wait for the TC furnace to read 900°C before beginning analysis. The gauges on the soil module for the gas lines should read 150psi and 200psi from right to left. Also place any sample boats that will be used in the furnace for several minutes to bake off any remaining residue.

7.3. To run a calibration, minimize the sample run. Click the “New” icon followed by “calibration curve”. This activates the calibration curve wizard.

7.4. Choose SSM-5000A next to system by using the pull down menu, the click **NEXT** twice. Type in the file name specifying that it is a curve in the name (i.e toccurve030510), and then click **NEXT**. Using the pull down menu select ug as the units and click **NEXT**.

7.5. A 6-point curve (for either TC or IC) must be run at least every 3 months to verify the calibration. The calibration may also be updated as necessary as demonstrated by failure of the Laboratory Control Sample (LCS) or Continuing Calibration Verification (CCV). To add the points to the curve select “(1)” then click **ADD**. In the highlighted **Conc:** box type in the concentration of the first point (24000), and then press **OK**. Click on the next point “(2)” and **ADD**. Continue with previous steps until all 6-points

have been entered. Click **NEXT** twice and then **FINISH**. Now the curve needs to be run.

7.6. Maximize the sample run and highlight the first row. Select insert then calibration curve from the top menu. Select the calibration curve that was just saved and click **OK**. The calibration curve will be renamed with the date and time (i.e toccal030510, note: name must include the word curve or cal, whichever was not used in the original file name) when the start button is clicked.

7.7. Once all the boxes on the top of the sample run say “Ready”, click the start icon (traffic light) on the menu bar. Once the calibration has been started the instrument will prompt two windows.

7.8. The first will state which point will be running, verify the point and press **OK**. The second window will ask for the exact amount of dextrose that has been weighed out. Enter the amount and click OK. For the last point, the blank point, an empty sample boat is used and 500mg is used for the weight.

7.9. Once entered click **OK** and the instrument will prompt a message to move the sample boat to the measure position.

7.10. Open the TC sample port and insert the boat. Close the port tight and set timer for two (2) minutes. These two minutes ensures that the CO2 that entered the instrument has had enough time to go completely through the detector and not give any false positives. When the timer goes off slide the sample boat to the measurement position.

7.11. Once the instrument is done analyzing the sample, about 4-5 minutes, there will be a message prompt to slide the sample boat to the cooling position. After a few minutes it will then prompt again to move the sample boat back in to the port. As soon as the boat has been pulled all the way back the instrument will automatically start the process for the next point. Repeat steps 7.8 to 7.10 for the rest of the calibration.

7.12. When the entire curve is complete, print the sample report and check the curve and its linearity. The correlation coefficient must be greater than or equal to 0.995. The value of the calibration checks must fall within the control limits (80-120% recovery). If not rerun the sample up to two more times. If the calibration check is still out of the acceptable recovery range, recalibrate the instrument and repeat the procedure. If problem persists, remake the standards and repeat the procedure or perform instrument maintenance. If maintenance is required, record in TOC instrument maintenance notebook.

7.13. Once there is a valid calibration curve, the curve must be saved to the method. Click file, then open. Select method from the pull down menu and select tc method soils. Click the SSM-TC tab and change the calibration to the new one. Click OK. Samples

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

are now ready to run. Select "new" file icon on the menu bar and, then click on sample run. Highlight the first row on the sample run. Click the insert sample icon. The parameter box opens, click the method box and select tc method soils. Click next and enter the sample name. Click next twice then finish.

7.14. Using a calibrated analytical balance weigh out 100-500 mg, wet weight, of the sample into a tarred ceramic boat. Using a disposable pipet add 1:1 hydrochloric acid to the sample until there is no more effervescing, usually 2-4 drops. If a sample concentration is greater than 24000 ug/g, a smaller sample amount must be used to achieve a concentration that is within the curve. Samples that require less than 50 mg must be analyzed in quadruplicate and the standard deviations calculated.

For Method Blanks, LCSs and CCVs, 1:1 hydrochloric acid is not needed.

For TC or Percent TC analysis, 1:1 hydrochloric acid is not needed.

NOTE: Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling

NOTE: Samples analyzed by method SW846 9060 must be analyzed in quadruplicate.

7.15. Dry the samples, Method Blanks, LCSs and CCVs in a 103° - 105° C oven for ten minutes. This process removes the inorganic carbon and residual moisture from the samples.

7.16. To start the analysis, click the start icon. The first sample entered will bring up a file name box. Name the file by analysis and date, click OK. Then the box to enter the weight will open. Enter weight and press OK. Follow steps 7.10 and 7.11 with the exception that each sample is now entered separately throughout the batch.

7.17. Calculate the TC or IC (whichever is being preformed) concentration using the following equation:

7.18.

$$\frac{\text{Abs C value (instrument reading) in ug}}{\text{Sample Weight (g)}} \times \frac{100}{\%TS} = \text{TC or IC result in ug/g C}$$

7.18. Workgroup samples and get run ID. Enter true values for the LCS and MS and save. Go back to the spreadsheet and enter "LLOYD", "Percent", or "TOC" in the comments section for the samples you wish to report. Change QC to match workgroup. Data is then exported by selecting the ASCII export option from the file menu. Select "save as" and choose parsefiles on LVSlims. Select TOC and type file name. Click save and wait for data to export, then review data in wetrev.\

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.19. A batch sheet is generated (Figure 4). Raw data, calibrations, and batch sheets are reviewed for completeness and accuracy by the Wet Chemistry supervisor or other qualified designee.

7.20. Analysts file printouts of instrument calibrations and sample data in the lab for approximately 3 months for reference. Prior calibrations are archived and all are available for retrieval**.**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed below and in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A Method Blank is analyzed at the beginning of the run and a CCB is analyzed every ten samples thereafter and at the end of the run. The Method Blank and CCBs are ceramic boats filled approximately halfway with muffled sand. Results must not be greater than the reporting limit (PQL). The PQL for this test is 400mg/kg.

8.2 Analyze an LCS (40-50 mg of Dextrose = 16000-20000ugC for TC) / (28 mg Sodium Hydogen Carbonate = 4000 ug C for IC) with each batch of 20 samples. Acceptance criteria is 80-120% of expected value.

8.3 Run a CCV (40-50 mg Dextrose = 16000-20000 ug C for TC) / (35.3 mg Hydrogen Carbonate = 4000 ug C for IC) every 10 samples and at the end of each batch. Acceptance criteria is 80-120% of expected value.

8.4 Run a duplicate every 20 samples. Run a matrix spike every 10 samples by weighing out the sample and adding 10 mg of dextrose to it for the TC spike or 35.3 mg of hydrogen carbonate for the IC spike. Run a matrix spike duplicate every 10 samples for SW 9060 samples. The recovery can be determined by calculating the theoretical yield from the sample result based on the weight as compared to the native result and adding 4000 ug C that was added from the spike component. The actual yield divided by the theoretical yield will give the recovery.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of EPA SW846 9060 and the Lloyd Kahn method for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Determination of Total Organic Carbon in Sediment, Lloyd Kahn, USEPA Region II, 7/88.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9060.
TOC-V series SSM-5000A user's manual.

Installation and Operation of Shimadzu's Solid Sample Module.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January 2017

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

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TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/ICV	One per prep batch of twenty samples (Lloyd Kahn); one per fifteen samples (SW 9060)	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	CCV at the beginning of the analysis and one after every 10 samples: same conc. as LCS/ICV	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reanalyze all samples back to last acceptable CCV recovery
Sample Quadruplicate	One every twenty samples. SW 9060 – all samples are analyzed in quadruplicate	≤ 30% RSD	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Matrix spike/Matrix spike duplicate	One MS per ten samples(MSD required every 10 samples for SW 9060)	75-125% Recovery ≤ 30% RPD	(1) If LCS in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL and-or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-741-08	METHOD LLOYD KAHN/SW846 9060
Apparatus/Materials		
Reagents	Dextrose solid for TC calibration (Dextrose for LCS) Sodium Carbonate for IC calibration (Sodium Hydrogen Carbonate for LCS)	Potassium Hydrogen Phthalate solution used for calibration.
Sample preservation/ handling		
Procedures	Report one of the duplicate samples using an RPD of 30%.	Report average and range of quadruplicate analysis (SW 9060) Use 3 standard deviation limit (Lloyd Kahn)
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

R487211
WG234890

Katahdin Analytical Services

Carbon Analysis of Solid Samples - Shimadzu TOC-V_{CPH} / SSM-5000A

Analysis Type and Method (Check One)	
___ Total Carbon (SW846 9060M)	___ Total Inorganic Carbon (SW846 9060M)
___ Total Organic Carbon (SW846 9050)	___ Other (Specify):
✓ Total Organic Carbon (Lloyd Kahn)	___ Percent Carbon (SW846 9060M)

Spiking Information		Calibration Information	
LCS Spike Source ID / Compound: WI6776	TV: 16000ug	Calibration Date:	8/18/18
CCV Spike Source ID / Compound: WI6817	TV: 1	Calibration Analyst:	SC
MS Spike Source ID / Compound: WI6776	TV: 4000 ug	Calibration Source ID:	WI6817
Acid Lot Number: WI6677		Pipette IDs:	07, 08

Katahdin Sample Number	Sample Wt. (mg)	Sample Type * (Circle One)	% Recovery
CCV	40	(Wet) Dry	98%
CCB	500	(Wet) Dry	
LCS	40	(Wet) Dry	106%
LCSD	40	(Wet) Dry	108%
SL7696-28	242.7	Wet (Dry)	
-33	240.9	Wet (Dry)	
-34	258.6	Wet (Dry)	
-35	258.0	Wet (Dry)	
SL7786-1	279.3	Wet (Dry)	
-2	258.8	Wet (Dry)	
-3	265.5	Wet (Dry)	
-4	328.6	Wet (Dry)	
CCV	40	(Wet) Dry	103%
CCB	500	(Wet) Dry	
SL7786-5	301.0	Wet (Dry)	
-6	283.0	Wet (Dry)	
-7	242.0	Wet (Dry)	
-8	273.8	Wet (Dry)	
-9	250.2	Wet (Dry)	
CCV	40	(Wet) Dry	103%
CCB	500	(Wet) Dry	

* "Wet" = field-moist sample (as received). "Dry" = oven-dried sample. "TV" = True Value

Analyst: ZF	Analysis Date: 8/19/18
Reviewer:	Review Date:

WL-019- Revision 4 – 06/10/2017 QAWL902 0000017

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

FIGURE 2

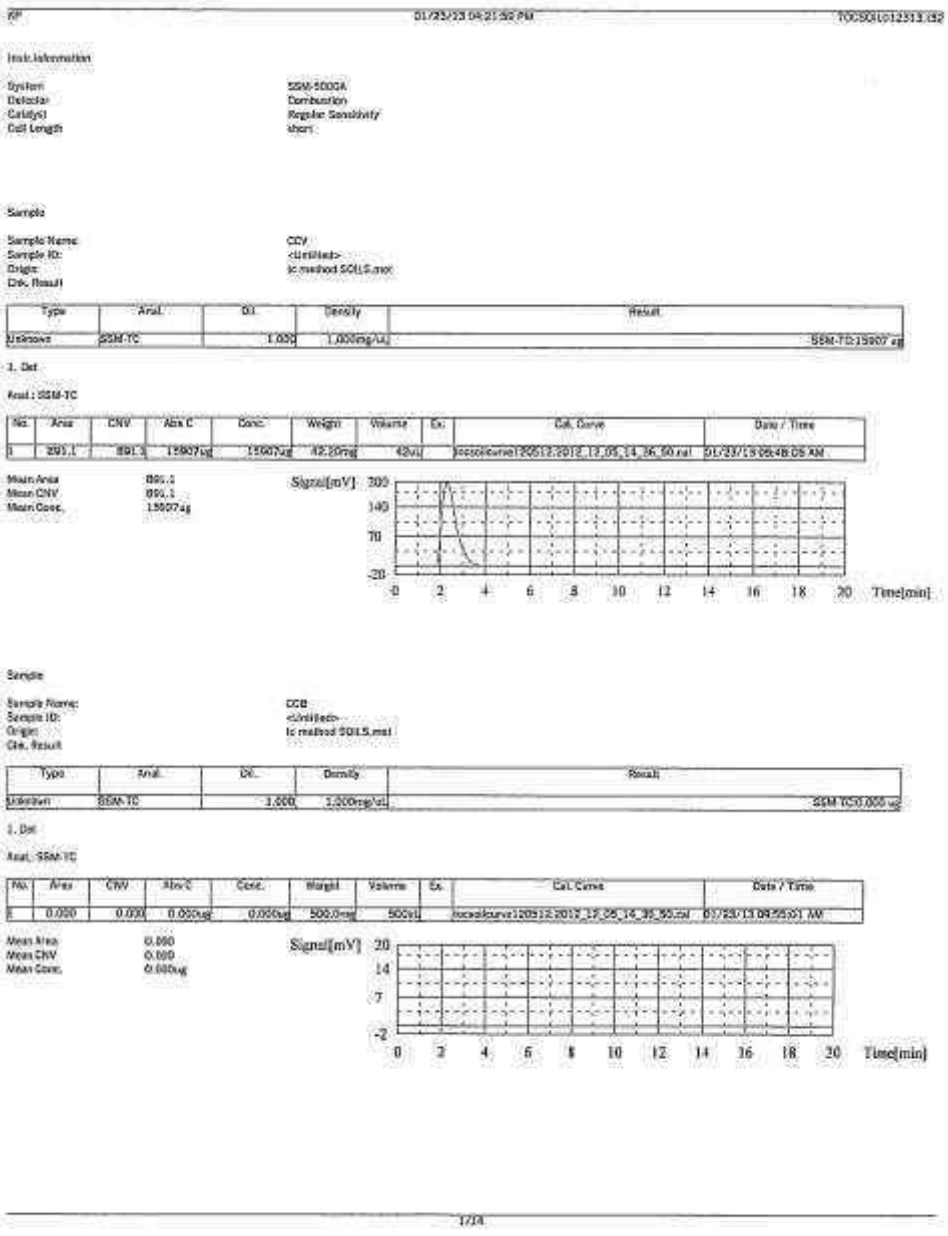
EXAMPLE OF TOC SOLIDS INSTRUMENT PRINTOUT

	Type	Analysis	Sample Name	Dilut	Result	Comment	Status	Date / Time
1	Unknown	SSM-TC	CCV	1.00	SSM-TC:15907 ug		Completed	01/23/13 09:48:06 A
2	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 09:55:01 A
3	Unknown	SSM-TC	LCS	1.00	SSM-TC:15615 ug		Completed	01/23/13 10:10:11 A
4	Unknown	SSM-TC	LCSD	1.00	SSM-TC:15254 ug		Completed	01/23/13 10:18:37 A
5	Unknown	SSM-TC	SG0288-1	1.00	SSM-TC:7567 ug		Completed	01/23/13 10:30:11 A
6	Unknown	SSM-TC	SG0421-1	1.00	SSM-TC:10697 ug		Completed	01/23/13 10:38:56 A
7	Unknown	SSM-TC	SG0421-2	1.00	SSM-TC:8713 ug		Completed	01/23/13 11:49:06 A
8	Unknown	SSM-TC	SG0365-1	1.00	SSM-TC:6723 ug		Completed	01/23/13 12:07:23 P
9	Unknown	SSM-TC	SG0365-1	1.00	SSM-TC:6614 ug		Completed	01/23/13 12:18:03 P
10	Unknown	SSM-TC	SG0365-6	1.00	SSM-TC:4359 ug		Completed	01/23/13 12:27:49 P
11	Unknown	SSM-TC	SG0365-6	1.00	SSM-TC:4475 ug		Completed	01/23/13 12:43:35 P
12	Unknown	SSM-TC	SG0443-1	1.00	SSM-TC:13287 ug		Completed	01/23/13 12:55:28 P
13	Unknown	SSM-TC	CCV	1.00	SSM-TC:15752 ug		Completed	01/23/13 01:07:03 P
14	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 01:14:02 P
15	Unknown	SSM-TC	SG0365-11	1.00	SSM-TC:0.000 ug		Completed	01/23/13 01:31:39 P
16	Unknown	SSM-TC	SG0365-11	1.00	SSM-TC:265.1 ug		Completed	01/23/13 01:43:54 P
17	Unknown	SSM-TC	SG0365-15	1.00	SSM-TC:6585 ug		Completed	01/23/13 01:52:34 P
18	Unknown	SSM-TC	SG0365-15	1.00	SSM-TC:7112 ug		Completed	01/23/13 02:05:11 P
19	Unknown	SSM-TC	SG0365-20	1.00	SSM-TC:7023 ug		Completed	01/23/13 02:19:50 P
20	Unknown	SSM-TC	SG0365-20	1.00	SSM-TC:6512 ug		Completed	01/23/13 02:29:21 P
21	Unknown	SSM-TC	SG0365-20MS	1.00	SSM-TC:11013 ug		Completed	01/23/13 02:41:57 P
22	Unknown	SSM-TC	SG0365-20MS	1.00	SSM-TC:9743 ug		Completed	01/23/13 02:54:30 P
23	Unknown	SSM-TC	SG0473-1	1.00	SSM-TC:105.2 ug		Completed	01/23/13 03:01:58 P
24	Unknown	SSM-TC	SG0473-1MS	1.00	SSM-TC:4586 ug		Completed	01/23/13 03:16:10 P
25	Unknown	SSM-TC	CCV	1.00	SSM-TC:14992 ug		Completed	01/23/13 03:26:56 P
26	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 03:35:08 P
27	Unknown	SSM-TC	SF0473-1MSD	1.00	SSM-TC:5927 ug		Completed	01/23/13 03:48:30 P
28	Unknown	SSM-TC	SX0488-2	1.00	SSM-TC:5709 ug		Completed	01/23/13 03:58:13 P
29	Unknown	SSM-TC	CCV	1.00	SSM-TC:15477 ug		Completed	01/23/13 04:13:04 P
30	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 04:21:43 P

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT



TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

FIGURE 4

EXAMPLE OF TOC SOLIDS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Jan 31 2013, 09:09 am
Batch: WG119373

Parameter: TOC In Soil
Date Analyzed: 23-JAN-13
Analyst Initials: EP

Prep Date: N/A
Prep Method: N/A
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SG0473-1	SAMP	SW846 M9060	141.9mg	200.00ug	1	288215.35693	290000 ug/gdrywt	33.	400	250	1200		
SG0473-1	SAMP	SW846 M9060	315.4mg	200.00ug	1	414.8496	0500 ug/gdrywt	83.	400	100	500		
WG119373-1	MBLANK	SW846 M9060	500mg	200.00ug	1	0	0300 ug/gdrywt	NA	400	85.	400		
WG119373-2	LCS	SW846 M9060	42mg	200.00ug	1	371788.71428	370000 ug/gdrywt	NA	400	85.	400		93
WG119373-3	LCSD	SW846 M9060	40.6mg	200.00ug	1	375714.28571	380000 ug/gdrywt	NA	400	85.	400	1	94
WG119373-4	MS	SW846 M9060	264.9mg	200.00ug	1	21537.40081	22000 ug/gdrywt	NA	400	100	500		112
WG119373-5	MSD	SW846 M9060	282.7mg	200.00ug	1	26082.56576	26000 ug/gdrywt	NA	400	100	500		116

Comments:

SG0473-1	MS/MSD, RE1E3D1-SOIL-011413-726-727
WG119373-1	SG0473-1
WG119373-2	SG0473-1
WG119373-3	SG0473-1
WG119373-4	SG0473-1
WG119373-5	SG0473-1

Entered by: R Date: 1/31/13 Accepted by: [signature] Date: 01/31/13

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

ATTACHMENT A

PARTICULATE ORGANIC CARBON (POC) PREPARATION AND CALCULATION

The following are the additional apparatus, materials, reagents, sample handling, procedures and quality control (in addition to those previously listed for TOC) required for preparing aqueous samples for particulate organic carbon.

4.0 APPARATUS AND MATERIALS

4.1 Oven - capable of maintaining a temperature of 104°C.

4.2 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.3 Glass fiber filter discs without organic binders, 142 mm diameter, nominal pore size 0.7 um. Pre-combusted / pre-cleaned.

4.4 Analytical balance - capable of weighing to 0.1 mg; balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 100g, 50g, 5g, 2g, 0.2g

4.5 Spex Certiprep 8000 Mixer/Mill or equivalent (Ceramic Ball Mixer)

4.6 Desiccator with conditioned indicating desiccant (desiccant is conditioned by drying at 210°C for one hour)

4.7 Graduated cylinder – 1000 mLs.

4.8 Aluminum tins to hold filters for drying

5.0 REAGENTS AND STANDARDS

5.1 Analyte-free water: Water that has been treated to remove organic carbon

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

For this POC method, holding time for aqueous samples has been established as 48 hours from time sampled to time filtered. The analytical hold time of the prepared (dried/homogenized) particulate sample is 28 days from date prepared to date analyzed.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

This holding time can be extended to one year by freezing the prepared/dried POC sample at < 10°C. Holding time is arrested as long as the sample is maintained frozen.

Note: A project may require analysis of Total Suspended Solids (TSS) and Dissolved Organic Carbon (DOC) on the same parent sample as for POC. In such cases, TSS is analyzed first (using pre-combusted / pre-cleaned filter), then the filter + particulates generated during TSS are prepared and analyzed using the procedures in this SOP Attachment A. The Filtrate is acidified and used for DOC analysis.

7.0 PROCEDURES

7.1 Filter Preparation

7.1.1 Pre-combust filters at 500 ºC

7.1.2 Cool combusted filters to room temperature, in a desiccator

7.1.3 The filters are then weighed to 0.0001g. Record the dried filter weight on a small piece of paper. The filters are placed back into their original box with the weight placed on top of the filter. The box of filters must be stored in a desiccator until the time of use

7.2 Assembly filter apparatus



7.3 Place the filter on the support screen and secure.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.4 Record the weight of the prepared filter in the Special Project TSS logbook

7.5 Measure the volume of sample using a graduated cylinder. Typically, the sample volume will be 4L, 1L or 500mL. For POC analysis on aqueous samples, note that for some projects, the entire sample bottle volume must be filtered.

7.6 Quantitatively transfer the sample to the filter holder.

7.7 Gradually apply gentle pressure of 10-30 psi until sample moves through the filter. Collect the filtrate in a 2L polyethylene container.

7.8 When the pressurizing gas begins to move through the filter, or when the liquid flow has decreased at 30 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration. If the project requires the entire sample volume to be used for POC analysis, multiple filters may need to be used to filter the entire sample.

7.10 Rinse the filter apparatus, graduated cylinder and filter 3 times with 50 mL aliquots of filtrate. If DOC is to be measured on the filtrate, fill 3 40 mL VOA vials preserved with H_2SO_4. This MUST be done before any rinses with analyte free water.

7.11 Rinse the filter 3 times with 100 mL aliquots of analyte-free water (this step is especially important for saline samples, where salts may be retained on the filter and affect the final filter weights).

7.12 <u>The material retained on the filter is defined as the POC.</u>

7.13 Filters are placed in an aluminum weighing dish and dried overnight at 104 ºC.

7.14 Cool the filters in a desiccator to room temperature and weigh. Repeat cycle of heating, cooling, and weighing until a constant weight is obtained or until the difference between successive weighings is 0.5mg or 4%, whichever is less. Record the weight in the Special Project TSS logbook.

7.15 Using a ceramic ball mill, grind filter and the POC retained on the filter. Homogenize sample.

7.16 Remove an approximately 0.5g aliquot of the dried, pulverized, homogenized sample and place in sample boat, treat with several drops of HCl solution to remove inorganic carbon (IC) and analyze for organic carbon as for the Lloyd Kahn Method (see TOC method procedure for analysis details).

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.17 Calculations:

$$\frac{\mu g\ C}{\text{Analysis Aliquot (mg)}} * \frac{\text{Total wt mg (Sample + Residue)}}{\text{Residue wt (mg)}} * \frac{\text{Residue wt (g)}}{\text{Vol. Filtered (L)}} = \text{POC } \mu g/L$$

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 A filter Blank is prepared daily for each batch of ≤ 20 samples filtered using 1 L of analyte-free water, or project-specified volume of analyte-free water, and processed in the same manner as the samples. Filter blank result should be non-detect at the sample QL (QL dependent on sample volume).

8.2 A Certified Reference Material (CRM) should be analyzed with the POC at a frequency of 1 per batch of ≤ 20 samples. Acceptance limits are ± 30% of certified value.

8.3 Additional method QC as per TOC method including Method Blank, LCS, MS/MSD or MS/LR, and CCV with same acceptance criteria as TOC (see Table 1 of TOC SOP). Quadruplicate analyses not required for POC. MS/MSD or MS/Lab replicate (LR) frequency 1 per 20 samples.

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

Prepared By: Carrie Pierce Date: 3/11/08

Approved By:

Department Manager: Gary Blewer Date: 03/11/08

Operations Manager: Patrick J. Nadeau Date: 3.11.08

QA Officer: Leslie Dmond Date: 3.11.08

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added definitions to section 6.1. Added references to CA-769 (Konelab) and CA-762 (KIMS). to sections 7 + 10. Added additional references to section 10. Revised Table 1 to include DOD QSM version 4.1 requirements.	DH	08/09	08/09
02	Sect. 8: Changed CAR to Non-conformance Report. Added references to Sect. 10. Added Table 2: DODQSM QC criteria. Removed DoD criteria from Table 1. Added Figure 4 - Test Flow Diagram.	LAD	09/10	09/10
03	Sect. 9 - Added additional MDL, LOD and LOQ information. Sect. 10 - Added information to the Standard Method reference. Figure 5 - Update Run Information Sheet example.	LAD	02/13	02/13
04	Updated title of Sect. 1.4 + 5.0, Changed KAS, KC. to KAS throughout. Updated Method reference. Sect 7.6 - added note for NO MS recovery. Table 3 - added reagent modification. Updated Figure 5.	LAD	09/17	09/17
05	Sect. 5 - Added buffer and NaOH solutions, added to prepare Diphenylcarbazide weekly. Sect. 9 - Added Sample prep according to SM3500 CrC 22nd. Updated references and Figure 5	LAD	01/20	01/20

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-772-05, titled COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-772-05, titled COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER.

Recipient: _____ Date: _____

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin analytical personnel to determine the concentration of hexavalent chromium in groundwater, as well as domestic and industrial wastewaters. This SOP is applicable to samples analyzed using SW846 method 7196 and Standard Methods 3500-Cr B. The effective analytical range is from 0.025 to 1.0 mg/L Cr+6.

1.1 Definitions

CCB- Continuing Calibration Blank. The CCB is deionized water with no reagents added. One CCB is run every ten measurements.

CCV- Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten measurements.

LCS/ICV- Laboratory Control Sample / Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve

LOD – Limit of Detection. The smallest amount or concentration of an analyte that must be present in a sample to be detected at a 99% confidence level. At the LOD, the false negative rate is 1%.

MDL – Method Detection Limit. The minimum amount of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

Method Blank – A deionized water sample that is carried through the entire analytical procedure in the same manner as the sample.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of hexavalent chromium by Method 7196 and standard methods 3500-Cr B. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the analysis of hexavalent chromium by Method 7196 and standard methods 3500-Cr B to read and

understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to indicate periodic review of the associated logbooks

1.3 Safety

WARNING: Diphenylcarbohydrazide is a known carcinogen.

Acidify sample and make reagents in a hood.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

2.0 SUMMARY OF METHOD

Hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. The absorbance of the red-violet color produced is measured photometrically at 540 nm.

3.0 INTERFERENCES

3.1 The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

3.2 Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong, and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

4.0 APPARATUS AND MATERIALS

4.1 Konelab Autoanalyzer, software, and autosampler operation manuals

4.2 Volumetric flasks, Class A

4.3 0.45 micron syringe filters and syringes

4.4 Calibrated adjustable pipettors

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Reagent Grade Water

5.2 Hexavalent Chromium Stock Standard “A”, 1000 ug Cr(VI)/mL – a purchased standard prepared from high purity metals or salts, and supplied by the vendor with certificates of analysis and traceability. Standards must be traceable to NIST standards. This stock standard is the source for calibration standards and CCVs.

5.3 Hexavalent Chromium Working Standard – Place approximately 90 mL of laboratory reagent grade water to a graduated snap-lid container. Pipet 2.5 mL of Hexavalent

Chromium Stock Standard "A" into the container, and bring to a final volume of 100 mL. The concentration of this working standard is 25 mg Cr(VI)/L.

5.4 Hexavalent Chromium Stock Standard "B", 1000 ug Cr(VI)/mL – a purchased standard prepared from high purity metals or salts, and supplied to the vendor with certificates of analysis and traceability. Standards must be traceable to NIST standards. This standard is the source for the LCS.

5.5 Hexavalent Chromium Working LCS – Place approximately 90 mL of laboratory reagent grade water to a graduated snap-lid container. Pipet 2.5 mL of Hexavalent Chromium Stock Standard "B" into the container, and bring to a final volume of 100 mL. The concentration of this working LCS is 25 mg Cr(VI)/L.

5.6 Concentrated sulfuric acid, H_2S0_4 – trace-metals grade.

5.7 Sulfuric acid, 10% (v/v): Add 10 mL of concentrated sulfuric acid to 50 mL of laboratory reagent grade water in a graduated container, and bring to a final volume of 100 mL with laboratory reagent grade water or purchase equivalent.

5.8 1,5 Diphenylcarbazide, analytical reagent grade.

5.9 Acetone (analytical reagent grade): Avoid or redistill material that comes in containers with metal or metal-lined caps.

5.10 Diphenylcarbazide solution: Dissolve 250 mg 1,5-diphenylcarbazide in 50 mL acetone. Store in a brown bottle. Prepare weekly. Discard when the solution becomes discolored.

5.11 Buffer solution: Dissolve 33 g ammonium sulfate in 75 mL of reagent water. Add 6.5 mL ammonium hydroxide and dilute to 100 mL with reagent water.

5.12 Sodium hydroxide, 5N: Dissolve 200 g sodium low-metal impurity NaOH in 1L of reagent water. Store in a plastic bottle.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic containers. Care should be taken to leave little or no headspace in the container. The samples should be stored at 4 (±2) °C until analyses. The maximum holding time prior to analysis is 24 hours.

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

7.0 PROCEDURES

7.1 Sample Preparation (Method 3500-Cr C only): Filter sample through a 0.45 um filter, using a portion of the sample to rinse syringe filter and syringe before collecting the appropriate volume for analysis. Adjust sample pH to between 9.3 and 9.7 by adding 1 mL buffer solution and 600 uL 5N NaOH. More NaOH may be required to bring the sample pH into range.

7.2 Refer to Konelab operation SOP (CA-769) for instrument operation and maintenance procedures.

7.3 Load the 10% sulfuric acid and the hexavalent chromium color reagent onto the reagent wheel as described in the Konelab operation SOP.

7.4 Prepare a 1.0 mg/L calibration standard on the day of analysis by pipetting 1.0 mL of the 25 mg/L working standard into a 25 mL volumetric flask and bringing to volume with DI. Select the CRVI-1.0 and CRVI-0 from the calibrator menu. The Konelab is programmed to make up the other calibrators by diluting the 1.0 mg/L to concentrations of 0.5, 0.10, 0.05, 0.02, and 0.01 mg/L. DI water is used for the CRVI-0. Calibrate the instrument as instructed in the Konelab operational SOP.

7.5 Prepare a 0.50 mg/L LCS on the day of analysis by pipetting 0.50 mL of the 25 mg/L working LCS into a 25 mL volumetric flask and bring it to volume with DI.

7.6 Prepare a 0.50 mg/L CCV on the day of analysis by pipetting 0.50 mL of the 25 mg/L working standard into a 25 mL volumetric flask and bring it to volume with DI.

7.7 Prepare a matrix spike by adding 0.5 mL of the 25 mg/L working LCS to 25 mL of sample.

NOTE: When there is no matrix spike recovery, or when the sample results indicate interference, the sample and sample matrix spike needs to undergo an external 5-fold dilution and be renanalyzed. When preparing a 5-fold dilution of the matrix spiked sample: dilute the sample first and then spike on top of the dilution to conserve the MS true value (See Table 2).

7.7 Refer to the SOPs for Konelab instrument operation (CA-769) and Wet Chemistry Data Entry (CA-762) for sample analysis and reporting procedures.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not

cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Laboratory Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Every instance of noncompliant method quality control requires the generation of a non-conformance report describing the problem, suspected cause and final resolution. Non-conformance reports must be signed by the initiator, Department Manager, QA officer, and lab management.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all

parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of Methods 7196A and 3500Cr-B for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 7196A.

"Standard Methods for the Examination of Water and Wastewater", Method 3500-Cr B, Colorimetric Method, 22nd Edition, 2012, approved by Standard Method Committee, 2009.

Aquakem Konelab Instrument Manual

Aquakem Chromium(VI) Method

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP CA-769, Operation and Maintenance of the Automated Konelab Multiwavelength Photometric Analyzer, current revision.

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS), current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Hexavalent Chromium / SW846 7196 and SM 3500Cr-B	Initial Instrument Calibration (ICAL) (6 standards and a blank)	Daily prior to sample analysis	R ≥ 0.995	Correct problem and recalibrate
	Method Blank	One per analytical batch of 20	No analyte detected >PQL	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.
	LCS/ICV	One per analytical batch of 20 samples (SM 3500) One per analytical batch of 15 samples (SW7196)	90-110%R	(1) If the LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
	CCV	One every 10 measurements	90-110%R	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
	Matrix Spike	One every 10 samples or per sample matrix, whichever is more frequent	80%-120% rcvy. for SM 3500Cr-B and 85%-115% rcvy. for SW-846 7196	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (3500Cr-B only) (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) For 7196, dilute a new pH adjusted aliquot, respike and reanalyze to confirm matrix
	Sample Duplicate or Matrix Spike Duplicate	One per 10 samples	RPD ≤ 20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration after consultation with client
	Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter.	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL study LOD/LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published in method; otherwise QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for the analyte that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Reference blank (reagent water)	Before beginning standards or sample analysis.	NA.	NA.	NA.	Used for blank subtraction of standards, field and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank (using a sample aliquot prepped in accordance with Method 7196A (Section 7.1)).
Initial calibration (ICAL) (minimum three standards and a calibration blank)	Daily ICAL prior to sample analysis.	$r \geq 0.995$.	Correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV) (also known as independently prepared check standard)	Before beginning a sample run.	Value of second source within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (Cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. Contact Client if samples cannot be reprepped within hold time.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per preparatory batch.	QC acceptance criteria specified by DoD; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Sample matrix verification (also known as matrix spike)	Once for every sample matrix analyzed.	Spike recovery within 85–115%.	If check indicates interference, dilute and reanalyze sample; persistent interference indicates the need to use alternative method or analytical conditions, or to use method of standard additions.	Flagging criteria are not appropriate.	Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in Method 7196A (Sections 7.4 and 7.5).
Matrix spike duplicate (MSD) or sample duplicate	Aqueous matrix: One per every 10 project samples per matrix. Solid matrix: One per preparatory batch per matrix.	Aqueous matrix: RPD ≤ 20% (between MS and MSD or sample and sample duplicate). Solid matrix: RPD ≤ 30%.	Examine project specific DQOs. Contact the client as to additional measures to be taken.	Flagging criteria are not appropriate.	Refer to sample matrix verification sample for MS data evaluation.
Pre-digestion matrix spikes (solid matrix samples only, Method 3060)	One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis.	MS recoveries within 75–125%.	Correct problem and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.	If corrective action fails, apply J-flag to the analyte in all samples in the associated preparatory batch.	

TABLE 2 (Cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Post-digestion matrix spike	NA.	One per preparatory batch.	Recovery between 85–115%.	Correct problem and rehomogenize, redigest, and reanalyze samples. Persistent interference indicates the need to use an alternative method or analytical conditions, or to use method of standard additions.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-772-05	Method
Apparatus/Materials		
Reagents	Diphenylcarbazide solution: Discard when solution becomes discolored.	3500-Cr B: Prepare weekly. Discard if the solution becomes discolored.
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF CALIBRATION CURVE

Calibration results AquaKem 6.5 Page: 1

KONELAB #1
KATAHDIN ANALYTICAL SERVICES

01.02.2008 09:47

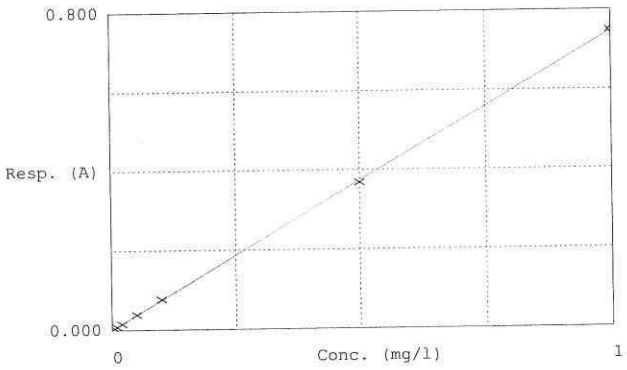
Test CHROMIUM

Accepted 01.02.2008 09:47 KK V. 2.01.08

Factor 1.341
Bias 0.001

Coeff. of det. 0.999926

Errors Meas. error



	Calibrator	Response	Calc. con.	Conc.	Errors
1	CRVI-0	0.001	0.00037	0.00000	
2	CRVI-1.0	0.008	0.01003	0.01000	Blank resp. low
3	CRVI-1.0	0.016	0.02015	0.02000	Blank resp. low
4	CRVI-1.0	0.039	0.05114	0.05000	Blank resp. low
5	CRVI-1.0	0.077	0.10202	0.10000	Blank resp. low
6	CRVI-1.0	0.368	0.49309	0.50000	Blank resp. low
7	CRVI-1.0	0.749	1.00319	1.00000	Blank resp. low

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 2

EXAMPLE OF SAMPLE PRINTOUT

Test results AquaKem 6.5 Page: 1

KONELAB #1
KATAHDIN ANALYTICAL SERVICES

01.02.2008 11:21

Test: CHROMIUM

KC 2.1.08

Sample Id	Result	Dil. 1 +	Response	Errors
CRVI-CCV	0.5157	0.0	0.385	Blank resp. low
CRVI-CCB	0.0009	0.0	0.001	Blank resp. low
CRVI LCS	0.5035	0.0	0.376	Blank resp. low
EB013108	0.0017	0.0	0.002	Blank resp. low
EB013108 -3	0.0006	0.0	0.001	Blank resp. low
EB013108MS -4	0.5034	0.0	0.376	Blank resp. low
CRVI-CCV	0.5162	0.0	0.386	Blank resp. low
CRVI-CCB	0.0017	0.0	0.002	Blank resp. low

SB0562-5

WG48012

R76175

N	4
Mean	0.2523
SD	0.28998
CV%	114.94

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 3

EXAMPLE OF EXCEL SPREADSHEET

AquaKem v. 6.5 AQ1									
Results from time period:									
Thu Jan 03 09:15:14 2008									
Thu Jan 03 09:40:01 2008									
Sample Id	Test short name	Result	Result unit	Result date and time	Dil. Factor	Response	Blank init abs. (A)	Main abs. 1	Comments
CL-0	CHLORIDE	0.3732	mg/l	1/3/08 9:27:19	1	0.0225	0.25516	0.27766	
CL-0	CHLORIDE	-1.0435	mg/l	1/3/08 9:27:20	1	0.01731	0.25708	0.2744	
CL-100	CHLORIDE	0.1236	mg/l	1/3/08 9:27:21	100	0.02159	0.25905	0.28064	
CL-100	CHLORIDE	0.8069	mg/l	1/3/08 9:27:22	100	0.02408	0.25568	0.27976	
CL-100	CHLORIDE	4.8214	mg/l	1/3/08 9:27:23	20	0.03859	0.2621	0.3007	
CL-100	CHLORIDE	4.7919	mg/l	1/3/08 9:27:24	20	0.03849	0.2565	0.29499	
CL-100	CHLORIDE	15.8169	mg/l	1/3/08 9:27:25	6.7	0.07689	0.25483	0.33173	
CL-100	CHLORIDE	16.0643	mg/l	1/3/08 9:27:26	6.7	0.07773	0.25392	0.33165	
CL-100	CHLORIDE	26.889	mg/l	1/3/08 9:27:27	4	0.11333	0.25754	0.37087	
CL-100	CHLORIDE	25.8632	mg/l	1/3/08 9:27:28	4	0.11004	0.25638	0.36642	
CL-100	CHLORIDE	47.6352	mg/l	1/3/08 9:27:33	2	0.17584	0.26128	0.43712	
CL-100	CHLORIDE	48.9049	mg/l	1/3/08 9:27:34	2	0.17942	0.26126	0.44068	
CL-100	CHLORIDE	96.9959	mg/l	1/3/08 9:27:35	1	0.29439	0.26116	0.55554	
CL-100	CHLORIDE	103.9449	mg/l	1/3/08 9:27:36	1	0.3078	0.28481	0.59261	
CL-CCV	CHLORIDE	50.9516	mg/l	1/3/08 9:39:14	1	0.18514	0.25954	0.44468	101
WG47148-1	CHLORIDE	0.2753	mg/l	1/3/08 9:39:15	1	0.02214	0.26106	0.2832	
WG47148-2	CHLORIDE	36.8478	mg/l	1/3/08 9:39:16	1	0.14427	0.25725	0.40152	
SA7479-1	CHLORIDE	14.926	mg/l	1/3/08 9:39:17	1	0.07387	0.25324	0.32711	
WG47148-3	CHLORIDE	16.8349	mg/l	1/3/08 9:39:18	1	0.08033	0.25925	0.33958	
WG47148-4	CHLORIDE	65.2696	mg/l	1/3/08 9:39:19	1	0.22307	0.25649	0.47956	
SB0009-1	CHLORIDE	3.5486	mg/l	1/3/08 9:39:20	1	0.03402	0.25261	0.28664	
SB0014-1	CHLORIDE	28.9095	mg/l	1/3/08 9:39:21	1	0.11975	0.25793	0.37768	
CL-CCV	CHLORIDE	51.4299	mg/l	1/3/08 9:40:00	1	0.18646	0.26173	0.44819	103
CL-BLANK	CHLORIDE	-0.704	mg/l	1/3/08 9:40:01	1	0.01855	0.26355	0.28211	

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 4

EXAMPLE OF BATCH SHEET

WET CHEMISTRY BATCH REPORT
Feb 01 2008, 08:02 pm
Batch: WG48012

Parameter: Chromium, Hexavalent
Prep Date: N/A

Date Analyzed: 01-FEB-08
Prep Method: N/A

Analyst Initials: KK
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SB0562-5	SAMP	SW846 7196A	95.000mL	95.000mL	1	.0017	J0.0017 mg/L	NA	.025	.00076	.025		
WG48012-1	MBLANK	SW846 7196A	95.000mL	95.000mL	1	.00092	U0.025 mg/L	NA	.025	.00076	.025		
WG48012-2	LCS	SW846 7196A	95.000mL	95.000mL	1	.50346	0.50 mg/L	NA	.025	.00076	.025		101
WG48012-3	DUP	SW846 7196A	95.000mL	95.000mL	1	.00063	U0.025 mg/L	NA	.025	.00076	.025	NC	
WG48012-4	MS	SW846 7196A	95.000mL	95.000mL	1	.50339	0.50 mg/L	NA	.025	.00076	.025		100

Comments:

SB0562-5	MS/MSD
WG48012-1	SB0562-5
WG48012-2	SB0562-5
WG48012-3	SB0562-5
WG48012-4	SB0562-5

Entered by: [signature] Date: 2.01.08 Accepted by: [signature] Date: 2/2/08

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 5

EXAMPLE OF ANALYSIS RUN INFORMATION SHEET

KATAHDIN ANALYTICAL SERVICES, LLC
Wet Chemistry Analysis Run Information Sheet

Analyte: HEXAVALENT CHROMIUM	**Analyst:**
Instrument: KONELAB 20	**Analysis Date:**

Analytical Method (Check all that apply):

☐ SW846 7196 ☐ SM 3500-Cr B ☐ Other (list): _____

Reagent Information:

Reagent Name	Reagent ID	Expiration Date
Diphenylcarbazide Solution		
10% v/v Sulfuric Acid		

Standards Information:

Standard Name	Concentration (mg/L as Cr^6)	ID	Expiration Date
Cal. Standard / CCV Stock	1000.0		
Cal. Std. / CCV Intermediate	25.0		
1.0 mg/L Calibration Standard - (1.00 mL / 25.0 mL dilution of Cal. Standard / CCV Intermediate)	1.00	HC_Cal	Day of use only
CCV - (0.50 mL / 25.0 mL dilution of Cal. Standard / CCV Intermediate)	0.50	HC_CCV	Day of use only
LCS / MS Stock	1000.0		
LCS / MS Intermediate	25.0		
LCS - (0.50 mL / 25.0 mL dilution of LCS / MS Intermediate)	0.50	HC_LCS	Day of use only

Notes:

1) Additional calibration standards (0.50, 0.10, 0.050, 0.020, 0.010 mg/L) are automatically prepared by the instrument during analysis through dilution of the 1.0 mg/L Calibration Standard.
2) Matrix spikes are prepared prior to analysis by adding 0.5 mL of LCS / MS Intermediate to 25 mL of sample. Matrix spike added = 0.50 mg/L as Cr6.

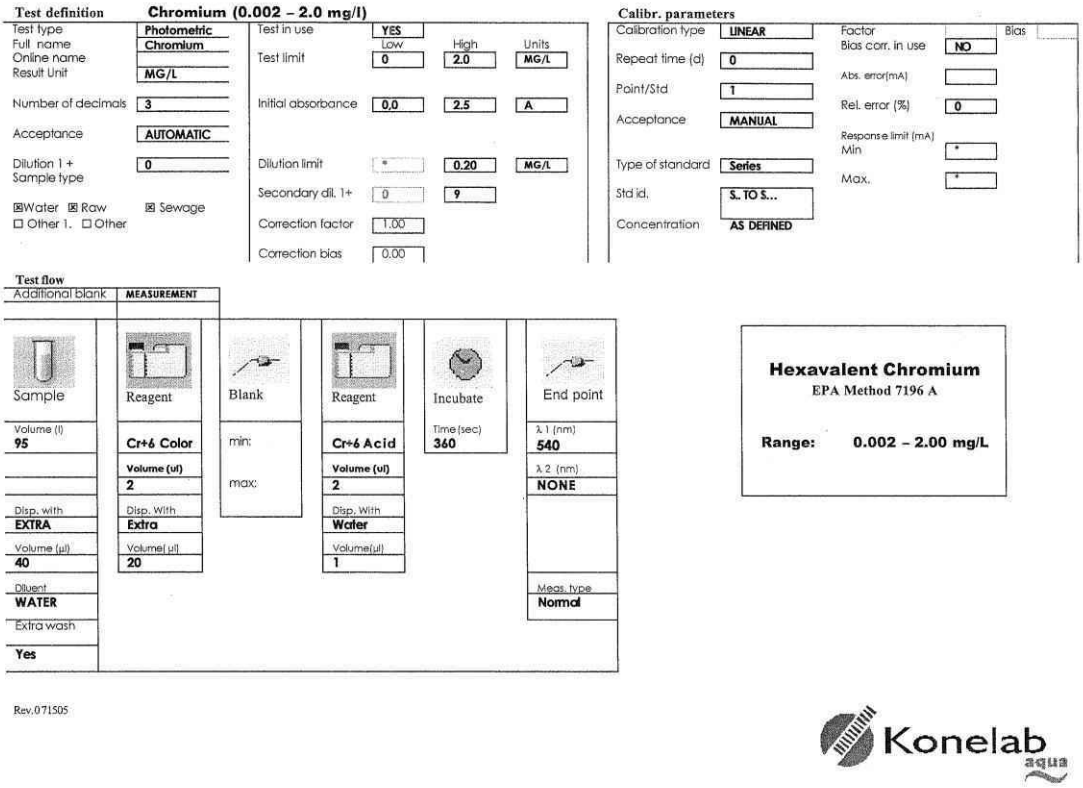
Comments:

WL-051 – Revision 2 – 01/06/2016

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 6

TEST FLOW DIAGRAM



CERTIFICATE OF ACCREDITATION

ANSI National Accreditation Board

11617 Coldwater Road, Fort Wayne, IN 46845 USA

This is to certify that

Katahdin Analytical Services, LLC
600 Technology Way
Scarborough, ME 04074

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)

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

L2223
Certificate Number

ANAB Approval

Certificate Valid Through: 02/01/2022
Version No. 004 Issued: 01/16/2019

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)

Katahdin Analytical Services, LLC
600 Technology Way
Scarborough, ME 04074
Leslie Dimond
207-874-2400

TESTING

Valid to: **February 1, 2022** Certificate Number: **L2223**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2, 4`-DDD
GC/ECD	EPA 8081B	2, 4`-DDE
GC/ECD	EPA 8081B	2, 4`-DDT
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDD
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDE
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDT
GC/ECD	EPA 608; EPA 8081B	Aldrin
GC/ECD	EPA 608; EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane/cis-chlordane
GC/ECD	EPA 608; EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Cis-Nonaclor
GC/ECD	EPA 608; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 608; EPA 8081B	delta-BHC
GC/ECD	EPA 608; EPA 8081B	Dieldrin
GC/ECD	EPA 608; EPA 8081B	Endosulfan I
GC/ECD	EPA 608; EPA 8081B	Endosulfan II
GC/ECD	EPA 608; EPA 8081B	Endosulfan sulfate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 608; EPA 8081B	Endrin
GC/ECD	EPA 608; EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane/trans-Chlordane
GC/ECD	EPA 608; EPA 8081B	Heptachlor
GC/ECD	EPA 608; EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 608; EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	trans-Nonachlor
GC/ECD	EPA 608; EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	Dalapon
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
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D MOD	Diesel range organics (DRO)
GC/FID	EPA 8015C/D MOD	Total Petroleum Hydrocarbon (TPH)
GC/FID	EPA 8015C/D MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	CT ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1, 1-Trichloroethane
GC/MS	EPA 624; 8260B/C; EPA 524.2	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1, 2-Trichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1-Dichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1-Dichloroethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloropropene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromoethane (EDB)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichlorobenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3, 5-Trimethylbenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3-Dichloropropane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C; EPA 524.2	2, 2-Dichloropropane
GC/MS	EPA 8260B/C; EPA 524.2	2-Butanone
GC/MS	EPA 624; EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C; EPA 524.2	2-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	2-Hexanone
GC/MS	EPA 8260B/C; EPA 524.2	4-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C; EPA 524.2	Acetone
GC/MS	EPA 8260B/C	Acetonitrile
GC/MS	EPA 624; EPA 8260B/C	Acrolein
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Acrylonitrile

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	Allyl chloride
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C; EPA 524.2	Bromobenzene
GC/MS	EPA 8260B/C; EPA 524.2	Bromochloromethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Bromodichloromethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Bromoform
GC/MS	EPA 8260B/C; EPA 524.2	Carbon disulfide
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Carbon tetrachloride
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chlorobenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C; EPA 524.2	cis-1, 2-Dichloroethene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	Cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Dibromochloromethane
GC/MS	EPA 8260B/C; EPA 524.2	Dibromomethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Dichlorodifluoromethane
GC/MS	EPA 8260B/C; EPA 524.2	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C; EPA 524.2	Ethyl methacrylate
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C; EPA 524.2	Isopropyl benzene
GC/MS	EPA 8260B/C; EPA 524.2	m p-xylenes
GC/MS	EPA 8260B/C	Methyl acetate
GC/MS	EPA 8260B/C	Methacrylonitrile
GC/MS	EPA 624; EPA 8260B/C	Methyl bromide (Bromomethane)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C	Methyl methacrylate
GC/MS	EPA 8260B/C; EPA 524.2	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Methylene chloride
GC/MS	EPA 8260B/C; EPA 524.2	Naphthalene
GC/MS	EPA 8260B/C; EPA 524.2	n-Butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	n-Propylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	o-Xylene
GC/MS	EPA 8260B/C	Pentachloroethane
GC/MS	EPA 8260B/C; EPA 524.2	p-Isopropyltoluene
GC/MS	EPA 8260B/C; EPA 524.2	Propionitrile
GC/MS	EPA 8260B/C; EPA 524.2	sec-butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C; EPA 524.2	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B/C; EPA 524.2	Tetrahydrofuran

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Toluene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	trans-1, 2-Dichloroethylene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C; EPA 524.2	trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Vinyl chloride
GC/MS	EPA 624; EPA 8260B/C	Xylene
GC/MS	EPA 8260B/C SIM	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethene
GC/MS	EPA 8260B/C SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C SIM	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C SIM	1,2-Dibromoethane
GC/MS	EPA 8260B/C SIM	1,2-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,2-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,3-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,3-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,4-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	2-Hexanone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C SIM	Benzene
GC/MS	EPA 8260B/C SIM	Bromodichloromethane
GC/MS	EPA 8260B/C SIM	Carbon Disulfide
GC/MS	EPA 8260B/C SIM	Carbon Tetrachloride
GC/MS	EPA 8260B/C SIM	Chloroform
GC/MS	EPA 8260B/C SIM	Chloromethane
GC/MS	EPA 8260B/C SIM	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Dibromochloromethane
GC/MS	EPA 8260B/C SIM	Ethylbenzene
GC/MS	EPA 8260B/C SIM	Isopropylbenzene
GC/MS	EPA 8260B/C SIM	Hexachlorobutadiene
GC/MS	EPA 8260B/C SIM	Methylene chloride
GC/MS	EPA 8260B/C SIM	m,p-Xylene
GC/MS	EPA 8260B/C SIM	o-Xylene
GC/MS	EPA 8260B/C SIM	Tetrachloroethene
GC/MS	EPA 8260B/C SIM	Toluene
GC/MS	EPA 8260B/C SIM	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Trichloroethene
GC/MS	EPA 8260B/C SIM	Trichlorofluoromethane
GC/MS	EPA 8260B/C SIM	Vinyl Chloride
GC/MS	EPA 8260B/C SIM	Xylenes (total)
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625; EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dinitrophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dinitrotoluene (2, 4-DNT)
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 6-Dinitrotoluene (2, 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 625; EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 625; EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 625; EPA 8270C/D	2-Methyl-4 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 625; EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3-Methylcholanthrene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 625; EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 625; EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 625; EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	3, 4-Methylphenol
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 625; EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7, 12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 625; EPA 8270C/D	Acenaphthene
GC/MS	EPA 625; EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 625; EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 625; EPA 8270C/D	Benzidine
GC/MS	EPA 625; EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 625; EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 625; EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 625; EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 625; EPA 8270C/D	Benzo(k)fluoranthene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane)
GC/MS	EPA 625; EPA 8270C/D	bis(2-Ethylhexyl)adipate
GC/MS	EPA 625; EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625; EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 625; EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 625	Dibenzo(a,c)anthracene
GC/MS	EPA 625; EPA 8270C/D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyladipate
GC/MS	EPA 625; EPA 8270C/D	Diethyl phthalate
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 625; EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 625; EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 625; EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Ethyl methacrylate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 625; EPA 8270C/D	Fluoranthene
GC/MS	EPA 625; EPA 8270C/D	Fluorene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 625; EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 625; EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 625; EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methy methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 625; EPA 8270C/D	Naphthalene
GC/MS	EPA 625; EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	o,o-Diethyl o-2pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 625; EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 625	Perylene
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 625; EPA 8270C/D	Phenanthrene
GC/MS	EPA 625; EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 625; EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 625; EPA 8270C/D	3, 3’-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3’-Dimethylbenzidine
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A/B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A/B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4, 6 -Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene
HPLC/UV	EPA 8330A/B	3-Nitrotoluene
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	4-Amino-2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene
HPLC/UV	EPA 8330A/B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A/B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A/B	Tetryl
CVAA	EPA 245.1; EPA 7470A	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 200.7; EPA 6010C/D	Aluminum
ICP/AES	EPA 200.7; EPA 6010C/D	Antimony
ICP/AES	EPA 200.7; EPA 6010C/D	Arsenic
ICP/AES	EPA 200.7; EPA 6010C/D	Barium
ICP/AES	EPA 200.7; EPA 6010C/D	Beryllium
ICP/AES	EPA 200.7; EPA 6010C/D	Boron

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/AES	EPA 200.7; EPA 6010C/D	Cadmium
ICP/AES	EPA 200.7; EPA 6010C/D	Calcium
ICP/AES	EPA 200.7; EPA 6010C/D	Chromium
ICP/AES	EPA 200.7; EPA 6010C/D	Cobalt
ICP/AES	EPA 200.7; EPA 6010C/D	Copper
ICP/AES	EPA 200.7; EPA 6010C/D	Iron
ICP/AES	EPA 200.7; EPA 6010C/D	Lead
ICP/AES	EPA 200.7; EPA 6010C/D	Magnesium
ICP/AES	EPA 200.7; EPA 6010C/D	Manganese
ICP/AES	EPA 200.7; EPA 6010C/D	Molybdenum
ICP/AES	EPA 200.7; EPA 6010C/D	Nickel
ICP/AES	EPA 200.7; EPA 6010C/D	Potassium
ICP/AES	EPA 200.7; EPA 6010C/D	Selenium
ICP/AES	EPA 200.7; EPA 6010C/D	Silicon
ICP/AES	EPA 200.7; EPA 6010C/D	Silver
ICP/AES	EPA 200.7; EPA 6010C/D	Sodium
ICP/AES	EPA 6010C/D	Strontium
ICP/AES	EPA 200.7; EPA 6010C/D	Thallium
ICP/AES	EPA 200.7; EPA 6010C/D	Tin
ICP/AES	EPA 200.7; EPA 6010C/D	Titanium
ICP/AES	EPA 200.7; EPA 6010C/D	Vanadium
ICP/AES	EPA 200.7; EPA 6010C/D	Zinc
ICP/MS	EPA 200.8; EPA 6020A/B	Aluminum
ICP/MS	EPA 200.8; EPA 6020A/B	Antimony
ICP/MS	EPA 200.8; EPA 6020A/B	Arsenic
ICP/MS	EPA 200.8; EPA 6020A/B	Barium
ICP/MS	EPA 200.8; EPA 6020A/B	Beryllium
ICP/MS	EPA 200.8; EPA 6020A/B	Boron
ICP/MS	EPA 200.8; EPA 6020A/B	Cadmium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020A/B	Calcium
ICP/MS	EPA 200.8; EPA 6020A/B	Chromium
ICP/MS	EPA 200.8; EPA 6020A/B	Cobalt
ICP/MS	EPA 200.8; EPA 6020A/B	Copper
ICP/MS	EPA 200.8; EPA 6020A/B	Iron
ICP/MS	EPA 200.8; EPA 6020A/B	Lead
ICP/MS	EPA 200.8; EPA 6020A/B	Magnesium
ICP/MS	EPA 200.8; EPA 6020A/B	Manganese
ICP/MS	EPA 200.8; EPA 6020A/B	Molybdenum
ICP/MS	EPA 200.8; EPA 6020A/B	Nickel
ICP/MS	EPA 200.8; EPA 6020A/B	Potassium
ICP/MS	EPA 200.8; EPA 6020A/B	Selenium
ICP/MS	EPA 200.8; EPA 6020A/B	Silver
ICP/MS	EPA 200.8; EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Strontium
ICP/MS	EPA 200.8; EPA 6020A/B	Thallium
ICP/MS	EPA 200.8; EPA 6020A/B	Tin
ICP/MS	EPA 200.8; EPA 6020A/B	Titanium
ICP/MS	EPA 200.8; EPA 6020A/B	Tungsten
ICP/MS	EPA 200.8	Uranium
ICP/MS	EPA 200.8; EPA 6020A/B	Vanadium
ICP/MS	EPA 200.8; EPA 6020A/B	Zinc
IC	EPA 300.0; EPA 9056A	Chloride
IC	EPA 300.0; EPA 9056A	Fluoride
IC	EPA 300.0; EPA 9056A	Nitrate as N
IC	EPA 300.0; EPA 9056A	Nitrite as N
IC	EPA 300.0; EPA 9056A	Nitrate + Nitrite
IC	EPA 300.0; EPA 9056A	Orthophosphate as P
IC	EPA 300.0; EPA 9056A	Sulfate

Non-Potable Water		
Technology	**Method**	**Analyte**
IC	SOP CA-776	Lactic Acid
IC	SOP CA-776	Acetic Acid
IC	SOP CA-776	Propionic Acid
IC	SOP CA-776	Formic Acid
IC	SOP CA-776	Butyric Acid
IC	SOP CA-776	Pyruvic Acid
IC	SOP CA-776	i-Pentanoic Acid
IC	SOP CA-776	Pentanoic Acid
IC	SOP CA-776	i-Hexanoic Acid
IC	SOP CA-776	Hexanoic Acid
Titration	EPA 310.1; SM 2320B	Alkalinity
Caculation	SM 2340B	Hardness
Gravimetric	EPA 1664A; EPA 9070A	Oil and Grease, Oil and Grease with SGT
Gravimetric	SM 2540B/C/D	Solids
ISE	EPA 120.1; SM 2510B	Conductivity
ISE	SM 2520B	Practical Salinity
ISE	SM 4500F- C	Fluoride
ISE	SM 4500H+ B	pH
ISE	SM 5210B	TBOD / CBOD
Physical	EPA 1010A	Ignitability
Physical	EPA 9040C	pH
Titration	SM 2340C	Hardness
Titration	SM 4500SO_3 B	Sulfite
Titration	EPA 9034; SM 4500-S^{2-} F	Sulfide
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
IR	EPA 9060A; SM 5310B	Total organic carbon
Turbidimetric	EPA 180.1; SM 2130B	Turbidity
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 335.4; EPA 9012B; SM 4500-CN G	Amenable cyanide

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	EPA 350.1; SM 4500-NH3 H	Ammonia as N
UV/VIS	SM 3500Fe D	Ferrous Iron
UV/VIS	EPA 351.2	Kjeldahl nitrogen - total
UV/VIS	EPA 353.2; SM 4500-NO3 F	Nitrate + Nitrite
UV/VIS	EPA 353.2; SM 4500-NO3 F	Nitrate as N
UV/VIS	EPA 353.2; SM 4500-NO3 F	Nitrite as N
UV/VIS	EPA 365.2; SM 4500-P E	Orthophosphate as P
UV/VIS	EPA 365.4	Phosphorus total
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	EPA 410.4	COD
UV/VIS	EPA 420.1; EPA 9065	Total Phenolics
UV/VIS	SM 4500-Cl G	Total Residual Chlorine
UV/VIS	SM 5540C	MBAS
UV/VIS	EPA 7196A; SM 3500-Cr D	Chromium VI
UV/VIS	EPA 9012B; EPA 335.4	Total Cyanide
UV/VIS	EPA 9251; SM 4500-Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	EPA 3640A	Gel Permeation Clean-up
Cleanup Methods	EPA 3630C	Silica Gel
Cleanup Methods	EPA 3660B	Sulfur Clean-Up
Cleanup Methods	EPA 3665A	Sulfuric Acid Clean-Up
Organic Preparation	EPA 3510C	Separatory Funnel Extraction
Organic Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2,4`-DDD
GC/ECD	EPA 8081B	2,4`-DDE
GC/ECD	EPA 8081B	2,4`-DDT
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane/cis-chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	Cis-Nonachlor
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
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 (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane/trans-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Trans-Nonachlor
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 5', 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
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
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D MOD	Diesel range organics (DRO)
GC/FID	EPA 8015C/D MOD	Total Petroleum Hydrocarbons (TPH)
GC/FID	EPA 8015C/D MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	MA DEP EPH EPA 3546	Extractable Petroleum Hydrocarbons Microwave Extraction Preparation
GC/FID	CT-ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/C	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B/C	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C	1, 1-Dichloroethane
GC/MS	EPA 8260B/C	1, 1-Dichloroethylene
GC/MS	EPA 8260B/C	1, 1-Dichloropropene
GC/MS	EPA 8260B/C	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 2-Dibromoethane
GC/MS	EPA 8260B/C	1, 2-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 2-Dichloroethane
GC/MS	EPA 8260B/C	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 3-Dichloropropane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	2, 2-Dichloropropane
GC/MS	EPA 8260B/C	2-Butanone
GC/MS	EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C	2-Chlorotoluene
GC/MS	EPA 8260B/C	2-Hexanone
GC/MS	EPA 8260B/C	4-Chlorotoluene
GC/MS	EPA 8260B/C	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C	Acetone
GC/MS	EPA 8260B/C	Acetonitrile
GC/MS	EPA 8260B/C	Acrolein
GC/MS	EPA 8260B/C	Acrylonitrile
GC/MS	EPA 8260B/C	Allyl chloride
GC/MS	EPA 8260B/C	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C	Bromobenzene
GC/MS	EPA 8260B/C	Bromochloromethane
GC/MS	EPA 8260B/C	Bromodichloromethane
GC/MS	EPA 8260B/C	Bromoform
GC/MS	EPA 8260B/C	Carbon disulfide
GC/MS	EPA 8260B/C	Carbon tetrachloride
GC/MS	EPA 8260B/C	Chlorobenzene
GC/MS	EPA 8260B/C	Chloroethane
GC/MS	EPA 8260B/C	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B/C	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	cis-1,3-Dichloro-2-butene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 8260B/C	Dibromochloromethane
GC/MS	EPA 8260B/C	Dibromomethane
GC/MS	EPA 8260B/C	Dichlorodifluoromethane
GC/MS	EPA 8260B/C	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B/C	Ethyl methacrylate
GC/MS	EPA 8260B/C	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
GC/MS	EPA 8260B/C	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C	Isopropyl benzene
GC/MS	EPA 8260B/C	m p-xylenes
GC/MS	EPA 8260B/C	Methyl acetate
GC/MS	EPA 8260B/C	Methacrylonitrile
GC/MS	EPA 8260B/C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B/C	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C	Methyl methacrylate
GC/MS	EPA 8260B/C	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 8260B/C	Methylene chloride
GC/MS	EPA 8260B/C	Naphthalene
GC/MS	EPA 8260B/C	n-Butylbenzene
GC/MS	EPA 8260B/C	n-proplybenzene
GC/MS	EPA 8260B/C	o-Xylene
GC/MS	EPA 8260B/C	pentachloroethane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	p-Isopropyltoluene
GC/MS	EPA 8260B/C	Propionitrile
GC/MS	EPA 8260B/C	sec-butylbenzene
GC/MS	EPA 8260B/C	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 8260B/C	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B/C	Tetrahydrofuran
GC/MS	EPA 8260B/C	Toluene
GC/MS	EPA 8260B/C	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B/C	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C	Trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 8260B/C	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B/C	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate
GC/MS	EPA 8260B/C	Vinyl chloride
GC/MS	EPA 8260B/C	Xylene
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 8270C/D	2, 4-Dinitrophenol
GC/MS	EPA 8270C/D	2, 4-Dinitrotoluene (2 4-DNT)
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 8270C/D	2, 6-Dinitrotoluene (2 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methyl-4, 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3, 3`-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C/D	3,4-Methylphenol
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270C/D	Bis(2-Ethylhexyl)adipate
GC/MS	EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyl phthalate
GC/MS	EPA 8270C/D	Diethyladipate
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 8270C/D	Hexachlorobutadiene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methyl methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 8270C/D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O, O, O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	o,o-Diethyl o-2-pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 8270C/D	Pentachlorophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A	1 ,3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4, 6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene
HPLC/UV	EPA 8330A	3-Nitrotoluene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene
HPLC/UV	EPA 8330A	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A	Tetryl
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4, 6 –Dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6,Dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene
HPLC/UV	EPA 8330B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330B	Tetryl

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330B	Nitroguanidine
CVAA	EPA 7471B	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 6010C/D	Aluminum
ICP/AES	EPA 6010C/D	Antimony
ICP/AES	EPA 6010C/D	Arsenic
ICP/AES	EPA 6010C/D	Barium
ICP/AES	EPA 6010C/D	Beryllium
ICP/AES	EPA 6010C/D	Boron
ICP/AES	EPA 6010C/D	Cadmium
ICP/AES	EPA 6010C/D	Calcium
ICP/AES	EPA 6010C/D	Chromium
ICP/AES	EPA 6010C/D	Cobalt
ICP/AES	EPA 6010C/D	Copper
ICP/AES	EPA 6010C/D	Iron
ICP/AES	EPA 6010C/D	Lead
ICP/AES	EPA 6010C/D	Magnesium
ICP/AES	EPA 6010C/D	Manganese
ICP/AES	EPA 6010C/D	Molybdenum
ICP/AES	EPA 6010C/D	Nickel
ICP/AES	EPA 6010C/D	Potassium
ICP/AES	EPA 6010C/D	Selenium
ICP/AES	EPA 6010C/D	Silicon
ICP/AES	EPA 6010C/D	Silver
ICP/AES	EPA 6010C/D	Sodium
ICP/AES	EPA 6010C/D	Strontium
ICP/AES	EPA 6010C/D	Thallium
ICP/AES	EPA 6010C/D	Tin
ICP/AES	EPA 6010C/D	Titanium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010C/D	Vanadium
ICP/AES	EPA 6010C/D	Zinc
ICP/MS	EPA 6020A/B	Aluminum
ICP/MS	EPA 6020A/B	Antimony
ICP/MS	EPA 6020A/B	Arsenic
ICP/MS	EPA 6020A/B	Barium
ICP/MS	EPA 6020A/B	Beryllium
ICP/MS	EPA 6020A/B	Boron
ICP/MS	EPA 6020A/B	Cadmium
ICP/MS	EPA 6020A/B	Calcium
ICP/MS	EPA 6020A/B	Chromium
ICP/MS	EPA 6020A/B	Cobalt
ICP/MS	EPA 6020A/B	Copper
ICP/MS	EPA 6020A/B	Iron
ICP/MS	EPA 6020A/B	Lead
ICP/MS	EPA 6020A/B	Magnesium
ICP/MS	EPA 6020A/B	Manganese
ICP/MS	EPA 6020A/B	Molybdenum
ICP/MS	EPA 6020A/B	Nickel
ICP/MS	EPA 6020A/B	Potassium
ICP/MS	EPA 6020A/B	Selenium
ICP/MS	EPA 6020A/B	Silver
ICP/MS	EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Strontium
ICP/MS	EPA 6020A/B	Thallium
ICP/MS	EPA 6020A/B	Tin
ICP/MS	EPA 6020A/B	Titanium
ICP/MS	EPA 6020A/B	Tungsten
ICP/MS	EPA 6020A/B	Vanadium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A/B	Zinc
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Orthophosphate
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9071A/B	Oil and Grease, Oil and Grease with SGT
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
Titration	Walkley-Black	Total Organic Carbon
IR	Lloyd Kahn	Total organic carbon
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 350.1; SM 4500-NH3 H	Ammonia as N
UV/VIS	EPA 9251; SM 4500-Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	SM 3500-Fe D	Ferrous Iron
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196	Chromium VI
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Grain Size	ASTM D422	
Preparation	**Method**	**Type**
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	EPA 3660B	Sulfur Clean-up

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
Cleanup Methods	EPA 3620C	Florsil Clean-up
Cleanup Methods	EPA 3630C	Silica Gel Clean-up
Cleanup Methods	EPA 3640A	GPC Clean-up
Organic Preparation	EPA 3540C	Soxhlet Extraction
Organic Preparation	EPA 3545A	Pressurized Fluid Extraction
Organic Preparation	EPA 3546	Microwave Extraction Preparation for EPA 8082A, 8081B and 8270C, D, 8015C/D
Organic Preparation	EPA 3550C	Sonication
Inorganics Preparation	EPA 3050B	Hotblock
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Volatile Organics Preparation	EPA 5035/5035A	Closed System Purge and Trap

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	1, 1, 1-Trichloroethane
GC/MS	EPA TO-15	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA TO-15	1, 1, 2-Trichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethylene
GC/MS	EPA TO-15	1, 2, 4-Trichlorobenzene
GC/MS	EPA TO-15	1, 2, 4-Trimethylbenzene
GC/MS	EPA TO-15	1, 2-Dibromoethane (EDB)
GC/MS	EPA TO-15	1, 2-Dichlorobenzene
GC/MS	EPA TO-15	1, 2-Dichloroethane
GC/MS	EPA TO-15	1, 2-Dichloroethenes (Total)
GC/MS	EPA TO-15	1, 2-Dichloropropane
GC/MS	EPA TO-15	1, 3, 5-Trimethylbenzene
GC/MS	EPA TO-15	1, 3-Butadiene

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	1, 3-Dichlorobenzene
GC/MS	EPA TO-15	1, 4-Dichlorobenzene
GC/MS	EPA TO-15	1, 4-Dioxane
GC/MS	EPA TO-15	1,1,2-Trichloro1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA TO-15	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
GC/MS	EPA TO-15	1,4-Difluorobenzene
GC/MS	EPA TO-15	2,2,4-Trimethylpentane
GC/MS	EPA TO-15	2-Butanone
GC/MS	EPA TO-15	2-Chlorotoluene
GC/MS	EPA TO-15	2-Hexanone
GC/MS	EPA TO-15	2-Propanol
GC/MS	EPA TO-15	3-Chloropropene (Allyl chloride)
GC/MS	EPA TO-15	4-Ethyltoluene
GC/MS	EPA TO-15	4-Methyl-2-pentanone
GC/MS	EPA TO-15	Acetone
GC/MS	EPA TO-15	Acrolein
GC/MS	EPA TO-15	Benzene
GC/MS	EPA TO-15	Benzyl chloride
GC/MS	EPA TO-15	Bromochloromethane
GC/MS	EPA TO-15	Bromodichloromethane
GC/MS	EPA TO-15	Bromoform
GC/MS	EPA TO-15	Carbon disulfide
GC/MS	EPA TO-15	Carbon tetrachloride
GC/MS	EPA TO-15	Chlorobenzene
GC/MS	EPA TO-15	Chloroethane
GC/MS	EPA TO-15	Chloroform
GC/MS	EPA TO-15	Cis-1, 2-Dichloroethene
GC/MS	EPA TO-15	Cis-1, 3-Dichloropropene
GC/MS	EPA TO-15	Cyclohexane

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Dibromochloromethane
GC/MS	EPA TO-15	Dichlorodifluoromethane (Freon 12)
GC/MS	EPA TO-15	Ethanol
GC/MS	EPA TO-15	Ethyl acetate
GC/MS	EPA TO-15	Ethylbenzene
GC/MS	EPA TO-15	Hexachlorobutadiene
GC/MS	EPA TO-15	Isopropyl alcohol
GC/MS	EPA TO-15	Isopropylbenzene
GC/MS	EPA TO-15	m, p-Xylene
GC/MS	EPA TO-15	Methyl bromide (Bromomethane)
GC/MS	EPA TO-15	Methyl chloride (Chloromethane)
GC/MS	EPA TO-15	Methyl methacrylate
GC/MS	EPA TO-15	Methyl tert-butyl ether
GC/MS	EPA TO-15	Methylene chloride
GC/MS	EPA TO-15	Naphthalene
GC/MS	EPA TO-15	n-Heptane
GC/MS	EPA TO-15	n-Hexane
GC/MS	EPA TO-15	o-Xylene
GC/MS	EPA TO-15	Propene
GC/MS	EPA TO-15	Styrene
GC/MS	EPA TO-15	tert-Butyl alcohol
GC/MS	EPA TO-15	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA TO-15	Tetrahydrofuran
GC/MS	EPA TO-15	Toluene
GC/MS	EPA TO-15	trans-1, 2-Dichloroethylene
GC/MS	EPA TO-15	trans-1, 3-Dichloropropylene
GC/MS	EPA TO-15	Trichloroethene (Trichloroethylene)
GC/MS	EPA TO-15	Trichlorofluoromethane (Freon 11)
GC/MS	EPA TO-15	Vinyl acetate
GC/MS	EPA TO-15	Vinyl bromide

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Vinyl chloride
GC/MS	EPA TO-15	Xylenes (Total)
GC/MS	MA DEP APH	Aliphatic C5-C8 range
GC/MS	MA DEP APH	Aliphatic C9-C12 range
GC/MS	MA DEP APH	Aromatic C9-C10 range
GC/MS	MA DEP APH	1,3-Butadiene
GC/MS	MA DEP APH	Benzene
GC/MS	MA DEP APH	Ethylbenzene
GC/MS	MA DEP APH	m+p-Xylene
GC/MS	MA DEP APH	Methyl tert-butyl ether
GC/MS	MA DEP APH	Naphthalene
GC/MS	MA DEP APH	o-Xylene
GC/MS	MA DEP APH	Toluene

Biological Tissue		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane/cis-Chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Cis-Nonaclor
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin

Biological Tissue		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane/trans-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	trans-Nonachlor
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
ICP/AES	EPA 6010C/D	Aluminum
ICP/AES	EPA 6010C/D	Antimony

Biological Tissue		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010C/D	Arsenic
ICP/AES	EPA 6010C/D	Barium
ICP/AES	EPA 6010C/D	Beryllium
ICP/AES	EPA 6010C/D	Boron
ICP/AES	EPA 6010C/D	Cadmium
ICP/AES	EPA 6010C/D	Calcium
ICP/AES	EPA 6010C/D	Chromium
ICP/AES	EPA 6010C/D	Cobalt
ICP/AES	EPA 6010C/D	Copper
ICP/AES	EPA 6010C/D	Iron
ICP/AES	EPA 6010C/D	Lead
ICP/AES	EPA 6010C/D	Magnesium
ICP/AES	EPA 6010C/D	Manganese
ICP/AES	EPA 6010C/D	Molybdenum
ICP/AES	EPA 6010C/D	Nickel
ICP/AES	EPA 6010C/D	Potassium
ICP/AES	EPA 6010C/D	Selenium
ICP/AES	EPA 6010C/D	Silver
ICP/AES	EPA 6010C/D	Sodium
ICP/AES	EPA 6010C/D	Thallium
ICP/AES	EPA 6010C/D	Tin
ICP/AES	EPA 6010C/D	Vanadium
ICP/AES	EPA 6010C/D	Zinc
ICP/MS	EPA 6020A/B	Aluminum
ICP/MS	EPA 6020A/B	Antimony
ICP/MS	EPA 6020A/B	Arsenic
ICP/MS	EPA 6020A/B	Barium
ICP/MS	EPA 6020A/B	Beryllium
ICP/MS	EPA 6020A/B	Boron
ICP/MS	EPA 6020A/B	Cadmium

Biological Tissue		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A/B	Calcium
ICP/MS	EPA 6020A/B	Chromium
ICP/MS	EPA 6020A/B	Cobalt
ICP/MS	EPA 6020A/B	Copper
ICP/MS	EPA 6020A/B	Iron
ICP/MS	EPA 6020A/B	Lead
ICP/MS	EPA 6020A/B	Magnesium
ICP/MS	EPA 6020A/B	Manganese
ICP/MS	EPA 6020A/B	Molybdenum
ICP/MS	EPA 6020A/B	Nickel
ICP/MS	EPA 6020A/B	Potassium
ICP/MS	EPA 6020A/B	Selenium
ICP/MS	EPA 6020A/B	Silver
ICP/MS	EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Thallium
ICP/MS	EPA 6020A/B	Tin
ICP/MS	EPA 6020A/B	Vanadium
ICP/MS	EPA 6020A/B	Zinc
CVAA	EPA 7471B	Mercury

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2223.

Vice President

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

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ENVIRONMENTAL

Valid To: November 30, 2020 Certificate Number: 0001.01

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, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.1.1 of the DoD Quality Systems Manual for Environmental Laboratories, 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 Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Demands				
COD	----------	EPA 410.4	----------	----------
Total organic carbon	----------	EPA 9060A SM 5310C-2011	EPA 9060A SM 5310C-2011	EPA 9060A SM 5310 B-2011
Anions				
Ammonia	----------	EPA 350.1	EPA 350.1	SM 4500-NH3 B/C-2011
Fluoride	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Nitrate (as N)	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Nitrite (as N)	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Bromide	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Chloride	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Sulfate	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Wet Chemistry				
Alkalinity	----------	SM 2320B-2011	----------	----------
Corrosivity	----------	----------	SW-846 Chapter 7	SW-846 Chapter 7
Cyanide	----------	EPA 9012B	EPA 9012B	EPA 9012B
Filterable residue (TDS)	----------	SM 2540C-2011	----------	----------
Flashpoint	----------	----------	EPA1010A	EPA 1010A
Grain size	----------	----------	----------	ASTM D422
Hexavalent chromium digestion	----------	----------	----------	EPA 3060A
Hexavalent chromium	----------	EPA 7196A EPA 7199	EPA 7196A EPA 7199	EPA 7196A EPA 7199
Ignitability	----------	----------	40 CFR 261.21	40 CFR 261.21
Nitrate/Nitrite	----------	----------	EPA 353.2	----------
Non-filterable residue (TSS)	----------	SM 2540D-2011	----------	----------
pH	----------	SM 4500 H+B-2011 EPA 9040B EPA 9040C	EPA 9040B EPA 9040C	EPA 9045C EPA 9045D
Phenol	----------	EPA 9066	EPA 9066	----------
Reactivity	----------	----------	SW-846 Chapter 7.3	SW-846 Chapter 7.3
Sulfide	----------	EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011	----------	----------
Total residue	----------	SM 2540B-2011	----------	SM 2540G-2011
Metals				
Metals digestion	----------	EPA 3005A EPA 3010A EPA 3020A	EPA 3010A EPA 3020A	EPA 3050B
Aluminum	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Antimony	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Arsenic	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Barium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Beryllium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Boron	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Cadmium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Calcium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Chromium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Cobalt	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Copper	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Iron	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Lead	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Lithium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Molybdenum	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Magnesium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Manganese	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Mercury	----------	EPA 7470A	EPA 7470A	EPA 7471A EPA 7471B
Nickel	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Potassium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Selenium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Silicon	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Silver	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Sodium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Strontium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Sulfur	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Thallium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Thorium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Tin	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Titanium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Tungsten	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Uranium	----------	EPA 6020A EPA 6020B	EPA 6020A EPA 6020B	EPA 6020A EPA 6020B
Vanadium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Zinc	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Zirconium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Purgeable Organics (Volatiles)				
Volatile Preparation	----------	EPA 5030C	EPA 5030C	EPA 5035A
Acetone	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Acetonitrile	----------	EPA 8260C	EPA 8260C	EPA 8260C
Acrolein	----------	EPA 8260C	EPA 8260C	EPA 8260C
Acrylonitrile	----------	EPA 8260C	EPA 8260C	EPA 8260C
Allyl chloride	----------	EPA 8260C	EPA 8260C	EPA 8260C
Tert-amyl alcohol	----------	EPA 8260C	EPA 8260C	EPA 8260C
Tert-amyl methyl ether	----------	EPA 8260C	EPA 8260C	EPA 8260C
Tert-butyl alcohol	----------	EPA 8260C	EPA 8260C	EPA 8260C
Tert-butyl Formate	----------	EPA 8260C	EPA 8260C	EPA 8260C
Benzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Bromobenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Bromochloromethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Bromodichloromethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Bromoform	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Bromomethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
2-Butanone	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
n-Butylbenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
sec-Butylbenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
tert-Butylbenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Carbon disulfide	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Carbon tetrachloride	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
2-Chloro-1,3-butadiene	----------	EPA 8260C	EPA 8260C	EPA 8260C
Chloroacetonitrile	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Chlorobenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1-Chlorobutane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Chlorodifluoromethane	----------	EPA 8260C	EPA 8260C	EPA 8260C
Chloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
2-Chloroethyl Vinyl Ether	----------	EPA 8260C	EPA 8260C	EPA 8260C
Chloroform	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
1-Chlorohexane	----------	EPA 8260C	EPA 8260C	EPA 8260C
Chloromethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
2-Chlorotoluene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
4-Chlorotoluene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Cyclohexane	----------	EPA 8260C	EPA 8260C	EPA 8260C
Cyclohexanone	----------	EPA 8260C	EPA 8260C	EPA 8260C
Di-Isopropyl ether	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Dibromochloromethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Dibromomethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,2-Dibromoethane (EDB)	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,2-Dichlorobenzene	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,3-Dichlorobenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,4-Dichlorobenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
trans-1,4-dichloro-2-butene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Dichlorodi-fluoromethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,1-Dichloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,2-Dichloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,1-Dichloroethene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
cis-1,2-Dichloroethene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
trans-1,2-Dichloroethene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Dichlorofluoromethane	EPA 524.2	----------	----------	----------
1,2-Dichloropropane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,3-Dichloropropane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
2,2-Dichloropropane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,1-Dichloropropene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
cis-1,3-Dichloropropene	----------	EPA 8260C	EPA 8260C	EPA 8260C
trans-1,3-Dichloropropene	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,4-Dioxane	----------	EPA 8260C EPA 8260C SIM	EPA 8260C EPA 8260C SIM	EPA 8260C EPA 8260C SIM
Ethanol	----------	EPA 8260C	EPA 8260C	EPA 8260C
Ethylbenzene	----------	EPA 8260C	EPA 8260C	EPA 8260C
Ethyl ether	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Ethyl Methacrylate	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Ethyl Tert-Butyl Ether	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Freon-113	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Gasoline Range Organics (GRO) [Volatile Petroleum Hydrocarbons (VPH)]	----------	EPA 8015C EPA 8015D EPA 8260C NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C NW TPH-Gx MA VPH AK101
Heptane	----------	EPA 8260C	EPA 8260C	EPA 8260C
Hexane	----------	EPA 8260C	EPA 8260C	EPA 8260C
2-Hexanone	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Hexachlorobutadiene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Hexachloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Isobutyl Alcohol	----------	EPA 8260C	EPA 8260C	EPA 8260C
Isopropyl Alcohol	----------	EPA 8260C	EPA 8260C	EPA 8260C
Isopropylbenzene	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,4-Isopropyltoluene	----------	EPA 8260C	EPA 8260C	EPA 8260C
Methylacrylonitrile	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Methyl Acetate		EPA 8260C	EPA 8260C	EPA 8260C
Methyl Acrylate	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Methyl Iodide	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Methyl Ethyl ketone	----------	EPA 8260C	EPA 8260C	EPA 8260C
Methylene Chloride	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Methyl Isobutyl Ketone	----------	EPA 8260C	EPA 8260C	EPA 8260C
Methyl Methacrylate	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Methyl Tert-Butyl Ether	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
4-Methyl-2-pentanone	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Methylcyclohexane		EPA 8260C	EPA 8260C	EPA 8260C
2-Nitropropane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Naphthalene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Pentachloroethane	----------	EPA 8260C	EPA 8260C	EPA 8260C
Propionitrile	----------	EPA 8260C	EPA 8260C	EPA 8260C
n-Propylbenzene	----------	EPA 8260C	EPA 8260C	EPA 8260C
Styrene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Tert-Amyl Ethyl Ether		EPA 8260C	EPA 8260C	EPA 8260C
1,1,1,2-Tetrachloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Tetrachloroethene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Tetrahydrofuran	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Toluene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,2,3-Trichlorobenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,2,4-Trichlorobenzene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,1,1-Trichloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,1,2-Trichloroethane	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Trichloroethene	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Trichlorofluoromethane	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,2,3-Trichloropropane	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,2,4-Trimethylbenzene	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,3,5-Trimethylbenzene	----------	EPA 8260C	EPA 8260C	EPA 8260C
Vinyl Acetate	----------	EPA 8260C	EPA 8260C	EPA 8260C
Vinyl Chloride	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
Xylenes, Total	----------	EPA 8260C	EPA 8260C	EPA 8260C
1,2-Xylene (o-Xylene)	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C
1,3+1,4-Xylene (m+p Xylene)	EPA 524.2	EPA 8260C	EPA 8260C	EPA 8260C

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Extractable Organics (Semivolatiles)				
Organic Extraction	----------	EPA 3510C EPA 3511	EPA 3510C EPA 3511	EPA 3540C EPA 3546 EPA 3550C
Acenaphthene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Acenaphthylene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Acetophenone	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Acetylaminofluorene	----------	EPA 8270D	EPA 8270D	EPA 8270D
Alkylated PAHs	----------	EPA 8270D SIM	EPA 8270D SIM	EPA 8270D SIM
4-Aminobiphenyl	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Amino-4,6-dinitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
4-Amino-2,6-dinitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
Aniline	----------	EPA 8270D	EPA 8270D	EPA 8270D
Anthracene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Atrazine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Benzaldehyde	----------	EPA 8270D	EPA 8270D	EPA 8270D
Benzidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Benzoic acid	----------	EPA 8270D	EPA 8270D	EPA 8270D
Benzo (a) anthracene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Benzo (b) fluoranthene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Benzo (k) fluoranthene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Benzo (ghi) perylene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Benzo (a) pyrene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Benzo (e) pyrene	----------	EPA 8270D SIM	EPA 8270D SIM	EPA 8270D SIM
Benzyl Alcohol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Biphenyl	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
bis (2-Chloroethoxy) Methane	----------	EPA 8270D	EPA 8270D	EPA 8270D
bis (2-Chloroethoxy) Ether	----------	EPA 8270D	EPA 8270D	EPA 8270D
bis (2-Chloroethyl) Ether	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
bis (2-Chloroisopropyl) Ether	----------	EPA 8270D	EPA 8270D	EPA 8270D
bis (2-Ethylhexyl) Phthalate	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
4-Bromophenylphenyl Ether	----------	EPA 8270D	EPA 8270D	EPA 8270D
Butyl benzyl Phthalate	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Butyl Benzyl Phthalate	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D
Caprolactam	----------	EPA 8270D	EPA 8270D	EPA 8270D
Carbazole	----------	EPA 8270D	EPA 8270D	EPA 8270D
Carbon Range Organics C8-C44 (including subsets of this range i.e. HRO, MRO, ORO, RRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
4-Chloroaniline	----------	EPA 8270D	EPA 8270D	EPA 8270D
4-Chloro-3-methylphenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Chlorobenzilate	----------	EPA 8270D	EPA 8270D	EPA 8270D
1-Chloronaphthalene	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Chloronaphthalene	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Chlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
4-Chlorophenyl phenyl ether	----------	EPA 8270D	EPA 8270D	EPA 8270D
Chrysene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Cresols (Methyl phenols)	----------	EPA 8270D	EPA 8270D	EPA 8270D
cis-/trans-Diallate	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,4-Diamino-6-nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
2,6-Diamino-4-nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
Dibenzo (a,h) acridine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Dibenzo (a,h) anthracene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Dibenzofuran	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Dibenzothiophene	----------	EPA 8270D SIM	EPA 8270D SIM	EPA 8270D SIM
1,2-Dichlorobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,3-Dichlorobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,4-Dichlorobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
3,3-Dichlorobenzidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Diesel Range Organics (DRO) [Extractable Petroleum Hydrocarbons (EPH)]	----------	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103
2,4-Dichlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,6-Dichlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Diethyl Phthalate	----------	EPA 8270D	EPA 8270D	EPA 8270D
Dimethoate	----------	EPA 8270D	EPA 8270D	EPA 8270D
p-Dimethylaminoazobenze	----------	EPA 8270D	EPA 8270D	EPA 8270D
7,12-Dimethylbenz (a) anthracene	----------	EPA 8270D	EPA 8270D	EPA 8270D

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
alpha-, alpha-Dimethyphenethylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,4-Dimethylphenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Dimethyl Phthalate	----------	EPA 8270D	EPA 8270D	EPA 8270D
3,3'-Dimethylbenzidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Di-n-butyl Phthalate	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Di-n-octyl phthalate	----------	EPA 8270D	EPA 8270D	EPA 8270D
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B	EPA 8330B
1,2-Dinitrobenzene	----------	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B
1,3-Dinitrobenzene	----------	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B
1,4-Dinitrobenzene	----------	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B
2,4-Dinitrophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,4-Dinitrotoluene	----------	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B
2,6-Dinitrotoluene	----------	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B
1,4-Dioxane	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Diphenylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Diphenyl ether	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,2-Diphenylhydrazine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Ethyl Methane Sulfonate	----------	EPA 8270D	EPA 8270D	EPA 8270D
Fluoroanthene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Fluorene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Hexachlorobenzene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Hexachlorobutadiene	----------	EPA 8270D	EPA 8270D	EPA 8270D
Hexachlorocyclo-pentadiene	----------	EPA 8270D	EPA 8270D	EPA 8270D
Hexachloroethane	----------	EPA 8270D	EPA 8270D	EPA 8270D
Hexachloropropene	----------	EPA 8270D	EPA 8270D	EPA 8270D
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	----------	EPA 8330B	EPA 8330B	EPA 8330B
Indeno (1,2,3-cd) Pyrene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Isodrin	----------	EPA 8270D	EPA 8270D	EPA 8270D
Isophorone	----------	EPA 8270D	EPA 8270D	EPA 8270D
Isosafrole	----------	EPA 8270D	EPA 8270D	EPA 8270D
Methapyriline	----------	EPA 8270D	EPA 8270D	EPA 8270D
3-Methycholanthrene	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Methyl-4,6-dinitrophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Methyl methane sulfonate	----------	EPA 8270D	EPA 8270D	EPA 8270D
1-Methylnaphthalene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
2-Methylnaphthalene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
2-Methylphenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
4-Methylphenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Naphthalene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
1,4-Naphthoquinone	----------	EPA 8270D	EPA 8270D	EPA 8270D
1-Naphthylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Naphthylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
4-Nitroquinoline-1-oxide	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Nitroaniline	----------	EPA 8270D	EPA 8270D	EPA 8270D
3-Nitroaniline	----------	EPA 8270D	EPA 8270D	EPA 8270D
4-Nitroaniline	----------	EPA 8270D	EPA 8270D	EPA 8270D
Nitrobenzene	----------	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B	EPA 8270D EPA 8330B
Nitroglycerin	----------	EPA 8330B	EPA 8330B	EPA 8330B
2-Nitrophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
4-Nitrophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
3-Nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
4-Nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
5-Nitro-o-toluidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitroso-di-n-butylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosodiethylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosodimethylamine	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
n-Nitrosodimethylethylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosomorpholine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosodi-n-propylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosodiphenylamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosopiperidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
n-Nitrosopyrrolidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	----------	EPA 8330B	EPA 8330B	EPA 8330B
2,2-Oxybis (1-chloropropane)	----------	EPA 8270D	EPA 8270D	EPA 8270D
Pentachlorobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
Pentachloronitrobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
Pentachlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Pentaerythritol Tetranitrate (PETN)	----------	EPA 8330B	EPA 8330B	EPA 8330B
Perylene	----------	EPA 8270D SIM	EPA 8270D SIM	EPA 8270D SIM
Phenacetin	----------	EPA 8270D	EPA 8270D	EPA 8270D
Phenanthrene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Phenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,4-Phenylenediamine	----------	EPA 8270D	EPA 8270D	EPA 8270D
2-Picoline	----------	EPA 8270D	EPA 8270D	EPA 8270D
Pronamide	----------	EPA 8270D	EPA 8270D	EPA 8270D
Pyrene	----------	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM	EPA 8270D EPA 8270D SIM
Pyridine	----------	EPA 8270D	EPA 8270D	EPA 8270D
Safrole	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,2,4,5- Tetrachlorobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,3,4,6-Tetrachlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
Tetraethyl dithiopyrophosphate	----------	EPA 8270D	EPA 8270D	EPA 8270D
Tetraethy lead	----------	EPA 8270D	EPA 8270D	EPA 8270D
Tetramethyl lead	----------	EPA 8270D	EPA 8270D	----------
Tetryl	----------	EPA 8330B	EPA 8330B	EPA 8330B
Thionazin	----------	EPA 8270D	EPA 8270D	EPA 8270D
o-Toluidine	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,2,4-Trichlorobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,3,5-Trinitrobenzene	----------	EPA 8330B	EPA 8330B	EPA 8330B
2,4,5-Trichlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,4,6-Trichlorophenol	----------	EPA 8270D	EPA 8270D	EPA 8270D
O,O,O-Tri-ethylphosphorothioate	----------	EPA 8270D	EPA 8270D	EPA 8270D
1,3,5-Trinitrobenzene	----------	EPA 8270D	EPA 8270D	EPA 8270D
2,4,6-Trinitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
Organochlorine Pesticides				
Organic Extraction		EPA 3510C EPA 3511	EPA 3510C EPA 3511	EPA 3540C EPA 3546 EPA 3550C
Aldrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
alpha-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
beta-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
delta-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-BHC (Lindane)	----------	EPA 8081B	EPA 8081B	EPA 8081B
alpha-Chlordane	----------	EPA 8081B	EPA 8081B	EPA 8081B
Chlordane (Technical)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Chlorobenzilate	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDD	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDE	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDT	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDD	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDE	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDT	----------	EPA 8081B	EPA 8081B	EPA 8081B
Diallate	----------	EPA 8081B	EPA 8081B	EPA 8081B
1.2-Dibromo-3-chloropropane (DBCP)	----------	EPA 8081B	EPA 8081B	EPA 8081B

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Dieldrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Dinoseb	----------	EPA 8270D	EPA 8270D	EPA 8270D
Endosulfan I (alpha)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan II (beta)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan Sulfate	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Aldehyde	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Ketone	----------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-Chlordane	----------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor	----------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor Epoxide	----------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorobenzene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorocyclopentadiene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Isodrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Methoxychlor	----------	EPA 8081B	EPA 8081B	EPA 8081B
Mirex	----------	EPA 8081B	EPA 8081B	EPA 8081B
PCBs (Aroclors)				
PCB-1016 (Arochlor)	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1221	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1232	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1242	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1248	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1254	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1260	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1262	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1268	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB congeners (209)	----------	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C
Toxaphene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Organophosphorus Pesticides				
Azinphos-methyl (Guthion)	----------	EPA 8141B	EPA 8141B	EPA 8141B
Bolstar (Sulprofos)	----------	EPA 8141B	EPA 8141B	EPA 8141B
Carbophenothion (Trithion)	----------	EPA 8141B	EPA 8141B	EPA 8141B
Chlorpyrifos	----------	EPA 8141B	EPA 8141B	EPA 8141B
Coumaphos	----------	EPA 8141B	EPA 8141B	EPA 8141B
Demeton	----------	EPA 8141B	EPA 8141B	EPA 8141B
Diazinon	----------	EPA 8141B	EPA 8141B	EPA 8141B
Dichlorvos (DDVP)	----------	EPA 8141B	EPA 8141B	EPA 8141B
Disulfoton	----------	EPA 8141B	EPA 8141B	EPA 8141B
EPN	----------	EPA 8141B	EPA 8141B	EPA 8141B
Ethion	----------	EPA 8141B	EPA 8141B	EPA 8141B
Ethoprop	----------	EPA 8141B	EPA 8141B	EPA 8141B
Famphur	----------	EPA 8141B	EPA 8141B	EPA 8141B
Fenthion	----------	EPA 8141B	EPA 8141B	EPA 8141B
Malathion	----------	EPA 8141B	EPA 8141B	EPA 8141B

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Methyl Parathion	----------	EPA 8141B	EPA 8141B	EPA 8141B
Parathion	----------	EPA 8141B	EPA 8141B	EPA 8141B
Phorate	----------	EPA 8141B	EPA 8141B	EPA 8141B
Ronnel	----------	EPA 8141B	EPA 8141B	EPA 8141B
Stirphos	----------	EPA 8141B	EPA 8141B	EPA 8141B
Tokuthion	----------	EPA 8141B	EPA 8141B	EPA 8141B
Trichloronate	----------	EPA 8141B	EPA 8141B	EPA 8141B
Herbicides				
2,4,5-T	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4,5-TP (Silvex)	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-D	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-DB	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dalapon	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dicamba	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dichlorprop	----------	EPA 8151A	EPA 8151A	EPA 8151A
MCPA	----------	EPA 8151A	EPA 8151A	EPA 8151A
MCPP	----------	EPA 8151A	EPA 8151A	EPA 8151A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A	EPA 8151A
PCB Homologues				
Monochlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Dichlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Trichlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Tetrachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Pentachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Hexachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Heptachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Octachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Nonachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Decachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Dioxins/Furans				
2,3,7,8-TCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
2,3,7,8-TCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8-PeCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
2,3,4,7,8-PeCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8-PeCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,7,8-HxCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,6,7,8-HxCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
2,3,4,6,7,8-HxCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
1,2,3,7,8,9-HxCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,7,8,-HxCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,6,7,8-HxCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8,9-HxCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,6,7,8-HpCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,7,8,9-HpCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,6,7,8-HpCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
OCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
OCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HpCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HpCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HxCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HxCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total PeCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total PeCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total TCDD	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total TCDF	----------	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Misc. Headspace Analysis				
Carbon dioxide	----------	RSK-175	RSK-175	----------
Ethane	----------	RSK-175	RSK-175	----------
Ethene	----------	RSK-175	RSK-175	----------
Methane	----------	RSK-175	RSK-175	----------
Hazardous Waste Characteristics				
Toxicity Characteristic Leaching Procedure	----------	----------	EPA 1311	EPA 1311
Synthetic Precipitation Leaching Procedure	----------	----------	EPA 1312	EPA 1312
ASTM Leaching Procedure	----------	----------	ASTM D3987-85	ASTM D3987-85

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Other				
Perchlorate	----------	EPA 6850	EPA 6850	EPA 6850
Hydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Formaldehyde	----------	----------	EPA 8315A	EPA 8315A
Methylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	----------	EPA 5030A EPA 5030C	EPA 5030A EPA 5030C	EPA 5035 EPA 5035A
Organic Extraction	----------	EPA 3510C EPA 3511	EPA 3510C EPA 3511	EPA 3540C EPA 3546 EPA 3550C

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Per and Polyfluoroalkyl Substances (PFAS)			
N-ethyl Perfluorooctane-Sulfonamidoacetic Acid (NetFOSSA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
N-methyl Perfluoroctane-Sulfonamidoacetic Acid (NMeFOSAA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorobutanesulfonic Acid (PFBS)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorodecanoic Acid (PFDA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorododecanoic Acid (PFDoA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluoroheptanoic Acid (PFHpA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorohexanesulfonic Acid (PFHxS)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorohexanoic Acid (PFHxA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorononanoic Acid (PFNA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorooctanesulfonic Acid (PFOS)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Perfluorooctanoic Acid (PFOA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorotetradecanoic Acid (PFTeDa)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorotridecanoic Acid (PFTrDA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluoroundecanoic Acid (PFUnDA)	EPA 537	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluoro-n-butanoic Acid (PFBA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluoro-n-pentanoic Acid (PFPeA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
8:2 Fluorotelomersulfonate (8:2FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
4:2 Fluorotelomersulfonate (4:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluoropentanesulfonate (PFPeS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
6:2 Fluorotelomersulfonate (6:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluoroheptanesulfonate (PFHpS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorononanesulfonate (PFNS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorodecanesulfonate (PFDS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
10:2 Fluorotelomersulfonate (10:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorododecanesulfonate (PFDoDS)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorohexadecanoic Acid (PFHxDA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Perfluorooctadecanoic Acid (PFODA)	----------	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
Perfluorooctanesulfonamide (PFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
N-methylperfluoro-1-octanesulfonamide (NMePFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
N-ethylperfluoro-1-octanesulfonamide (NEtPFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
N-methylperfluoro-1-octanesulfonamidoacetic acid (NMeFOSAA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15
N-ethylperfluoro-1-octanesulfonamidoacetic acid Perfluoroundecanoic acid (NEtFOSAA)	----------	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15

End of DoD ELAP section of scope

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KATAHDIN ANALYTICAL

SERVICES, LLC

QUALITY ASSURANCE MANUAL

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Analytical Laboratory Quality Assurance Manual

TITLE: Katahdin Analytical Services, LLC. Quality Assurance Manual

Prepared By/Date: Leslie Dimond October 2018

Approved By Management/Date:

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Katahdin Analytical Services, LLC

QUALITY ASSURANCE MANUAL

Document Number QAM-001, Revision 12

September 2018

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1.0 INTRODUCTION

1.1 Purpose and Scope

This manual details the quality assurance program in effect at Katahdin Analytical Services, LLC. It is meant to be a teaching tool and source of information for laboratory personnel and clients. The manual describes the quality assurance policies and their implementation as they relate to the type, range and volume of environmental work performed at Katahdin. The Manual is divided into sections, each dealing with a different phase of laboratory operation, yet all sections overlap and function together to form a complete quality assurance program. The Manual is based on Good Laboratory Practices, technical knowledge, industry-accepted standard analytical practices and common sense. This manual meets the requirements of the current versions of the National Environmental Laboratory Accreditation Program (NELAP), the Department of Defense Quality Systems Manual (DoD QSM), including the requirements of ISO 17025:2005, the State of Maine certification program and other Federal and state specific requirements.

The Manual must be read and understood by all laboratory personnel as part of their training program. The Manual should also be referred to regularly as a source of information. A system of continuous updating is built into the Manual to allow it to change as laboratory conditions change or as new regulations are promulgated. This manual is a controlled document, which means that its identity, development, distribution, and status must be known and traceable at all times. All Katahdin permanent laboratory personnel are assigned a controlled temporary copy. Upon completion of reading, the employee must return the controlled copy to the Quality Assurance Officer and sign an attestation stating they have read and understood the QA manual. Temporary personnel are expected to read and understand the QA Manual.

Whenever a technician or analyst is in doubt as to proper procedures in a specific circumstance, the Manual should be consulted. Omissions or errors should be immediately reported to the Quality Assurance Officer for corrective action. **IT IS THE RESPONSIBILITY OF EACH LABORATORY WORKER TO ENSURE THAT THE PROVISIONS OF THIS MANUAL ARE FOLLOWED**. Disagreement with specific requirements or knowledge of changes causing deviation from the procedures should be discussed with the immediate supervisor before further work is completed. Laboratory personnel are encouraged to comment on the Manual and make recommendations for more efficient procedures.

The latest revision of each section of the Manual is the applicable rule. Therefore, revisions will be announced to all laboratory personnel. An uncontrolled copy of the Manual is offered to clients and regulatory agencies as the definitive quality assurance program used at Katahdin.

1.2 Quality Assurance Policy and Commitment

Katahdin is committed to quality as priority number one. Katahdin's quality assurance policy is based on the definition of quality as conformance to requirements governed by company policies, government regulations and standard operating procedures. This commitment recognizes the need for data to be representative of the environmental

conditions under investigation supported by measurements generated within a system that is designed to address applicable regulatory compliance criteria. Katahdin's Quality Assurance (QA) Manual and QA Program have been developed to fulfill this commitment. Our Quality Assurance Program contains provisions for establishing, maintaining and executing protocols to produce data of known quality and defensibility through the proper documentation of all measurement activities. No other concerns will be permitted to interfere with the execution of the elements of this QA Program and the quality of the data generated and delivered.

This manual describes the set of policies and principles, which guide day-to-day operations. Specific protocols are included by reference and are contained in a series of volumes cited in Section 8.0 of this document.

1.3 Management Commitment to Quality Assurance

Katahdin Management is committed to providing the highest quality analytical services and customer satisfaction available in the industry. To ensure this, Katahdin management is committed to:

- maintaining a quality system that supports the generation of data that are scientifically sound, legally defensible, meet client objectives, and are appropriate for their intended use,
- maintaining compliance with all applicable statutes, regulatory agencies and/or programs as described in the purpose and scope above,
- providing all employees with guidelines and an understanding of the ethical and quality standards required to work in the environmental testing industry,
- providing all employees with an understanding of the importance of meeting client requirements and providing superior service,
- improving the quality system through continuous feedback from staff and clients,
- maintaining a professional working environment that fosters open communication with both clients and staff,
- continued investment in new technologies, automation and quality improvements.

1.4 Quality Assurance Objectives

Katahdin Analytical Services, LLC is committed to the philosophy that quality operations result from quality planning, design, and work performance by skilled operational personnel. Katahdin's policy is to perform its varied types of technical work in accordance with standard quality assurance practices such as those put forth in the Good Laboratory Practices (GLP), the National Environmental Laboratory Accreditation Council (NELAC) standards, the Department of Defense Quality Systems Manual (DoD QSM), the International Organization for Standardization (ISO 17025:2005) manual,

other various EPA guidance documents, and Standard Methods. The Quality Assurance Officer is responsible for maintenance of standard operating procedures, laboratory audits, performance evaluations, federal and state certifications and quality assurance training documentation.

Each laboratory worker is responsible for reviewing and understanding standard operating procedures; adhering to these procedures during routine analyses; recording quality control information required by those procedures in the proper location, and taking appropriate corrective action including suspending analyses when quality control criteria are not met.

Objectives of the quality program are:

- to provide representative data of documented quality to clients and regulators
- to provide a quality organization independent of the pressures of project performance with the responsibility and authority for auditing and recommending corrective action;
- to provide a quality organization with clear paths of communication with management;
- to perform regularly scheduled audits and thereby document an objective evaluation of quality-related practices;
- to promptly identify variances and implement corrective actions and root-cause analysis;
- to maintain readily identifiable and retrievable records that provide documentary evidence of the quality of activities performed;
- to provide procedures for implementing project-specific quality plans;
- to define responsibility and authority for developing and implementing quality plans; and
- to provide quality reference documentation for each project.

Quality Assurance objectives for measurement data can be expressed in terms of completeness, representativeness, accuracy, precision**,** comparability and traceability.

1.4.1 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. The QA objective for completeness is to maximize the number of valid results. This can be attained by:

- selecting the appropriate methodology
- minimizing sample loss and breakage
- performing sufficient QC samples to document control
- documenting all aspects of the analytical system
- effectively understanding and communicating Data Quality Objectives (DQOs)
- adherence to designated holding times

The goal for completeness is dependent on project-specific DQOs and relies on planning and communication between the client and the lab. Realistic completeness goals are 80-95%.

The equation for completeness is:

$$\frac{\text{\# of data points obtained}}{\text{\# of data points expected}} \times 100 = \text{\% completeness}$$

1.4.2 Representativeness

Representativeness is the extent to which reported analytical results truly depict the chemistry of the sampled environment. Representativeness is a qualitative objective that is optimized through selection of appropriate sampling protocols, proper sample handling procedures, appropriate selection of holding times and analytical procedures, proper sample preservation, and prompt extraction and analysis.

Clients, Katahdin Sales and Marketing, Katahdin Project Managers and Katahdin Managers are responsible for selection of the appropriate sampling and analytical procedures.

Sample preservation follows USEPA guidance (40 CFR 136, Table II and SW-846) and Standard Methods unless dictated by other programs. Field preservation is checked upon sample receipt in the laboratory with the exception of volatiles that are checked immediately prior to analysis. Refer to the current revision of Katahdin Analytical Services SOP SD-902, Sample Receipt and Internal Control.

The Laboratory follows EPA guidance for sample holding times as stipulated in 40CFR 136, Table II and SW-846, Standard Methods or individual references. Refer to Table 6.1 for Katahdin Analytical Services sample container, preservation, and holding time requirements.

1.4.3 Accuracy and Precision

Accuracy and precision data are optimized through the use of analytical procedures that minimize biases through the use of standard procedures, traceable standards and QA lots, calibration of analytical equipment within

established acceptance limits and by implementing corrective action when measured accuracy and precision exceed pre-established limits.

Accuracy and precision are assessed through the analysis of several types of quality control (QC) samples. Laboratory generated QC samples, such as method blanks, laboratory control samples (LCSs), and sample duplicates, are used to assess the accuracy and precision of measurements due to laboratory activities. Surrogate spikes, matrix spike/matrix spike duplicates (MS/MSDs), and sample duplicates are used to monitor the effects of the sample matrix on precision and accuracy. Field blanks, field duplicates and trip blanks are used to assess the accuracy and precision of both sampling and laboratory activities. Accuracy and precision goals for the laboratory are based on laboratory historical data, specific method requirements and the requirements of each specific project. A more detailed discussion of these goals is provided in Section 11.0.

1.4.4 Comparability

Comparability is the extent to which comparisons among different measurements of the same quantity or quality will yield valid conclusions. Comparability is a qualitative objective that is attained by utilizing standard techniques for sample analysis and by reporting analytical data in appropriate units. Comparability between Katahdin's analytical results and those obtained by other environmental analytical laboratories will be ensured through the use of EPA, ASTM, and other recognized methods.

1.4.5 Traceability

Traceability is the extent to which results can be substantiated by hard copy and/or electronic documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis. Refer to Sections 6.0 and 10.0 for more specifics on Katahdin Analytical Services procedures.

1.5 Terms and Definitions

Acceptance: Criteria	Specific limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation:	The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accuracy:	The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.
Aliquot:	A measured portion of a sample taken for analysis.
Analyte:	The specific component or constituent that the analytical measurement seeks to determine.
Audit:	A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.
Batch:	A group of samples that are treated similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group [first] is greater than 20, then each group [successive] of 20 samples or less will all be handled as a separate batch.
Blank:	See Equipment Rinsate, Method Blank, Trip Blank, Field Blank, Calibration Blank.
Blind Sample:	A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst.
Calibration:	The process of establishing the relationship between instrumental response and known traceable quantities of analytes of interest.
Calibration Blank:	A quality control sample prepared in the same manner as calibration standards with the exception of the addition of the analytes of interest. A calibration blank is used to establish solvent/reagent and system contributions to the analytical result.
Calibration Verification:	The process of analyzing a mid-level calibration standard to verify the validity of the calibration curve.
Chain of Custody:	An unbroken trail of accountability that verifies the physical security of samples, data and records.

Comparability:	Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	Measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions
Continuing Calibration:	The process of analyzing standards periodically to verify the maintenance of calibration of the analytical system.
Control Chart:	A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.
Control Limit:	A range within which specified measurement results must fall to signify statistical control. A process is considered in control if data falls within the prescribed limits. A process is considered "out-of-control" if data falls outside the established control limits. These data are considered suspect and require corrective action including, but not limited to, qualification of the data.
Data Quality Objectives:	Qualitative and quantitative statements derived from the DQO Planning Process clarifying the purpose of the study, defining the most appropriate type of information to collect, determining the most appropriate conditions from which to collect that information, and specifying tolerable levels of potential decision errors.
Data Validation:	The internal process of review by which data are shown to be valid as evidenced by the soundness of the analytical system and successful meeting of the Data Quality Objectives (DQOs).
Dry Weight:	The weight of a sample based on percent solids. The weight after drying in an oven. Refer to Katahdin Analytical Services SOP, CA-717, Total Solids (% Moisture).
Duplicate Analysis:	A second measurement made on the same sample, sample extract or sample digestate to assist in the evaluation of precision of analysis.
Duplicate Sample:	See Field Duplicate, Matrix Duplicate, and Matrix Spike Duplicate.
Equipment Blank:	A field blank used to verify the effectiveness of equipment decontamination procedures. Laboratory deionized water is passed over sampling equipment after decontamination, collected, and analyzed by the lab.

Field Blank:	Samples of analyte-free media (generally water) taken from the laboratory to the field as: 1) distinct aliquots in the same containers used to collect samples with the appropriate preservative reagents added, or; 2) a single reserve to be aliquoted in the field into the appropriate containers with the appropriate preservatives for the parameters of interest. The intent of the field blank is to ascertain and document any contamination attributable to shipping, field handling procedures and potentially to ambient conditions.
Field Duplicate:	Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.
Field Sample:	A portion of material received by the laboratory to be analyzed, that is contained in single or multiple containers and identified by a unique field ID number.
Holding Time:	The elapsed time expressed in days (except for parameters requiring analysis in < 48 hours, which are expressed in hours) from the date of sample collection by the field personnel until the date of its processing/analysis. Holding time requirements are dictated by the EPA Federal Register 40CFR Part 136, Table II, or other appropriate method references, QAPPs, State regulation, or specific agency programs (i.e. AFCEE, NFESC, etc.).
Homogeneity:	The degree to which a property or substance is evenly distributed throughout a material.
Instrument Detection Limit:	The smallest signal above background noise that an instrument can detect at a 99% confidence level. The IDL does not consider any effects that the sample matrix, handling or preparation may have.
Initial Calibration:	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response, linearity and dynamic range of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a continuing calibration do not conform to the requirements of the method in use or at a frequency specified in the method.
Internal Standards:	Analytes, selected as non-targets or in a deuterated state, which are added to all standards and samples at the time of instrumental analysis. Internal standard quantitation takes into account the variability of the instrumental components at the discrete time of standard or sample analysis. The quantitation of the internal standard provides the evaluation of the influence upon the analysis and provides for the application to final sample data.

KIMS:	Katahdin Information Management System
Lab Control Sample:	A control sample whose matrix is of known composition or analyte-free matrix spiked with a known concentration of analytes of interest. Laboratory control samples are handled using the same preparation, reagents, and analytical methods employed for field samples. Laboratory Control Samples are utilized as indicators of the accuracy of the analysis.
Limit of Detection (LOD):	An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific. An LOD is the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). The false negative rate at the LOD is 1%.
Limit of Quantitation (LOQ):	The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence. The LOQ is the lowest concentration that produces a quantitative result within specified limits of precision and accuracy. The LOQ is set at or above the concentration of the lowest initial calibration standard.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix:	The component or substrate (e.g. surface water, drinking water) which contains the analyte of interest.
Matrix Duplicate:	An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.
Matrix Spike:	Aliquot of sample fortified (spiked) with known quantities of specified analytes and processed through the entire procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.
Matrix Spike Duplicate:	Intralaboratory split samples spiked with identical concentrations of target analyte (s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.
Method Blank:	An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document analyte contribution resulting from the analytical process. Acceptable levels of contamination are defined in individual SOPs and/or by project specific data quality objectives.

Method Detection Limit:	The statistically derived minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The false positive rate at the MDL/DL is 1%. Method detection limits are determined using replicate spike samples prepared by the lab and taken through all preparation and analysis steps of the method. The method detection limit is calculated using the appropriate Student's t parameter times the standard deviation of a series of spiked samples.
Performance Audit:	A process to evaluate the compliance of actual laboratory practices with relevant project requirements, regulations, contract specifications or internally stated standard operating procedures and practices.
Performance Evaluation:	A process to evaluate the proficiency of an analyst or laboratory by evaluation of the results obtained on known test materials.
Precision:	The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used estimates of precision are the relative standard deviation (RSD), when two or more samples are available and the relative percent difference (RPD), when only two samples are available.
Preservation:	Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
Protocol:	A stated plan that clearly defines the objectives, methods and procedures for accomplishing a task.
Practical Quantitation Limit:	Practical Quantitation Level is the laboratory's standard reporting limit for routine analytical work. The PQL is generally three to five times greater than the corresponding MDL or IDL and represents a level that the laboratory can routinely and reliably detect and quantitate in a variety of sample matrices. For many methods the PQL corresponds to the lowest level initial calibration standard.
Proficiency Test Sample:	A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.
QAPP:	A Quality Assurance Project Plan or QAPP is a project specific document that describes the policies, organization, objectives, functional activities, and specific QA and QC activities designed to achieve the data quality goals of a specific project.

Quality Assurance Program:	A system of policies and procedures whose purpose is to ensure, confirm and document that the product or service rendered fulfills the requirements of Katahdin and its client. Quality Assurance includes quality planning, quality control, quality assessment (auditing), quality reporting and corrective action.
Quality Control:	A system of checks and corrective measures, integrated with the activities that directly generates the product or service that serves to monitor and adjust the process to maintain conformance to predetermined requirements.
Quantitation Range:	The range of values in a calibration curve between the reporting limit (PQL/LOQ) and the highest successfully analyzed initial calibration standard.
Raw Data:	Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include instrument printouts, including dictated observations, and recorded data from automated instruments.
Reporting Limit:	The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, the dry weight of solids, and instrument dilution factors, unless otherwise specified.
Retention Time:	The time between sample injection and the appearance of a solute peak at the detector.
Rounding Rules:	If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. If the figure following those to be retained is 5, the figure 5 is dropped and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

Original #	Rounded To
11.443	11.44
11.446	11.45
11.435	11.44
11.425	11.42

Sensitivity:	Capability of methodology or instrumentation to discriminate between samples having different concentrations or containing differing amounts of an analyte.
Significant Figures:	The number of digits in a value that are justified by the accuracy and precision of the method being used. A value is made up of significant figures when it contains all digits known to be true and one last digit in doubt.
Spike:	A known mass of target analyte added to a blank sample or sub-sample that is used to determine recovery efficiency.
Split Sample:	Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision.
Standard:	A substance or material the properties of which are known with sufficient accuracy to permit its use to evaluate the same property in a sample.
Standard Operating Procedure:	A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps that outline expected limits of achievement and will produce consistent performance with repetitive use. This document must be officially approved as the method for performing certain routine or repetitive tasks.
Surrogates:	A compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. When employed, these compounds are added to every field and quality control sample prior to processing or preparation. Surrogate compounds are used to evaluate analytical efficiency by measuring recovery. Surrogates are generally utilized for organic analyses.
Systems Audit:	An on-site inspection or assessment of a laboratory's quality system or one of its components.
Traceability:	The ability to trace the source and accuracy of a material (i.e., standard) to a recognized primary reference source such as the National Institute of Standards and Technology (NIST) or USEPA. Also, the ability to independently reconstruct and review all aspects of the measurement system through available documentation.

Trip Blank:	A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.
Validation:	The internal process of review by which data are shown to be valid as evidenced by the soundness of the analytical system and successful meeting of the DQOs (not to be confused with data validation by an outside independent source).
Verified Time of Sample Receipt:	VTSR is the time that the laboratory accepts samples into its custody as documented by Sample Management signature and record of time of receipt on the chain-of-custody accompanying the samples. VTSR includes the inspection and resolution of any inconsistencies or issues regarding sample status, condition, or analytical request. VTSR begins at the time of final resolution.
Warning Limits:	The limits (typically 2 standard deviations either side of the mean) within which most analytical results are expected to lie with a 95% probability while the system remains in a state of statistical control.
Work Cell:	A well-defined group of analysts with specifically defined tasks that together perform the test method. The members of the work group and their specific functions must be fully documented.

TABLE 1-1: ACRONYMS

ACOE	Army Corps of Engineers
AFCEE	Air Force Center for Environmental Excellence
AOAC	Association of Analytical Communities
APHA	American Public Health Association
ASTM	American Society for Testing & Materials
BAM	Bacteriological Analytical Manual
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation & Liability Act
CLP	Contract Laboratory Program
COC	Chain of Custody
CRDL	Contract Required Detection Limit
DoD	Department of Defense
DQO	Data Quality Objectives
EHSO	Environmental Health & Safety Officer
EPA	Environmental Protection Agency
GC	Gas Chromatograph
GCMS	Gas Chromatograph Mass Spectrometer
GFAA	Graphite Furnace Atomic Absorption
GLP	Good Laboratory Practices
IC	Ion Chromatography
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma (Spectrophotometer)
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
KMS	Katahdin Information Management System
LCS(D)	Laboratory Control Sample (Duplicate)
LOD	Limit of Detection
LOQ	Limit of Quantitation
LRS	Linear Range Standard
MCP	Massachusetts Contingency Plan
MDL	Method Detection Limit
MIS	Management Information System
MS(D)	Matrix Spike (Duplicate)
MSDS	Material Safety Data Sheet
NBS	National Bureau of Standards
NELAP	National Environmental Laboratory Accreditation Program
NFESC	Naval Facilities Engineering Service Center
NJDEP	New Jersey Department of Environmental Protection
NPDES	National Pollutant Discharge Elimination System
PE	Performance Evaluation
PM	Project Manager
PQL	Practical Quantitation Limit
PT	Proficiency Test
QA	Quality Assurance
QAM	Quality Assurance Manual

TABLE 1-1: ACRONYMS cont'd

QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
RCRA	Resource Conservation & Recovery Act
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
S&HW	Solid & Hazardous Waste
SD	Sample Duplicate
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SRCR	Sample Receipt Condition Report
USGS	United States Geological Survey
VTSR	Verified Time of Sample Receipt
WP	Water Pollution
WS	Water Supply

2.0 QA ORGANIZATION, PERSONNEL, & TRAINING

Katahdin Analytical Services, LLC (tax ID # and Articles of Corporation) is a privately held, full service environmental testing firm. The company consists of one permanent laboratory facility located at 600 Technology Way in Scarborough, Maine. The company is comprised of analytical services, field sampling and measurement services, accounting, business development, financial management, human resources development, information systems, marketing and quality activities. A copy of all job classifications, including responsibilities and minimum requirements, is kept on file with the Human Resources Department and with the Quality Assurance Officer. The organizational structure of the company is provided in Figure 2-1.

It is important for efficient laboratory operation that all laboratory employees understand the operational structure, specific areas of responsibility and lines of authority within the organization. It is equally important for laboratory personnel to understand that the structures of the Quality Organization may be separate from other laboratory operations but that the quality function is totally integrated into every aspect of laboratory operation. All laboratory personnel are responsible for knowing and following proper methods and standard operating procedures; recording quality control information required by those procedures in the proper location; and suspending analyses when quality control criteria are not met.

2.1 Business Organization

The business is managed by the President. The Quality Assurance Officer reports directly to the President.

The President is directly responsible for the following functional groups:

- Quality Assurance
- Project Management
- Sales & Marketing Department
- Management Information Systems
- Reporting/Data Management Department
- Laboratory Operations Support Services
    - Sample Management Department
    - Waste Management Department
    - Field Services
    - Courier Services
- Laboratory Operations
    - Organic Department
    - Inorganic Department
    - Food/Microbiology Department

The inorganic department, headed by the Inorganic Department Manager consists of the Wet Chemistry and Metals Preparation and Analysis groups. The Wet Chemistry group is further managed by the Wet Chemistry Supervisor.

The organic department, headed by the Organic Department Manager, consists of the Organic Extractions (sample preparation), Extractables Analysis, and Volatiles Analysis groups. The Organic Extractions (sample preparation) and part of Extractables Analysis is further managed by the Extractions Supervisor.

Laboratory Operations Support Services is headed by the Sample Management Supervisor, consists of Sample Receipt, Bottle Preparation, Waste Disposal, Field Operations and Courier services. Field Operations is further headed by the Field Operations Supervisor.

Food/Microbiology is headed by the Food/Microbiology Supervisor.

Each department manager or supervisor is responsible for operations in their department on a daily basis. Environmental chemists, analysts, laboratory technicians and laboratory assistants report to the department managers and supervisors. Resumes of all key personnel employed at Katahdin Analytical Services, LLC are retained by the Human Resource Department.

2.2 Responsibilities

It is the individual responsibility of all employees to perform their assigned tasks according to this QA Manual, applicable SOPs, QA Project Plans, Study Protocols, Scopes of Work, Work Plans, and to specifically assigned job classifications. This includes responsibility for performing quality control analyses as specified in the method SOP and for entering the QC data in the appropriate logbook, electronic database, or method control file system. Failure to comply with the policies and/or requirements contained in this QA Manual or other Katahdin SOPs will result in mandatory further training and/or disciplinary action, up to, and including termination.

It is the responsibility of the Management Hiring Team, including the President, Department Managers, Quality Assurance Officer or other Supervisors/Technical Directors, to formulate the goals appropriate for each job task/title with respect to educational, technical, productivity and training requirements.

Educational, technical, and/or productivity requirements for each individual job title are filed with the Human Resources Department.

Additionally, it is the responsibility of the Management Hiring Team to ensure that only personnel with the appropriate educational and/or technical background are hired to perform testing and/or supportive functions at Katahdin. This responsibility includes ensuring that any specific regulatory (i.e. NELAC, DoD, etc.) educational or technical background requirements for certain positions are met.

All laboratory managerial and technical personnel will be given the authority and resources needed to carry out their duties. All laboratory managerial and technical personnel are responsible for identifying any occurrences of departures from the quality system or from procedures for performing environmental tests and for initiating actions to prevent or minimize such departures.

Department Managers and Group Supervisors shall assure that analysts and technicians are instructed in and are compliant with the requirements of the Laboratory QA Manual, QA Project Plans, SOPs, Protocols, and other regulatory criteria such as the NELAC standards and the DoD QSM. Department Managers and group Supervisors shall review sample QC data to assure that QC analyses are being performed at the required frequency, that data are documented in the appropriate logbook, electronic database, or

method control file system, and that established corrective action procedures for out-of-control situations are followed and the results documented. It is the responsibility of the Department Manager or group Supervisor to assure that data have been validated and reported to the Reporting/Data Management. Department Managers shall report to the President.

In the absence of the Department Manager or group supervisor, it shall be the responsibility of a designated senior analyst, other department manager, other qualified individual to carry on his/her duties.

The MIS manager is responsible for the management and quality control of all computing systems (hardware, software, documentation and procedures), generating, updating, and controlling quality of automated deliverables. The MIS Manager is also responsible for maintaining archives of electronic data including software. The MIS Manager is responsible for the operations and maintenance of all PC based computer systems and for integration with the LIMS and other data software. The MIS Manager is responsible for managing databases, developing macros and required programs as well as providing electronic data in various formats such as Access, ASCII, dBase, Excel, Monitor and GISKey. The MIS Manager shall report to the President.

In the absence of the MIS Manager, it shall be the responsibility of his/her designee, who may be a senior technical person, department manager, or other manager to carry on his/her duties.

The President is responsible for managing all activities related to laboratory services, including the Quality Assurance Program. The President shall assure that there is a Quality Assurance Department, that personnel and other resources are adequate, that personnel have been informed of their responsibilities, that deficiencies are reported to the responsible parties, that corrective actions are taken and documented and that the quality assurance program is effective in accomplishing the underlying goals. The President provides guidance and assistance in the development of laboratory quality control procedures, approves quality control limits for methods, works with Department Managers and group Supervisors to bring out-of-control methods back to within established acceptance limits, and assists Department Managers/Group Supervisors in correcting analytical problems revealed by QA audits.

The President shall work seamlessly with the Department Managers and Supervisors to promote and improve employee productivity, to address laboratory constraints, to improve turn-around time and to provide continuous improvement and automation in the lab.

Additionally, as the owner of Katahdin Analytical Services, the President shall ensure that all management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work. These policies are outlined in the company ethics agreement and training (refer to section 2.3 of this manual and SOP QA-811) and in the company Conflict of Interest Policy (refer to Figure 2-2).

In the absence of the President, it shall be the responsibility of his/her designee, who may be a senior technical person or department managers, to carry on his/her duties.

The Quality Assurance Officer (QAO) shall have experience in QA/QC procedures and be knowledgeable in the quality systems defined under NELAC and under the DoD QSM. The QAO shall have a general knowledge of the tests performed by the laboratory. The QAO shall assure that Department Managers and group Supervisors are instructed in and are compliant with the requirements of the Laboratory QA Manual, QA Project Plans, SOPs, Protocols, and other regulatory criteria such as the NELAC standards and the DoD QSM. The QAO shall be responsible for conducting systems audits and inspections for compliance with this manual, SOPs and QA Project Plans or other project-specific protocols. The QAO shall be responsible for maintaining historical files of all QA documents, reviewing QC charts, documenting findings and corrective actions, reviewing training records, managing PTs, maintaining conformance with certification requirements and reporting findings related to all of the above to management. The QAO shall ensure that communication takes place at all levels within the laboratory regarding the implementation and effectiveness of the QA Program. The QAO shall utilize available tools such as audits, PT results, corrective and preventive actions, customer feedback and management reviews to monitor and continuously improve the quality system. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems. The Quality Assurance Officer shall report directly to the President.

In the absence of the Quality Assurance Officer, it shall be the responsibility of his/her designee, who shall not be involved in the direct production of the work in the area of concern, to carry out his/her duties.

Project Managers (PMs) are responsible for managing sample projects within the laboratory. PMs are responsible for communicating on a daily basis with all external clients as well as with internal Department Managers and analysts. PMs are responsible for providing bids to clients, for reviewing bids compared to the lab's capabilities, for providing clients with bottles and necessities on a timely basis, for communicating with clients on a timely basis prior to work being received and throughout the project as needed. PMs must communicate any analytical issues to the client with professionalism and sufficient knowledge to resolve any issues with the client. PMs will act as the main contact for the assigned clients. Additional tasks may include sample log-in, bottle order preparation, working on QAPP tables and/or client visits.

In the absence of a Project Manager, it shall be the responsibility of his/her designee, who may be the Sales and Marketing Representative, a senior manager or qualified analyst , to carry on his/her duties.

Technical Directors shall be named on the title page of this QA Manual. Technical Managers shall include the Department Managers, Group Supervisors and senior analysts. All technical directors shall meet the educational and experience requirements dictated by NELAC and the DoD QSM for the laboratory areas that could fall under their responsibility for any amount of time.

2.3 Training and Orientation

The training program at Katahdin can be divided into four parts, Orientation Training, Technical Training, Quality Assurance Training, and Environmental Health & Safety Training. The Orientation Training is further divided into five parts, Human Resources Training, Basic Safety Training & Tour, An Introduction to Katahdin, Basic QA Training (an overview of the QA Program) and Ethics Training. The Ethics Training provides employees with information relating to their legal and ethical responsibilities. The Technical Training consists of the initial training for a method or process and the Initial Demonstration of Proficiency and any continuing or retraining that is deemed necessary by the individual Department Managers/Group Supervisors, or Quality Assurance Officer. The Quality Assurance Training provides more details on quality related to data review and reduction. Environmental Health & Safety Training provides an overview of the entire safety, chemical hygiene and hazardous waste programs at Katahdin.

On the first day of beginning employment, each new employee will receive a new employee orientation consisting of a Human Resources orientation, Ethics training, An Introduction to Katahdin, Basic QA training and a basic safety training & tour. Within the next few days of employment, each employee will begin their departmental technical training. Within a couple months each employee will have more advanced environmental health & safety training.

2.3.1 Human Resources Orientation

The Human Resources Director will conduct the human resources part of the orientation first. At this time the New Employee Human Resources Check List (Figure 2-3) will be used to document orientation and receipt of all human resources material. Each new employee will receive an Employee Handbook, which they will be asked to read over the next few weeks.

2.3.2 Ethics Training

Each employee will be asked to sign a Katahdin Code of Ethics, which will be maintained by the Human Resources Manager. Each new employee will be trained at the beginning of employment, and annually thereafter, on their ethical and legal responsibilities at Katahdin. This training is supported by Katahdin Analytical Services Code of Ethics (Figure 2-6). The training is conducted by the Quality Assurance Officer and will cover, but is not limited to, the following: (Refer to the current revision of Katahdin SOP, QA-811, Ethics and Data Integrity in the Laboratory, for further information.

- responsibilities of data handling
- responsibilities of documentation
- confidentiality
- responsibilities for reading, understanding, and adhering to all Katahdin documents
- compliance and regulatory framework

- valid data (true and representative of the measurement)
- data defensibility
- manual integration policy
- deviations from QA/QC criteria
- repercussions of unethical behavior

It is every employee's responsibility to read, understand and adhere to the Katahdin Code of Ethics. It is also a Katahdin policy to participate in an ethics training session. Attendance is mandatory and will be documented. Failure to comply with these policies and/or requirements associated with ethics training will result in mandatory further retraining and/or disciplinary action, up to, and including termination.

Training materials will be filed with the QA Officer.

This training will be documented as a sign-off provided during the training. This sign-off will be filed with the QA Officer.

2.3.3 Safety Tour

Safety training is an essential part of the orientation of each new employee. The training provides an overview of the safety considerations specific to the employee's lab area. On the first day of employment, each employee will be given a basic safety orientation/tour including information on the location and use of Personal Protective Equipment (PPE), emergency response equipment, MSDS's, the locations of general chemical hazards in the pertinent laboratory(ies), and information regarding the names of emergency and management personnel.

2.3.4 An Introduction to Katahdin

This training will provide each new employee with information on Katahdin's background, philosophy, and scope of work. It will incorporate keys to success, expectations of each employee and a brief summary of how training will be accomplished.

2.3.5 Basic Quality Assurance

When an employee begins employment, the QA Officer will assign each employee a controlled copy of the company QA Manual. As part of new employee orientation, the QA Officer will conduct the Quality Assurance orientation. QA Training will involve a summary of the QA Program contained within the QA Manual.

Each employee has the responsibility to read the Quality Assurance Manual and understand the systems and company policies it sets forth. An overview of the laboratory QA program will be discussed as well as an overview of the contents of

the QA manual. Each analyst shall have a clear concept of the quality objectives of Katahdin, the laboratory organization and paths of communication, standard practices and an understanding of their individual responsibilities.

Training will include discussions of the following:

Organization, Responsibilities & Training
Materials & Apparatus
Sample Handling and Chain-of-Custody
Holding Times & Preservation
Measurement & Calibration Techniques
Methodology & Regulations
Standard Operating Procedures
Reporting Limits – PQLs & MDLs
Record Keeping
Accuracy & Precision Measurements
Auditing & Proficiency Testing
Preventive Maintenance
Corrective Action

The training will focus on Katahdin's policies, the execution of these policies and the reasons for the policies. Analysts will be given sufficient time to ask questions about any aspect of the QA Program. In addition, the QA Officer remind analysts of the importance of documenting the training that they are receiving and will provide additional forms if necessary. Training materials will be filed with the QA Officer.

It is the responsibility of the Human Resources Department, the QA Officer, and the Environmental Health & Safety Officer to schedule, conduct, and document their respective orientations with the new employee.

2.3.6 Technical Training

Technical competency and proficiency means that an analyst has achieved an acceptable level of skills and understanding, sufficient to generate data of documented and acceptable precision and accuracy as defined in the applicable method. Training shall be conducted in a two-fold manner. First, the trainer demonstrates the procedure to the employee. Second, the employee conducts the procedure while the trainer observes and discusses the procedure with the employee. A Certification Statement (Figure 2-5) must be completed for each procedure in which the analyst is being trained. It is the intent of this training program to train each analyst to the level of competence required for the completion of the analyst's assigned tasks.

Department managers and group supervisors shall be responsible for providing documentation of training and proficiency in accordance with Katahdin Analytical Services SOP QA-805, for each employee under their supervision. The training documentation file indicates what procedures (SOPs) an analyst or technician is capable of performing either independently or with supervision. As part of the certification statement, employees must complete an Initial Demonstration of

Proficiency (IDP) that meets the requirements of the method. Please refer to Katahdin SOP QA-807, Method Performance / Precision & Accuracy Requirements, and individual methods for IDP requirements. This SOP gives specific details for this demonstration including all acceptable forms to meet this requirement. A summary of results and documentation of the applicable raw data files is maintained in the employee's training file. Each individual is responsible for maintaining an updated and current training documentation file.

SOP QA-805 further describes specific procedures for documenting training when an employee comes to Katahdin with prior training already or when an experienced employee "trains" themselves on a new procedure through method development.

Continued Demonstration of Proficiency (CDP) must be documented annually within each employee's training files. This may be accomplished by comparing the results of four LCSs to laboratory or method limits. CDP is also accomplished through the continuous implementation of Katahdin's QA program. QA/QC elements such as LCSs, blanks, matrix spikes, MDLs, performance evaluations, sample duplicates and surrogates are all functions of the QA Program. Evaluation of these elements on a daily or routine basis can reveal where additional training may need to occur.

It is Katahdin's policy that all preparation and analytical chemists complete their IDP and CDP independently of each other. Each individual must maintain their IDP and CDP in their training files. Even though a procedure may consist of same "work cell", or group of analysts working together to perform a method, each member of the "work cell", must document their own IDP and CDP.

2.3.8 Environmental Health & Safety Training

Environmental Health & Safety Training is performed in accordance with the current revision of Katahdin's Environmental Health & Safety Manual. The training will encompass the Laboratory Safety Plan, the Chemical Hygiene/Hazard Communication Plan, the Hazardous Matter Spill Prevention, Control, & Clean-Up (SPCC)/ Contingency & Emergency Response Plan, the Hazardous Waste Management Program, and HAZMAT Security Awareness Training.

Katahdin's training program is extensive and involves both initial and continuing training in many different areas of environmental health & safety. Katahdin is committed to meeting all Federal and State regulations regarding training. Refer to Figure 2-7 for a summary of the training and frequency of training.

The Katahdin Environmental Compliance Officer (ECO) or the Katahdin Environmental Health and Safety Officer (EHSO) will conduct the training associated with the Environmental health & Safety Manual. All employees who complete the training will have to sign an attendance sheet. A copy of the training materials and records of training will be kept by the ECO.

Figure 2-1

Katahdin Analytical Services, LLC
Organizational Chart

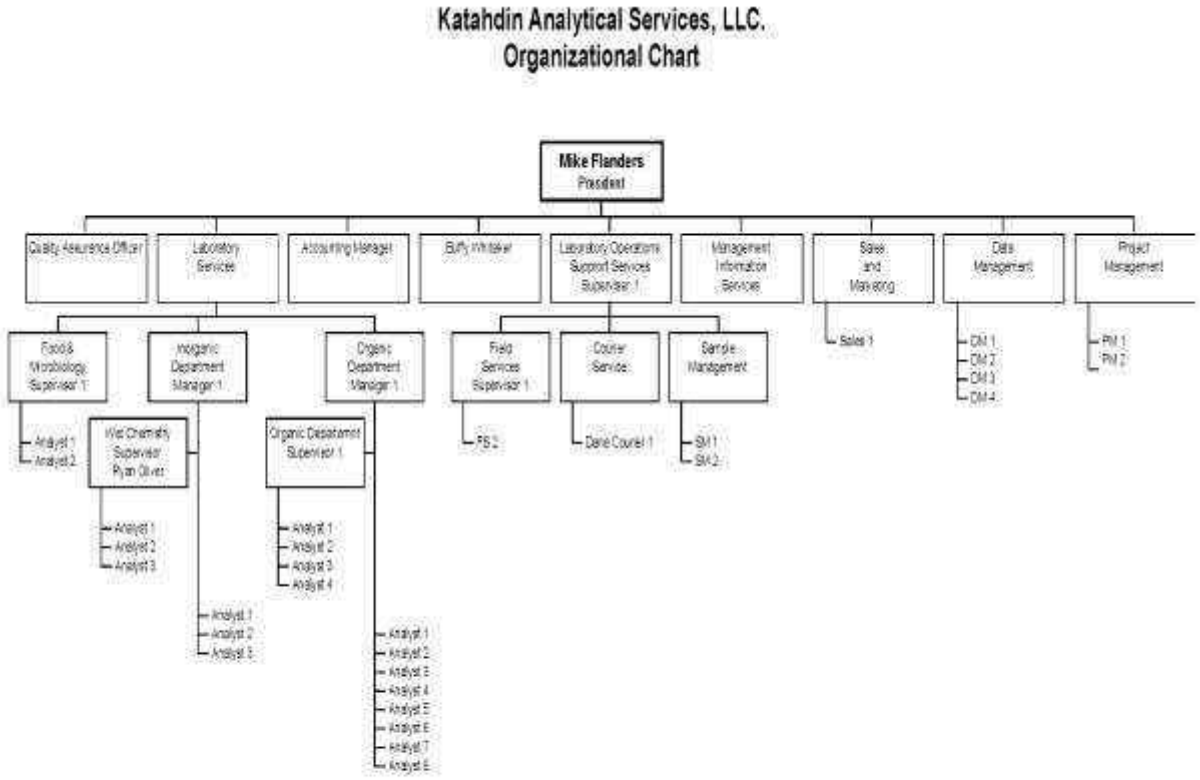


Figure 2-2

Katahdin Conflict of Interest Policy

CONFLICT OF INTEREST POLICY

Katahdin Analytical Services has several programs and policies in place in order to ensure that commercial or financial concerns of the laboratory operations do not affect or place undue pressures on employees that might adversely affect the quality of their work while employed by Katahdin. Specifically, the Quality Assurance Program is designed to establish, maintain and execute protocols to produce data of known quality and defensibility through the proper documentation of all measurement activities. The QA Manual states that, "No other concerns will be permitted to interfere with the execution of the elements of this QA Program and the quality of the data generated and delivered." Furthermore, the Katahdin Ethics Policy states that, "If a supervisor or a member of Katahdin management or any outside source requests an employee to engage in or perform an activity that they feel is compromising data validity or quality, the employee will not comply with the request and report this action immediately to a member of upper management." This last statement shall include any form of unethical behavior as defined in the current revision of Katahdin SOP, QA-811, Ethics in the Laboratory, or any other circumstance that may constitute a conflict of interest. A conflict of interest is a situation that arises when an individual, group of individuals, or organization is in, or could be in, circumstances that are or could be, questionable. Katahdin has the following policies concerning conflicts of interest.

No Katahdin employee shall accept gifts or gratuities valued at greater than $50 from customers or suppliers. All personnel are expected to act with integrity and good judgment and to recognize that the acceptance of personal gifts from those doing business or seeking to do business with Katahdin, even when lawful, may give rise to legitimate concerns about favoritism depending upon the circumstances.

All Katahdin employees shall disclose any personal, business, or financial relationship that he/she or any member of their household or immediate family has with a client or potential client of Katahdin Analytical Services for whom the lab is performing work which could be deemed as a conflict of interest.

AD-007 – Revision 1 – 03/23/2011

Figure 2-3

Katahdin New Employee Human Resources Checklist

NEW EMPLOYEE HUMAN RESOURCES CHECK LIST		
NAME:	EFFECTIVE DATE:	
ITEM	RECEIVED?	DATE COMPLETED
Status Change Form		
I-9		
Keycard Access Agreement		
Application		
Resume		
Health Insurance Forms – Medical & Dental		
Health Insurance Benefits Summary/Highlights		
Health Insurance Plan Book		
Create Employee File		
Enter information into Evolution		
Enter information into TimeTrak		
Create swipe card		
Set up PTO accrual		
W-4 Federal/State		
Direct Deposit		
Disability Forms		
New Hire Form		
Phone/Voice Mail Instructions		
Employee Handbook		
Employee Handbook Sign-off		
Drug-Free Workplace Sign-off		
Confidentiality Agreement Sign-off		
Ethics Agreement Sign-off		
Safety Glasses		

AD-006 – Revision 1 – 03/23/11

Figure 2-4

Safety Tour Training Form

Katahdin Analytical Services Safety Tour Training Form

Employee Name:	Date:
Employee's Work Area:	Administration or Laboratory
Safety Committee Member Performing Tour:	

Personal Protective Equipment (PPE)/Emergency Equipment & Information

Have safety glasses and a lab coat been issued?

Show location of the following and a description of how to use (if applicable). Circle each item shown and described:

Safety Shower	First Aid Kit	Fire Extinguisher	Spill Kit materials
Eyewash Station	Gloves	Goggles/Aprons	Main Shower/Spare Clothing
MSDS's (lab)	MSDS's (master)	Building exits (4)	Fire alarm pull boxes (4)
MSDS's (server_a/gendb/waste/KAS MSDS's)		Emergency Phone Numbers	

Other (specify):_____

Chemical Hazards

Show location of the following and a brief description of each (if applicable). Circle each item shown and described:

Acids Bases Solvents Flammables Poisons/Toxic Compressed Gases

Other(Specify):_____

Environmental Health & Safety Manual (EHSM) Reading

Show location of the following and a brief description of each (if applicable).

EHSM Bulletin Board – Appendix B-1 showing required/suggested PPE for each task and whether employee may work alone. Also note that it is important that employees let somebody know when they are leaving the building in case of emergency evacuations and so that another employee is not left alone unknowingly.

Each new employee is required to read sections B (Laboratory Safety Plan), C (Chemical Hygiene/Hazard Communication), and D (Hazardous Material Spill Prevention, Control & Clean-Up (SPCC)/Contingency & Emergency Response Plan of the EHSM.

Signature after reading:_____

Personnel Information

Katahdin Analytical Services Owner	Mike Flanders
Environmental Health & Safety Officer	Galen Nickerson
Emergency Coordinators	George Brewer (1st), Galen Nickerson (2nd), Mike Flanders (3rd)
Department Managers	George Brewer (inorganic), Pete Lemay (organic)

Employee Signature:_____ Date:_____

Trainer Signature:_____ Date:_____

HZ-010 – Revision 3 – 10/03/2016

Figure 2-5

Katahdin Training Certification Statement

Demonstration of Capability - Certification Statement

Date:	
Laboratory Name:	Katahdin Analytical Services, LLC 600 Technology Way Scarborough, Maine 04074
Analyst(s):	
Matrix (circle):	Aqueous Soil Other
Method # and SOP #	

We, the undersigned, CERTIFY that:

1. The analyst(s) identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program and other state and federal certifications, have met the Demonstration of Capability.
2. The test method(s) was performed by the analyst(s) identified on this certification.
3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.
4. The demonstration of capability has been performed in accordance with the current revision of Katahdin SOP, QA-807, *Method Performance/Precision & Accuracy Requirements*. Acceptance criteria are referenced in this SOP.
5. The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory(*).
6. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.
7. The analyst(s) identified above, have read, understood, and agreed to perform the applicable (approved method or SOP as specified by laboratory management) revision of the method and/or SOP.

Department Manager's or Trainer's Name	Signature	Date
Department Manager's or Trainer's Title		
Analyst's Name	Signature	Date
Quality Assurance Officer's Name	Signature	Date

* True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance data.
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

QA-005 – Revision 3 – 09/16/2016

Figure 2-6

Katahdin Code of Ethics

KATAHDIN ANALYTICAL SERVICES, INC.
CODE OF ETHICS

Our clients rely upon the integrity of our work. They may be homeowners who want to know whether their drinking water contains pollutants that may be harmful to their families. They may be government agencies or regulatory authorities that will rely upon our analytical test results as the basis for decisions that will affect public health and policy. In every case, it is vitally important that the information we provide to our clients be of a known and defensible quality.

As such, it is Katahdin's policy to incorporate the highest standard of data quality within all analytical programs. All Katahdin employees must remain committed to consistently demonstrating compliance with high quality. The integrity of our work depends upon the individual integrity of each employee. Accordingly, each employee is required to sign and adhere to the following policies.

I, _____(print name) understand that high standards of integrity are required of me with regard to the responsibilities that I have and to the data that I produce while employed at Katahdin. I agree that in the performance of my duties at Katahdin:

- I will adhere to all Standard Operating Procedures, including procedures described in the Katahdin Analytical Services' Quality Assurance Manual.
- I will not intentionally falsify data or results.
- I will not intentionally record or enter data or results that are not a true and accurate representation of the measured values or which are not validly quantitated.
- I will be accountable for my work.
- I will not intentionally misrepresent another individual's work.
- I will immediately inform my supervisor or a member of upper management if I am aware of misrepresentation of data.
- If a supervisor or a member of Katahdin management or any outside source requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of upper management.
- I agree to inform my supervisor or a member of management of any accidental reporting of non-authentic data by me or any other employee in a timely manner.

I have read and understand this Ethics Agreement. I understand that failure to comply with these policies and/or requirements associated with ethics training will result in mandatory retraining and/or disciplinary action, up to, and including termination.

Signature_____ Date_____

QA-079 – Revision 1 – 03/23/2011

3.0 QA DOCUMENTS & DOCUMENT CONTROL

All documents issued to personnel in the laboratory as part of the quality system are reviewed and approved by members of management (President, Department Manager, Group Supervisors, MIS Manager, and Quality Assurance Officer) before distribution. The distribution is controlled so as to ensure that all personnel follow the current and relevant versions of these quality documents.

3.1 QA Manual

This document describes management policies related to operation of the analytical laboratories. It provides overall guidance regarding acceptable practices and discusses each element of the Quality Assurance Program. It functions as the Project QA Manual where no other Quality Assurance Project Plan, Statement of Work or other contractually mandated project plan has been specified. Adherence to the practices described in this manual is required of all employees. This manual must be reviewed and updated, if changes are required, at least annually. If there are no changes, a No-Change form shall be filled out and filed. This manual may be revised and/or superseded only with the written authority of the Katahdin President. The QA Department administers distribution of controlled copies of this manual.

In order that this document achieves the goals outlined in Section 1.0, it is necessary that each Katahdin laboratory employee be familiar with the current provisions of this document. It is also necessary that this document represent agreement among Katahdin management and operational personnel as to the quality level desired and the means to that end.

Prior to its publication as a controlled document, this manual must be approved by the Katahdin Quality Assurance Officer, the Katahdin President and all Katahdin NELAC Technical Directors. To obtain such approval, the document proceeds through an iterative process of review and revision, involving the affected managers and their designated representatives. The signature page at the beginning of the manual represents acceptance.

Each time a revision is made to this manual, it must also be approved. The Quality Assurance Officer must approve each revision. If the revision constitutes a complete rewrite of the document, then review and approval by the Quality Assurance Officer, and the President becomes necessary. The Quality Assurance Officer will decide the appropriate approval process in each case.

The QA Manual is assigned the QA number of QAM-001 followed by a numerical revision number. Additionally, each section of the QA Manual is assigned a revision number. In this manner, the QA Manual may be updated in whole, or receive updates to only certain sections.

3.2 Standard Operating Procedures Manuals

All procedures related to sample collection, storage, preparation, analysis, disposal, data validation, data reporting and employee training and safety shall be contained in written Standard Operating Procedures (SOPs) Manuals. Each SOP shall contain the elements

outlined in the current revision of SOP QA-800, Preparation of SOPs. All sections shall be structured in a step-wise manner using numbered sections. All record-keeping requirements shall be described at each step in the SOP. Examples of forms used shall be included as tables or figures and referenced within the text. Preparation of SOPs shall be the responsibility of each department under the guidance of and review by the appropriate department manager. SOPs shall be assigned a number from the inventory list for SOPs maintained by the Quality Assurance Department. This number shall become part of the document control number when the SOP is accepted for implementation by Katahdin management. SOPs shall be reviewed and approved by the appropriate department manager, and the QA Officer, and submitted by the QA Department for approval prior to implementation. Refer to the current revision of Katahdin SOP, QA-800, Preparation of SOPs, and to section 12.0 of this manual for further information.

3.3 Project QA Plans and/or Quality System Manuals

Project QA Plans shall be implemented as required. These shall include such documents as Quality Assurance Project Plans (QAPPs). Additionally, the use of a specified Quality Systems Manual (QSM) may be required for some projects. For those projects that require specific QA/QC criteria the client provides a QAPP or specifies a QSM that has been approved by a regulatory agency (i.e. EPA or DoD). In either case, Katahdin works closely with the client and/or regulatory agencies to define the specific scope and Data Quality Objectives (DQOs) of each project. Only in this manner can the laboratory determine the specific criteria to follow to meet the needs of the client. Often, Katahdin works in conjunction with the client to write the analytical section of a QAPP. In this instance the QAPP is reviewed and approved by the Katahdin Quality Assurance Officer, Department Managers, Project Managers and/or Field Services Manager as appropriate. All project QAPPs received by the laboratory are submitted to the QA Department. Each QAPP is stored on the server for use by the analytical departments. Refer to the current revision of Katahdin SOP, QA-810, Project Management: Review and Communication of Client Bids, Contracts and Project Specific Information & Service to the Client.

3.4 Laboratory Logbooks

In order to ensure traceability and retrievability of records, all laboratory run logs, raw data logs, prep logs, standards prep logs, maintenance logs and any calibration logs are document controlled. A QA assigned number is stamped on every page at the center bottom. The QA number will resemble one of the following:

QAQCxxx for general QA logbooks
QAMSxxx for GC/MS logbooks
QAAAxxx for metals logbooks
QAWLxxx for wet chemistry logbooks
QAGCxxx for GC logbooks
QAEXxxx for extractions logbooks
QADMxxx for data management logbooks
QAFMxxx for food/microbiology logbooks
The assigned number is included at the bottom of each page of the logbook.

The Quality Assurance Officer keeps a Inventory of Laboratory Logbooks consisting of the logbook number, the lab area (e.g., GC/MS, Wet Chemistry, etc.), the logbook title, the date initiated, the name of the individual who requested the logbook and the serial number of the security zip cable.

Once the logbook is full and a new one is created, the appropriate department may maintain the logbook for several months to facilitate tracking of historical data. After this time, all completed and/or closed out logbooks will be relinquished to the QA department. Archived logbooks are retained for at least 5 years from date of closing.

3.5 Control of Other Printed Material

All laboratory designed forms, spreadsheets, and charts shall be controlled through an internal document control system to preclude the use of invalid and/or obsolete documents. The material covered in this system includes laboratory designed written material for QA, the analytical laboratory, support services and administration. All forms, charts, spreadsheets, etc. shall receive a control number with a revision number and date and an updated date, if applicable. The revision number shall apply to format changes in the structure of the form, chart, spreadsheet, etc. Forms will all begin with revision 1 effective with the start of this control system. The updated date shall apply to additional data being added to a form, chart, spreadsheet, etc., as with a dynamic form or inventory. The actual format does not change, only additional information is added.

Master electronic copies of these controlled documents are maintained by the Quality Control Officer. At this time, only the QAO has the authorization to make changes to these documents.

PDF copies of current forms are kept on a Katahdin server to be accessed by laboratory personnel.

Server_a\QAQC\QAQC\Current Forms

Outside written material applicable to the procedures and policies at Katahdin shall be controlled also. This material includes, but is not necessarily limited to, the DoD QSM, Accrediting Bodies accreditation documents, etc. For material that is provided already in manual form, an internal control number and effective date will be assigned to the cover and maintained by the responsible supervisor in a central location.

All controlled documents in this system must be reviewed and approved by the QAO, a Department Manager, and/or a Group Supervisor. All controlled documents must be revised as necessary to comply with the applicable methods, regulations or policies. All invalid or obsolete documents must be removed from the controlled folder on the company server and moved to the obsolete folder.

A master list of all controlled material shall be maintained by the QAO. The list shall include a title for the controlled document, the control number, the revision number and date. The list shall also indicate where copies of the controlled document have been distributed. Controlled documents shall not be copied and distributed elsewhere unless approved by the QAO.

The document control procedures described in this section are not applicable to software. Control of software is described in the current revision of Katahdin SOP SD-906, Software Quality Assurance.

3.6 Document Control, Distribution and Revision

Maintaining laboratory SOPs, the QA Manual and all other pertinent printed material so that they accurately reflect current practices is an ongoing effort at Katahdin, particularly in light of rapidly changing industry needs, analytical processes, and operating systems. Logbooks and benchsheets are used by Katahdin personnel to record raw data and must be used and preserved so as to maintain the integrity of that data. To ensure that methods and procedures are followed in a consistent manner, that confidential information is not distributed and that all current copies of QA documents are from the latest applicable revision, Katahdin has developed a document control system. All SOPs, QA Manuals, logbooks and other printed materials distributed within Katahdin are maintained under this document control system.

The document control system provides:

- A unique identification for each controlled document
- A central location for all controlled documents
- A systematic means for distribution of controlled documents
- A tracking system for controlled documents
- A means for identifying revisions
- A mechanism for consistent review of documents
- A mechanism for archival of outdated documents

Every SOP and the QA Manual must contain the colored Katahdin watermark identifier on each page. They must clearly indicate the date that the document became effective. Photocopies of SOPs or the QA Manual are not considered controlled documents. Photocopies are not allowed in the laboratory to prevent the use of outdated material.

All controlled copies of the QA Manual and SOPs will be distributed by the QA department, and an inventory of all distributed controlled copies will be maintained in the QA files. The QA department will also maintain a current index, including revision number and date, of all SOPs approved for use in the lab. Please refer to the current revision of Katahdin SOP QA-804, Document Control Procedures for detailed descriptions of the document control process.

4.0 FACILITIES

4.1 General Information

Katahdin Analytical Services currently employs a staff of approximately 40 environmental professionals and occupies a state of the art 14,500 square foot facility built in 2006, utilizing LEED-inspired (Leadership in Energy and Environmental Design) design and functionality. The facility utilizes a centralized office hub serving as the communications, data review, reporting, and operations point of origin. From this central hub, separate laboratories are arranged peripherally for Sample Receipt, Volatile Organics (GC and GC/MS), Extractable Organics (GC, GC/MS and HPLC), Wet Chemistry / Elemental Analysis, Elemental Preparation, Low-level Mercury Analysis, Microbiological Analyses / Food Testing, Air Analysis (GC/MS), and Grinding/Geotechnical Analysis. The floor plan offers hallways with access and entryways to all individual laboratory areas. Floor plans are provided in Figure 4-1.

The laboratory is designed to take into account environmental and accommodation conditions that could affect the results of a test. Each laboratory area is equipped with "reduced flow/velocity" hoods and separate HVAC systems. These hoods and all HVAC systems are integrated into a building direct computer control "DCC" system which allows for the building to be in monitored at any time. The system may be monitored off-site via "the web". Each laboratory functions on its own air system, efficiently managing the climate of the work environment and purifying the air to reduce and/or eliminate cross contamination between laboratories.

The Low-Level Mercury Analysis laboratory is designed as a clean room where special precautions have been taken to reduce dust and other contamination. The Microbiology/Food Testing laboratory has its own refrigerators to segregate food from environmental samples. Laboratory temperatures can be monitored and are recorded when required by the method. When such conditions are not met, or are out of criteria, the testing is redone with appropriate conditions, or the client is notified.

While the new facility is not a "LEEDS" certified building, many LEEDS specifications and materials are integral to the building. Lighting is provided by "high efficiency" florescent lights which are activated by motion sensors. To reduce air conditioning requirements, the roof is a "white" TPO (Thermoplastic Polyolefin) membrane. Some critical instrumentation such as GC/MS units and servers, including the data systems and Laboratory Information Management System (KIMS) are equipped with uninterruptable power supply systems (UPS) to minimize instrument downtime and damage during power surges or short duration power interruptions.

Additional features include walk-in refrigerators that can be locked when required and separate refrigerator storage for sample extracts, standards and volatile organics. An RO/DI water system is installed that delivers DI water to each individual laboratory room to increase efficiency and decrease the chance of cross-contaminated DI water. Added safety features include gas generators for some hydrogen, zero air and nitrogen, and a stainless steel pressure dispense system (SSPDS) that is plumbed to deliver methylene chloride to the extractables hoods.

4.2 Laboratory Safety

It is Katahdin Analytical Services' goal to maintain a safe and healthful work environment. Sample receiving areas and laboratories shall be equipped with suitable hoods, protective clothing and eye wear, gloves and/or other measures to prevent or minimize staff contact with hazardous substances. Safety equipment such as eyewash stations, drench showers, spill adsorbents and neutralizers, fire extinguishers, and first aid materials shall be available.

As a matter of policy, Katahdin Analytical Services shall not accept known initiator explosives, samples containing hydrofluoric acid, unusual biohazard materials or radioactive materials containing greater than background levels of radiation.

Environmental Health & Safety Training is performed in accordance with the current revision of Katahdin's Environmental Health & Safety Manual. The training will encompass the Laboratory Safety Plan, the Chemical Hygiene/Hazard Communication Plan, the Hazardous Matter Spill Prevention, Control, & Clean-Up (SPCC)/ Contingency & Emergency Response Plan, the Hazardous Waste Management Program, and HAZMAT Security Awareness Training. This document contains vital information related to training, safety equipment, personal considerations, chemicals, instrument / equipment operation, waste disposal, emergency situations and evacuation, glassware handling, housekeeping and first aid. Katahdin's training program is extensive and involves both initial and continuing training in many different areas of environmental health & safety. Katahdin is committed to meeting all Federal and State regulations regarding training. Refer to Figure 2-7 for a summary of the training and frequency of training.

The Katahdin Environmental Compliance Officer (ECO) will oversee the Environmental Health & Safety Program. The ECO shall designate a safety committee to help enforce and communicate the requirements of the Program. The committee shall consist of at least one staff member from each laboratory department. The Safety Committee shall meet on a quarterly basis to discuss any new safety or health related issues in the laboratory.

At the beginning of employment, each employee will be given a basic safety orientation/tour including information on the location and use of Personal Protective Equipment (PPE), emergency response equipment, MSDS's, the locations of general chemical hazards in the pertinent laboratory(ies), and information regarding the names of emergency and management personnel. Additionally, each employee will be given an Environmental Health & Safety Manual and be required to read sections B,C and D. Refer to Figure 2-4 for the Safety Tour Training Form. The safety orientation will be conducted by a member of the safety committee.

All employees will be trained on the EHS Manual and it's specific sections both initially and on as needed. The initial training should be conducted within 1-2 weeks of an employee's start date. The initial training will be extensive, but all employees will still be required to read and sign off on the manual. Refresher training will be conducted thereafter on an as needed basis. The ECO will conduct this extensive training.

Employees shall be responsible for their own safety. The Department Managers and Group Supervisors may require that certain levels of protective equipment be worn, when in their judgment, it is appropriate. Failure of an employee to wear required protective equipment will result in immediate disciplinary action.

4.3 Security

Two tiers of security shall be maintained within the Katahdin Analytical Services laboratory for the purpose of controlling external influences on samples, analytical processes, and data. This helps assure the completeness, representativeness, accuracy, and precision of analytical results.

4.3.1 Facility Security

The first tier of security maintained shall be controlled access to the laboratory building. Exterior doors to the laboratory building shall remain either locked or continuously monitored by a Katahdin Analytical Services staff member. Any keycard issued to an employee shall be relinquished to laboratory management at the time that employee terminates employment at Katahdin Analytical Services. All visitors to the facilities must sign the Visitors' Logbook maintained by the receptionist. All visitors shall be accompanied by a staff member during the duration of their stay on the premises. The staff member shall escort the visitor back to the reception area at the end of their visit where they shall sign out in the Visitor's Logbook. Refer to the current revision of Katahdin Analytical Services SOP AD-004, Laboratory Facility Security and Confidentiality for more information. At the close of each day, all doors shall be checked and locked by the last staff member leaving the building.

4.3.2 Data Security

The second tier of security may involve specific secure areas for sample, data and client report storage which shall be lockable within the facilities and to which access may be limited to specific individuals or their designees. No samples are to be removed without filling out the associated chain-of-custody records. Security of client report archives shall be the responsibility of the Quality Assurance Officer or the Human Resources Manager.

4.3.3 Electronic Security

As described in Section 2.0 of this manual and in the current revision of Katahdin SOP, QA-811, Ethics in the Laboratory, all employees are required to sign a Code of Ethics. This agreement requires the proper use and maintenance of computers and software at Katahdin. Katahdin utilizes dedicated computer work stations, which are used by only a group of cross-trained employees. This prevents the accidental "changing" of data. As described above, Katahdin maintains a secure facility, requiring all visitors to be escorted and monitored. Additionally, Katahdin's policy is for a software user to log-in under their unique log-in so that all software audit trails are accurate. Using a software program for which another user is already logged on is not acceptable. Log-in passwords for

the Laboratory Information System are changed on an annual basis as an added layer of security.

When test results are transmitted electronically, Katahdin will take steps to preserve confidentiality. Test results could be submitted via facsimile, website transmission, or via e-mail.

4.3.3.1 Facsimile Transmission

A statement is printed on the bottom of the fax cover page in the case of accidental incorrect transmission of results:

"The information contained in this facsimile transmission is privileged and confidential and intended for the use of the addressee named above. If the receiver of the following pages is not (one of) the above named recipient(s), you are hereby notified that any retention, dissemination, distribution, or copying of this facsimile is prohibited. If you received this facsimile in error, please notify us immediately by telephone. Thank you."

4.3.3.2 Website Transmission

Preliminary test results may be obtained on Katahdin's website using a Katahdin assigned unique client log-in and password. The site is secure, and clients may request several log-ins and/or passwords for different projects, etc.

4.3.3.3 E-mail Transmission

A statement, similar to the one below, is printed at the end of the e-mail message in the case of accidental incorrect transmission of results:

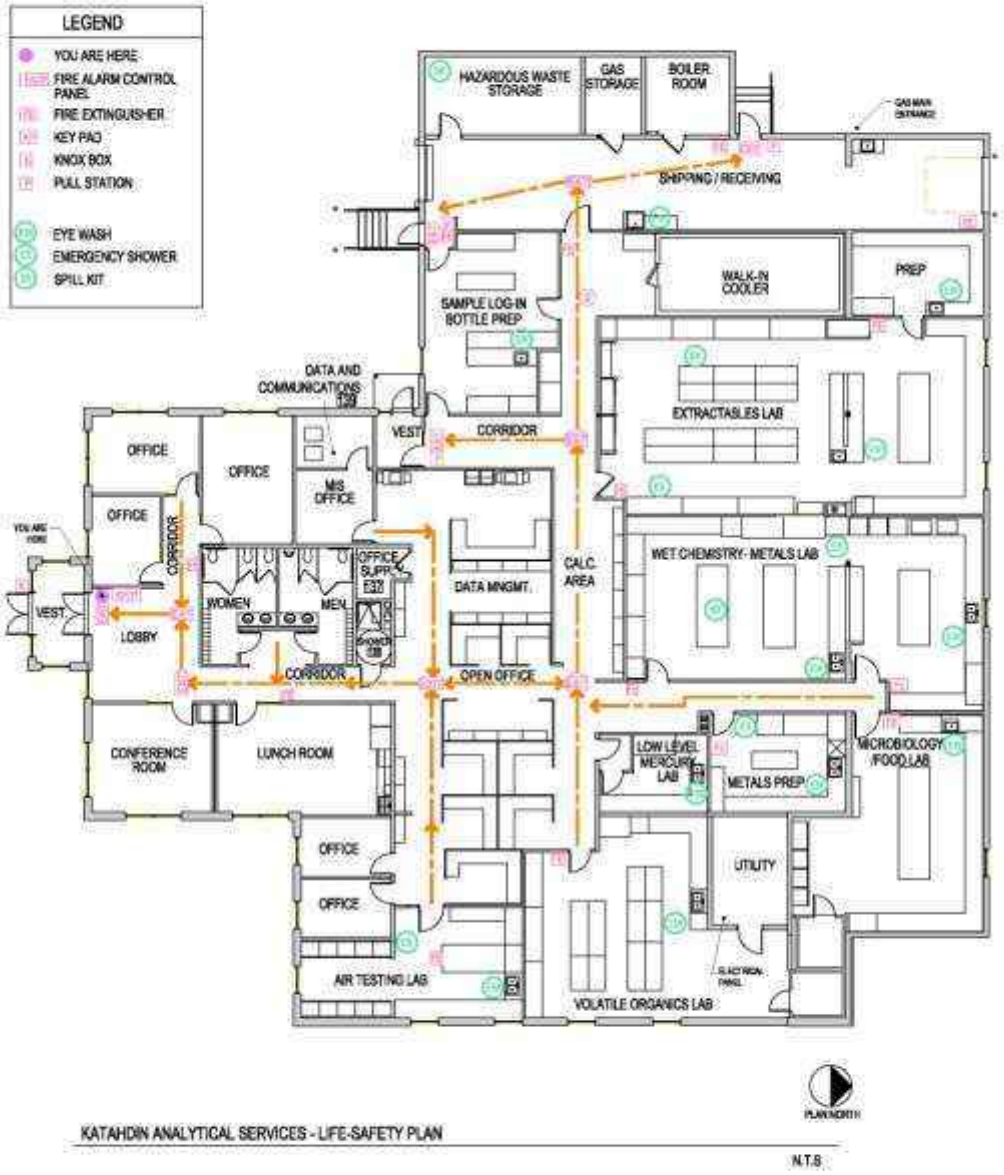
"Confidentiality Notice: This e-mail message, including any attachments, is for the sole use of the intended recipient(s) and may contain confidential and privileged information. If you are not the intended recipient please immediately contact the sender by reply e-mail and destroy/delete all copies of the original message. Any unauthorized review, use, copying, forwarding, disclosure, or distribution is prohibited."

4.4 Confidentiality

Standard business practices of confidentiality shall apply to all documents and information regarding client analyses. Specific protocols for handling confidential documents are described in the current revision of Katahdin Analytical Services SOP AD-004, Laboratory Facility Security and Confidentiality. Additional protocols for internal identification of samples and data by number only shall be implemented as required under contract-specific Quality Assurance Project Plans. No results from any testing are shared with anybody, except the client, without written consent from that client.

Figure 4-1

Katahdin Analytical Services, Incorporated
Facilities Floor Plan



5.0 MATERIALS AND EQUIPMENT

Evaluation and selection of suppliers and vendors is done primarily on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is accomplished through evaluation of supporting documentation provided by the vendor. This documentation may include certificates of analysis, recommendations and proof of historical compliance with similar programs for other clients.

To ensure that consumables and equipment conform to specified quality requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff. Reagents, solvents, gases, glassware, sample containers, standards, media and general supplies are ordered as needed to maintain sufficient quantities. Each department submits their own purchase orders to a member of management for approval. As these are approved, they are ordered by the purchasing department.

The Office Manager maintains an inventory of current approved vendors. Upon receipt of a purchase request for a “new” vendor, the Office Manager will set up an account with the new vendor and add them to the current record.

All consumables are received by the receiving personnel and distributed to the appropriate department. Each department will track the receipt of the different consumables as described below and in the specific Katahdin SOPs that are referenced below. Any problems with ordering including the receipt of broken materials, the receipt of incorrect materials or any other ordering issues shall be brought to the attention of the Purchasing department immediately.

The laboratory shall ensure that purchased supplies, reagents and consumables are not used until they have been inspected or otherwise verified. Katahdin Analytical Services strives to purchase high quality reagents and supplies from reputable sources. Solvent lots are checked for purity prior to use in laboratory. Method blanks are used to assess any laboratory, background or reagent contamination.

5.1 Reagents, Solvents, Media and Gases

Chemical reagents, solvents, gases, and media used in the laboratory are supplied by reputable chemical suppliers. All chemical reagents used for analyses shall be sufficient to meet the quality control requirements of the method, i.e. no contamination and no interferences. Individual methods may specify reagent requirements such as "Analytical Reagent Grade", “Ultra Pure” or “ACS (American Chemical Society) Grade”. When not specified by the method, “Analytical Reagent Grade” reagents shall be used.

Specific procedures for receiving reagents, solvents, media and gases are described in the current revision of Katahdin SOP CA-105, Reagent, Solvent and Media Receipt, Handling and Documentation.

All solvents, media and gases used shall be chosen to assure compliance with specific method and SOP requirements. Storage of solvents and chemical reagents shall comply with the current revision of Katahdin Analytical Service’s Environmental Health and Safety Manual.

5.2 Laboratory Equipment

All support equipment shall be maintained in proper working order. All support equipment shall be operated only in accordance with the applicable SOPs, or in the absence of an SOP, in accordance with the manufacturer's specifications. Documentation of any repairs, service calls, and corrective actions shall be maintained in the appropriate logbook. All support equipment shall be calibrated or verified at least annually using NIST traceable references when available over the entire range of use. Individual SOPs and/or logbooks contain specific calibration criteria suitable for the use of the equipment. All equipment not meeting the specified criteria must be marked as not usable or removed from service until corrective action brings the equipment back into compliance. All records of calibration must be maintained. Refer to Table 5-1 for a summary of support equipment requirements. This table also contains some information described in section 7.0 of this manual.

Note: If more stringent support equipment criteria are included in a mandated test method or by regulation, those criteria must be used. If it is not apparent which standard is more stringent, the requirements of the regulation or mandated test method must be followed.

5.2.1 Refrigerator/Freezer Temperature Logs

Refrigerators and freezers are checked every weekday to ensure that they are operating properly and within established temperature ranges. All information is recorded in bound logbooks. Additionally, refrigerators used for sample and extract storage (thermal preservation) are checked on the weekends. These temperatures are recorded by wireless data loggers and transferred to a computer program held on the lab server. This data is monitored by the Quality Assurance Officer and the President.

Routine maintenance such as defrosting is performed as needed. Refer to the current revision of Katahdin Analytical Services SOP QA-803, Laboratory QA: Self-Inspection System. Responsibility for performing the checks is assigned within the laboratory section where the units are located. The QA Department is responsible for ascertaining that checks have been performed and that necessary corrective actions have been instituted when needed. The QA Department is responsible for archiving all historical temperature logsheets and/or logbooks.

5.2.2 Adjustable Pipettors

Adjustable pipettes are calibrated each day of use at the maximum volume and monthly at the minimum, mid and maxiumum volumes. All calibrations must be performed on a NIST calibrated balance. When adjustable pipettors are outside of the tolerances expressed in the current revision of SOP CA-103, Calibration of Adjustable Pipettors, corrective actions are taken to bring the pipette into conformance or remove it from service.

5.2.3 Balances, Ovens, and Incubators

Refer to section 7.2 of this manual for calibration criteria.

5.2.4 Microbioloy Support Equipment

Specific checks are required for incubators, water baths, autoclaves, UV equipment and glassware used for microbiological testing. Refer to Figure 5-1 for a summary of these requirements.

5.3 Solvent Lot Checks

Some of the more commonly used solvents undergo a solvent lot check to identify any trace contamination. These solvents include methylene chloride, hexane and acetone. When a new lot is opened, the chromatogram for a concentrated volume of the new solvent lot is checked for any contamination. All information relevant to the check is recorded and maintained in a solvent check file. Responsibility for performing the checks and maintaining the records is assigned to the extractions personnel.

5.4 Glassware

All glassware used in the laboratory must be maintained in good condition, cleaned, properly stored, and separated according to its specific laboratory application. Cracked, excessively chipped or otherwise defective glassware is either discarded or repaired. Katahdin Analytical Services purchases all glassware from recognized commercial laboratory glassware suppliers such as Fisher Scientific and Baxter. All volumetric glassware utilized shall be sufficient to meet the quality control requirements of the method, i.e. to meet the precision and accuracy of the method. Class "A" certified glassware should be used whenever possible.

Refer to Table 5-1 for general guidance on volumetric and non-volumetric glassware. Also refer to the current revision of Katahdin SOP CA-108, Basic Laboratory Techniques, for further detail on lab glassware.

Each laboratory maintains its own set of glassware, completely independent from the other laboratories. Glassware is segregated for cleaning within each preparation laboratory to ensure that the glassware remains dedicated for use by specific laboratories.

5.5 Glassware Cleaning

Laboratory glassware is cleaned in accordance with its intended use at the end of the analytical process. Different cleaning procedures exist for different types of analyses and glassware. Refer to the current revision of Katahdin Analytical Services SOP CA-100, Lab Ware Cleaning.

5.6 Sample Containers

Please refer to Section 6.2.1 of this manual for Katahdin’s standard practices concerning sample containers.

5.7 Instruments

Laboratory instrumentation used shall be as specified in the protocol for the analytical method. Table 5-2 lists the major analytical instrumentation currently in use in by Katahdin Analytical Services. All laboratory instrumentation shall be operated only in accordance with the applicable SOPs, or in the absence of an SOP, in accordance with the manufacturer's specifications. All laboratory instrumentation and associated hardware and software must be operated by trained employees only to safeguard instruments from accidental changes that would invalidate data.

Preventive maintenance is performed for each instrument by manufacturers, analysts and field service technicians on an ongoing basis and the activities documented in a bound instrument maintenance logbook or in the instrument runlogs as described in the current revision of Katahdin Analytical Services SOP CA-101, Equipment Maintenance, and section 14 of this manual.

Corrective maintenance shall be provided as required for all instruments and equipment and documented in appropriate logbooks. Factory replacement parts, trained service technicians and first quality materials shall be used whenever necessary. It is Katahdin Analytical Services' policy to conduct repairs at the lowest level of complexity necessary and to obtain parts directly from primary manufacturers where critical. The purpose of this policy is to maintain efficiency, economy and reliability of quality maintenance.

5.8 Stock Standards

Analytical standards are prepared from pure compounds or are purchased prepared from reputable vendors. These standards provide the stock used to prepare serial dilutions for calibration and spiking standards. Katahdin Analytical Services strives to purchase only the highest quality materials. To that end, reference standards must be traceable to national standards of measurement (e.g. NIST) whenever possible. Standards used for calibration must be traceable, when possible, to national standards of measurement, either directly through supplier documentation or by verification against a second source traceable reference standard. Supplier documentation is filed within each analytical section for ease in traceability. If assayed materials are unavailable, the material of highest purity available shall be obtained.

Each laboratory section is responsible for the preparation, storage and disposal of its standards. Pertinent standards preparation information is recorded into laboratory specific standards logbooks in order to document traceability of prepared standards to their source material(s). Lot information is kept within each department. Manufacturers may keep purity records on file at their facilities. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability.

5.9 Reagent Water

The performance of trace level analytical work requires that the water used for sample preparation, preparation of reagent solutions, and final rinsing of glassware be "theoretically pure," i.e. free from interferences, electrolytes, and other contaminants. In all cases, unless specified by the analytical SOP, reagent grade water is used. The electrical resisitivity of all water used for analytical work is monitored daily to meet the

requirement of 16.7 Megohm-cm (maximum conductivity of approximately 0.06 umho/cm). Refer to the current revision of Katahdin SOP CA-104, Use of Laboratory Water Systems, for further details.

5.10 Holding Blanks

Holding blanks are prepared bimonthly by filling a 40 mL VOA vial with prepurged laboratory reagent grade water. These are then acidified to a pH less than 2 with HCL. The holding blanks are logged in and given a work order number. The holding time is 14 days. Two vials are placed in each of the refrigerators used to store Volatile samples. Two vials are also placed in the freezer used to store DI preserved Volatile samples. The holding Blanks are then analyzed and evaluated to track any consistent contamination above the laboratory PQL. Contamination is deemed consistent if detected in holding blanks analyzed over a 4-week interval.

TABLE 5-1

SUPPORT EQUIPMENT REQUIREMENTS

Table 5-1 - Support Equipment Requirements

Check	Frequency	Acceptance Criteria	Documentation
NIST Traceable Weights	Every 5 years	Certificate of Calibration from outside accredited service.	Records of calibration maintained by Quality Assurance Officer.
Working class weights	Annually using NIST traceable weights	Refer to the current revision of Katahdin SOP CA-102.	Records of calibration maintained by Quality Assurance Officer.
Balances	Daily or before use with working class weights	Refer to the current revision of Katahdin SOP CA-102 and balance calibration logbooks.	Calibration records documented in individual balance logbooks maintained at each balance location.
Refrigerators/ Freezers	Daily (7 days) for sample and extract storage. Daily (working) for others.	Refrigerators: 0 °C to 6 °C Freezers: ≤ -10 °C	Calibration records documented in individual temperature logbooks maintained at each location.
Ovens	Before and after on days of use.	Specific criteria set by methods (i.e. TSS method)	Recorded in individual analytical logbooks for methods requiring oven use.
Incubators	Daily (7 days) for environmental samples. Daily (working) for food samples.	Specific criteria set by methods (i.e BOD method)	Calibration records documented in individual temperature logbooks maintained at each location.
Water Baths	Before and after use.	Specific criteria set by methods (i.e fecal coliform method)	Calibration records documented in individual temperature logbooks maintained at each location.
NIST traceable Thermometers	Replace or recertify by expiration date on Traceable Certificate of Calibration (Digital).	Certificate of Calibration from outside accredited service.	Records of calibration maintained by Quality Assurance Officer.
Working Thermometers	Annually for spirit thermometers, quarterly for digital thermometers, semi-annually for food and microbiolory thermometers, using NIST traceable thermometer	Refer to the current revision of Katahdin SOP QA-809.	Records of calibration maintained by Quality Assurance Officer.
Autoclave	Annually for temperature and pressure. Cycle time, temperature and pressure with each use.	Specific criteria set by methods (i.e standard methods)	Records documented in autoclave logbook.
Adjustable Pipettors	Daily at maximum volume (Inorganics). Weekly at maximum volume (Microbiology). Monthly at minimum, mid and maximum.	Refer to the current revision of Katahdin SOP CA-103.	Calibration records documented in individual pipette logbooks maintained at each location.
Volumetric Glassware	Class A or B – Upon evidence of deterioration Class B – By lot before first use Also refer to Figure 5-1 for microbiology requirements.	Accuracy – Mean within ±2% of nominal value Precision – RSD ≤1% of nominal value (based on 10 replicate measurements)	Records of calibration maintained by Quality Assurance Officer.
Non-volumetric glassware (when used for measuring initial or final volumes)	By lot before use.	Accuracy – Mean within ±3% of nominal value Precision – RSD ≤3% of nominal value (based on 10 replicate measurements)	Records of calibration maintained by Quality Assurance Officer.

FIGURE 5-1

MICROBIOLOGY SUPPORT EQUIPMENT
REQUIREMENTS

COLIFORM QC & MAINTENANCE LOGBOOK INSTRUCTIONS

Temperature Distribution – This check must be performed for each incubator and for the autoclave one time and annually thereafter to the incubators or autoclave. Using the thermometer located in the apparatus, check and record the temperature in different locations. This should be done on at least every shelf of the incubators in several locations on each shelf, depending on the size. The autoclave should be checked in several locations also. The acceptance ranges for the temperatures must meet the acceptance ranges for the use of each piece of equipment. The incubator temperature range must be 35 °C ± 0.5°C. The autoclave temperature range must be 121 °C ± 1°C. If a certain shelf or area does not fall within the range, corrective action may be taken. Corrective action may include maintenance or temperature adjustment. Be sure to record any changes in the logbook. If corrective action fails to improve the temperature, please note in the log that any area out of range should not be used for the test.

Autoclave Sterility Check – This check must be performed on a monthly basis to ensure autoclave sterility standards. Autoclave a Raven Labs Pro Spore (Biological Indicator) ampule for 15 minutes at 121 °C @ 15 psi. Upon completion of the autoclave cycle, completely submerge the ampule in the water bath @ 60 ±2 degrees C for 48 hours. If the ampule turns a turbid orange-yellow color, then the criteria for the autoclave sterility performance have not been met. Repeat the test to rule out analyst contamination error. Corrective action/autoclave maintenance must be performed if a positive is verified during the second analysis. If the ampule remains its purple color, this indicates an adequate sterilization cycle. Record all information in the autoclave maintenance logbook section.

Timing Device Check – This check must be performed quarterly or after any major maintenance to the autoclave. Using a stopwatch, check the timer on the autoclave. The autoclave timer should agree within 10% of the stopwatch.

Analyst Counting Check – Each analyst who may be counting colonies, must verify their counting accuracy monthly. Compare the counts of the primary and secondary analysts. They need to be within 10% of each other.

Sample Container Sterility Check – This check must be performed on every new lot # of IDEXX and Greenwood Products (or equivalent) 100 mL plastic containers. Aseptically pour approximately 20 mL of sterilized TSB into 3 containers of each new lot #. Close the lid and swirl the container so that all inner wall surfaces contact the TSB. Incubate the containers at 35 °C for 2 days. Any bacterial turbidity seen in the TSB could be indicative of analyst contamination, so the test should be repeated. If the vessels contain turbidity on the second analysis, then the vessels fail to meet the sterility criteria and should not be used. New lots should be ordered.

Membrane Filter Sterility Check - This check must be performed on every new lot # of membrane filters. Aseptically fill four IDEXX 100 mL vessels with 80 to 100 mL of sterilized TSB. Using forceps and flame technique, add one filter membrane to each vessel, ensuring that it is completely submerged (reserve one vessel for blank control). Incubate the containers at 35 °C for 2 days. Any bacterial turbidity seen in the TSB could be indicative of analyst contamination, so the test should be repeated. If the vessels contain turbidity on the second analysis, then the membrane filters fail to meet the sterility criteria and should not be used. New lots should be ordered.

Precalibrated Sample Container Accuracy Verification – This check must be performed with each new lot of precalibrated sample containers. Fill the sample container to the precalibrated 100 mL mark with DI water. Transfer the water to a graduated cylinder and record the volume of water. Repeat this step nine more times and record the volumes. Determine the average volume. Determine the % deviation (% D) using the formula provided.

Autofluorescence Check of Sample Containers - This check must be performed with each new lot of sample containers. Place the container within 5 inches under a 6 watt (365 nm) UV light source in a darkened room. Shine the UV light away from eyes and wear protective UV eyewear and gloves. The container should not fluoresce.

Bromothymol Blue Test- Check batches of clean glassware monthly for pH reaction (from cleaning solutions). Add a few drops of 0.04% bromothymol blue (BTB) and observe the color reaction. BTB should be blue-green in the neutral range.

UV Light Box Efficiency – Prepare a dilution (~1:100,000) of E coli (assayed at > 40,000 CFU's/mL). Plate this dilution on 6 m-Fc pour plates, place 3 plates of the dilution into the UV light box and expose for 20 minutes. Incubate all six plates for 24 hours at 44.5 ± 0.2 ° C. Determine an average plate count of each set of plates. Calculate the % die off using the calculation: ((No UV) – (With UV) / (No UV)) *100. The reduction in counts after UV exposure should be 99%.

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TABLE 5-2

ANALYTICAL INSTRUMENTATION

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Agilent 7890/5975 GC/MS with EPC, Archon 5100 Autosampler and EST ENCON Evolution Purge and Trap Concentrator with heated purge capability	2013/2015	5030/8260, 5035/8260, 624 & 524.2	D
Agilent 6890/5973 GC/MS with EPC, Centurion Autosampler capable of low soils per Method 5035 and EST ENCON Evolution Purge and Trap Concentrator with foam sensor option.	2006/2011	5030/8260, 5035/8260, 624 & 524.2	T
Agilent 6890/5973 GC/MS with EPC, Centurion 5100 Autosampler with foam sensor option and ENCON Evolution Purge and Trap concentrator.	2014	5030/8260, 5035/8260, 624 & 524.2	P
Agilent 6890/5973 GC/MS with EPC, Centurion WS Autosampler with foam sensor option and EST ENCON Evolution Purge and Trap Concentrator with heated purge capability	2014	5030/8260, 5035/8260, 624	W
Agilent 6890/5973 GC/MS with EPC, Entech Instruments 7100AR Preconcentrator, 7410 10-position Robotic Headspace/Canister Inlet System and 3100A Canister cleaning system	2006/2011	TO-15 / MADEP APH	AIR
Hewlett Packard 5973 GC/MS with EPC and Model 6890N GC and Agilent 7673 autosampler.	2008	8270, 625	G
Hewlett Packard 5975B GC/MS with EPC and Model 6890 GC and Agilent 7683 autosampler.	2011	8270, 625	N
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7673 autosampler	1999/ 2001	8270, 625	U
Agilent Model 6890 gas chromatograph with one EPC injection ports and micro electron capture detectors; Agilent Model 7673 autosampler	2008	608, 8081, 8082 (including congeners), 8151	GC01

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TABLE 5-2

ANALYTICAL INSTRUMENTATION (cont.)

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard Model 6890 gas chromatograph with flame ionization detector and photo ionization detector; Eclipse 4660 Purge and Trap Sample Concentrator, 4551A Autosampler, LV20 Vial Autosampler	1988/2010	8015 MOD., MAVPH, GRO	GC02
Hewlett Packard Model 5890 gas chromatograph with a flame ionization detector; Agilent Technologies G1888 Network Headspace Analyzer.	1991/ 2005	Methane, Ethane and Ethene	GC05
Hewlett Packard Model 5890 gas chromatograph with nitrogen-phosphorous and flame ionization detectors; Hewlett Packard Model 7673 autosampler.	1993	DMF 8141 (method development)	GC06
Agilent Model 6890 gas chromatograph with dual EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	2000	608, 8082 (including congeners)	GC07
Agilent Model 6890 gas chromatograph with one EPC injection ports and micro electron capture detectors; Agilent Model 7673 autosampler	2009	504, 556, 608, 8011, 8081, 8082 (including congeners), 8151	GC08
Hewlett Packard Model 6890 gas chromatograph with flame ionization detector and photo ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 3000 purge and trap.	2008/2015	8015 MOD., GRO, MAVPH	GC09
Hewlett Packard Model 6890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	2013	8015 MOD., MAEPH, DRO	GC10
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector; Hewlett Packard Model 7673 autosampler.	1992/ 1996	ALCOHOLS, GLYCOLS	GC11

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TABLE 5-2

ANALYTICAL INSTRUMENTATION (cont.)

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	1987	8015 MOD., MAEPH, DRO	GC12
Hewlett Packard series 1100 HPLC with Quaternary pump, Multiwavelength detector and autosampler	2008	8330, 8332	HPLC02
HP Series 1100HPLC Quaternary pump, autosampler, and Foxy R2 fraction collector.	2010/2013	EPH Fractionation	HPLC03
J2 Scientific AccuPrep MPS™ GPC with internal UV detection	2009	3640	GPC01
J2 Scientific AcPrepLine™ GPC with internal UV detection	2014	3640	GPC03
Horizon SPE-DEX 3000 Automated Extractor System equipped with 3 extractors.	2006	1664, 9070, 9071	Horizon #2
Dionex Accelerated Solvent Extractor ASE 200	2003	3545	ASE #1
Milestone Ethos EX Microwave Extraction System	2011	3546	MW01
Organomation 24-position N-Evap systems (2)	1998/2010	GC and GC/MS Extractables	EXT01 & 02
Organomation 8-position S-Evap system	2001	GC and GC/MS Extractables	EXT03
Thermo ICAP 6500 ICP Emission Spectrometer with autosampler.	2006	6010, 200.7	I
Agilent 7500a ICP-MS with autosampler	2007	6020, 200.8	J
CETAC M-6100 Automated Mercury Analyzer with Autosampler	2004	7470/7471, 245.1, 245.5	H

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TABLE 5-2

ANALYTICAL INSTRUMENTATION (cont.)

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Tekran Series 2600 automated mercury analyzer with gold amalgam preconcentration and atomic fluorescence detector; Model 2620 autosampler.	2000	Ultra-trace level mercury (1631)	G
CPI ModBlock™ Metals Digestion Unit – Four 48 Place Units.	Various	Metals Aqueous Digestions	Digestion Unit #1, #2, #3, #4
Environmental Express Hot Block – One 36 Place Unit	2017	Metals Aqueous Digestions	Digestion Unit #5
LACHAT Quickchem 8500 Series Z – Automated Ion Analyzer and autosampler.	2013	Various	WC1
Shimadzu TOC-V Combustion Analyzer, PC Controlled, High Sensitivity, Auto-Aqueous TOC Autosampler w/ 40 mL vials; Model SSM-5000A Solid Sample Module.	2002	TOC	TOC1
Shimadzu TOC-V Combustion Analyzer, PC Controlled, High Sensitivity, Auto-Aqueous TOC Autosampler w/ 40 mL vials; Model SSM-5000A Solid Sample Module.	2015	TOC	TOC2
Dionex ICS-2000 with pump, degasser, AS-DV automated sampler, anion self - regenerating suppressor, continuously regenerated anion trap column.	2011	Ion chromatography – Various Anions and VFAs	IC2
Dionex ICS-2100 with pump, degasser, AS-1 automated sampler, anion self -regenerating suppressor, continuously regenerated anion trap column.	2015	Ion chromatography – Various Anions	IC3
10 position Lab Crest Cyanide Midi-Distillation system.	2012	Cyanide	WC3
HF Scientific Dert 100B Turbidimeter	2017	Turbidity	WC14
Accumet pH/Conductivity Meter, Model 20	1998	Various	WC8

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TABLE 5-2

ANALYTICAL INSTRUMENTATION (cont.)

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Mettler T50 Autotitrator & Mettler Rondo 60 sample changer.	2013	Alkalinity	WC12
Orion 5-Star Multiparameter Meter	2011	Dissolved Oxygen & Biochemical Oxygen Demand	WC11
Konelab 20 Multi-Wavelength Photometric Analyzer.	2003	Various	Konelab #1
Lachat Micro-distillation Heating Unit	2016	Wet Chemistry	WC15
Thermo Spectronic Genesys 10uv spectrophotometer	2005	Wet Chemistry and Food Testing	Food #1
HP Series 1050 HPLC System	2011	Food Testing	HPLC01
Biomerieux Vitek® Immuno Diagnostic Assay System (VIDAS®)	2010	Food Testing	Food #2
Biomerieux Tempo Preparation Station – Barcode reader, Media stand, Dispensers, Tempo filler and filling racks.	2013	Food Testing	Food #3
Biomerieux Tempo Reading Station – Tempo Reader and incubator/Reader racks	2013	Food Testing	Food #4

6.0 SAMPLE CUSTODY

Chain-of-Custody encompasses three major elements: field sampling, laboratory analysis and final data file. A Chain-of-Custody (COC) documents possession of a sample from time of receipt through the analytical process. Katahdin Analytical Services has implemented standard operating procedures to ensure that sample custody objectives of traceability and responsibility are achieved for every project. This section covers quality related activities from the receipt of samples at the laboratory through the issuance of final analytical data and the storage of data in its final data file.

All areas of the laboratory in which samples are received, stored, processed, or analyzed shall be kept in a condition that minimizes the risk of samples becoming lost or accidentally destroyed, contaminated, degraded, mis-identified, improperly handled or otherwise compromised.

6.1 Chain-of-Custody

The National Enforcement Investigations Center (NEIC) of EPA defines evidence of custody in the following manner:

- It is in your actual possession, or
- It is in your view, after being in your physical possession, or
- It was in your possession and then you locked or sealed it up to prevent tampering, or
- It is in a secure area.

Katahdin Analytical Services sample custody and sample control procedures ensure that:

- All samples are uniquely identified;
- Samples are analyzed as requested and are traceable to their records;
- Important sample characteristics are preserved;
- Samples are protected from loss or damage;
- Any alteration of samples (e.g., filtration, preservation) is documented; and
- A record of sample integrity is established for legal purposes.

Samples may be physical evidence and should be handled according to certain procedural safeguards. In order to facilitate documentation of sample monitoring from collection through analysis, several forms are used by Katahdin Analytical Services personnel. Field personnel document field activities on standardized field data records

and laboratory personnel record sample handling activities on internal chain-of-custody forms and/or sample control records.

A Chain-of-Custody Form (Figure 6-1) is completed by field personnel or client representatives for all samples received by the laboratory; the form accompanies the samples received by the laboratory. The completed Chain-of-Custody Form should include the following information:

- Client and project name
- Project location
- Field sample number/identification
- Number and type of containers
- Date and time sampled
- Sample matrix
- Preservative
- Analysis requested
- Sampler signature
- Signature of person relinquishing samples
- Date and time relinquished
- Sampler remarks
- Custody Seal Number (if applicable)

The record is filled out completely and legibly. Errors are corrected by drawing a single line through and initialing and dating the error. The correct information is then recorded with indelible ink. All transfers of samples except to and from commercial couriers must be recorded on the Chain-of-Custody via the "relinquished" and "received by" sections. All information except signatures may be printed.

When samples are received by the laboratory (refer to Section 6.3), Sample Management Personnel sign and note date/time received on the accompanying airbill, if present. The Chain-of-Custody is signed after Sample Management Personnel have verified the contents of the sample shipment. Sample Management Personnel verify the integrity of samples as they are unpacked, ascertain whether a Custody Seal is present and intact, whether the samples are received intact or broken, whether the samples are appropriately preserved and properly identified, what packing material was present in the shipping container, the temperature of the container, and any other notable observations. This information is explicitly documented on the Sample Receipt

Condition Report (SRCR) and the project manager (PM) is notified if any discrepancies or problems are found. The PM then contacts the client to receive further instructions pertaining to the affected samples. If the integrity requirements are met or after any discrepancies are resolved, the sample is assigned a unique laboratory identification number and transferred to the appropriate storage location for storage until preparation and analysis. Pertinent information concerning each sample is entered into the Katahdin Information Management System (KIMS); refer to Section 6.3 for more information on log-in procedures.

Once samples are in the laboratory, an internal custody record is generated to track the transport and status of each sample from storage to the laboratory and back to storage. After sample log-in, a project file containing the external Chain-of-Custody record and all sample receipt documentation is started by a client services representative. Further detail on internal laboratory custody procedures is provided in subsequent subsections.

6.2 Sampling Kits

In general, sampling kits are comprised of the following:

- Sampling containers
- Preservatives (upon request) and appropriate MSDSs
- Chain-of-Custody forms
- Custody Seals (upon request)
- Sample labels
- Packing Material
- Shipping containers
- Ice Packs (upon request), although ice is preferred
- Temperature blanks

Sample kit requests are received by the laboratory personnel from a project manager or sampling team members via telephone request, memo, or facsimile.

Based upon the specific request, the Sample Custodian or Project Manager determines the appropriate containers, preservatives and the necessary volume/quantity to specify for the analysis. This information is outlined in Table 6-1. The pertinent information is recorded on a Container Request Form. Refer to the current revision of Katahdin Analytical Services SOP QA-901, Sample Container Preparation and Shipping, for further information on this form.

6.2.1 Sample Containers

All volatile organic analysis (VOA) vials (40 mL) and sample containers for other analyses are purchased pre-cleaned from a commercial source. In some cases pre-cleaned containers are received with certificates of analysis documenting the concentration levels of applicable analytes for each container type and lot. Certificates of analysis accompanying container lots are maintained by the laboratory, and a record is kept of sample container lot numbers utilized for each project. All sample containers are purchased from reputable suppliers with products meeting project or protocol requirements.

Under no circumstances are used bottles sent out to the field.

Sampling containers are stored in an area of the Katahdin Analytical Services laboratory designated for staging sampling events and separate from the analytical laboratories. The containers are stored by lot number in the boxes in which they are received. Refer to the current revision of Katahdin Analytical Services SOP QA-901, Sample Container Preparation and Shipment, for more information.

6.2.2 Assembling Kits

All glass containers are surrounded with packing material to prevent damage.

Bottles requiring preservative may be pusrchased already preserved or the preservative may be added in the laboratory. All pre-preserved bottles must be labeled with the preservative added.

The appropriate number of labels and COCs are enclosed in the ziplock plastic bag that is included with the kit.

All the above contents are placed inside a cooler with an appropriate cooling medium (if requested) and a temperature blank. Field personnel are responsible for packing the shipping container to maintain the proper temperature. Ice packs may be provided upon request, however, ice is preferred for shipment of samples back to the lab. Sample containers are secured for shipment using appropriate packing materials.

Sample kits are delivered to the sampling team via the appropriate courier or personally picked up by the sampling team.

Refer to current revision of Katahdin Analytical Services SOP QA-901, Sample Container Preparation and Shipment, for further information on sampling kits.

6.3 Sample Receipt and Log-In

Refer to the current revision of Katahdin Analytical Services SOP SD-902, Sample Receipt and Internal Control.

Typically, samples are received by the laboratories during normal business hours (8:00 am to 6:00 pm), Monday through Friday.

Shipments for after hours and weekend delivery are prearranged with laboratory personnel to ensure that personnel will be available to sign the airbill, record the date and time of sample receipt and place the cooler in the appropriate location in the sample management area (under refrigeration) until the next business day.

Upon sample receipt, the coolers are inspected for the general condition of the Custody Seal, if present. The coolers are then opened and each sample is inspected for damage. The sample containers are removed from the packing material and identities are verified against the Chain-of-Custody. All information regarding sample condition upon receipt is documented on the Sample Receipt Condition Report (SRCR). The report documents:

- Name of person if hand delivered;
- Presence/Absence of COC forms and Custody Seals;
- Condition of the custody seals if present;
- Discrepancies noted;
- Holding times;
- Proper preservation (i.e. pH of all samples, except volatiles samples, is verified). The pH of volatile samples is checked at analysis and recorded in the analytical run log. If additional preservative is added, a sticker with the type of preservative, date and time added, final pH and custodian's initials must be placed on the sample container. Samples must be preserved for at least 24 hours for trace metals analysis.
- Proper sample containers, properly labeled according to the COC and unbroken;
- Appropriate sample volume; and
- Cooler temperature

The Sample Receipt Condition Report is completed by signing and recording the date and time of sample receipt. If there are any discrepancies or problems with the samples or accompanying documentation, the Sample Custodian immediately notifies the client or the appropriate Katahdin Analytical Services project manager for resolution.

The samples are logged into the Katahdin Analytical Services Information Management System (KIMS) by the Sample Custodian or Project Manager. Each group of samples received is assigned a unique laboratory number (work order number) at log-in. Each sample within the group is assigned a unique Sample Number by appending to the work order number a numerical suffix serialized to account for the number of samples in the sample group. All pertinent sample information, as detailed on the Chain-of-Custody,

SRCR, and other project related documentation, is entered into the KIMS and dispatched to the analytical groups for KIMS generation of preparation and analysis worklists. Upon completion of the log-in process, samples are placed into the appropriate storage area. Transfer of the samples from the Sample Custodian to each storage location is documented by the Sample Custodian on internal chain-of-custody records. Refer to the current revision of Katahdin Analytical Services SOP SD-902, Sample Receipt and Internal Control, for more information.

6.4 Sample Preservation and Storage

Samples are preserved according to the EPA's recommendations (refer to Table 6.1), unless there are overriding considerations, e.g. QAPP, and stored to minimize sample contamination and degradation. The laboratory must rely upon information supplied by the sampling team to document any known hazards. If it is known from past experience that samples contain high levels of contamination, those samples are segregated. For sample log-in procedures and supporting documentation, see Section 6.3 and the current revision of Katahdin Analytical Services SOP SD-902, Sample Receipt and Internal Control.

A holding blank comprised of deionized (DI) water is placed into the volatiles storage refrigerators in the volatiles lab to monitor the ambient concentration of analytes of concern in that storage unit. The Sample Custodian creates holding blanks on a bimonthly basis. The QAO is responsible for dispatching holding blanks for analysis of volatile organics by GC or GC/MS. Holding blank reports are monitored and maintained by the QA department.

6.5 Initiation of Testing Program

Once samples have been logged into the laboratory system, the external Chain-of-Custody, Sample Receipt Condition Report, and other related documentation are assembled into a project file. If a sample group is a priority or rush order, or if samples have short holding times, Sample Management Personnel will immediately provide notification to the appropriate laboratory personnel that samples have arrived and are ready for processing; the Sample Custodian may deliver the samples directly to the lab. KIMS-generated worklists for each laboratory section list all samples requiring preparation and analysis, the type of quality control samples required, the priority status, and test(s) required.

6.5.1 Internal Chain-of-Custody

A bar code system for tracking the status and chain-of-custody of samples within the laboratory is in place once sample processing begins. The bar code system is used by sample receipt, sample preparation and analysis (as applicable) personnel to electronically document the transfer of samples to and from storage locations and to and from the preparation/analysis labs. Hardcopy Internal Chain-of-Custody records are used to document the transfer of prepared sample extracts and/or digestates from sample preparation through analysis. Each electronic internal chain-of-custody record documents the following:

- Who removed the sample or extract from storage;
- When the sample or extract was removed from storage;
- Which sample(s) was removed from storage;
- If the sample was entirely consumed; and
- When and by whom the remainder of the sample was returned to storage.

Completed hardcopy internal chain-of-custody records are archived by the Quality Assurance department.

6.5.2 Subsampling for Sample Preparation or Analysis

In almost all cases, the laboratory receives more sample than is typically used for a specific analytical method. Therefore, a smaller aliquot, or subsample, must be obtained from the container for sample preparation or analysis. Obtaining a representative subsample, i.e. one that has the same characteristics and chemical composition as the original sample, can be difficult without employing complex techniques. In general, the following techniques shall be used.

Soil Samples

Soil samples must be physically mixed well prior to removal of the first aliquot for analysis. All visible foreign objects such as sticks, rocks, or leaves should be removed. The sample should be mixed with a scupula until it is visually homogenous. This process should not be used on soil samples for volatile analysis. For those volatile samples not preserved with methanol, an aliquot should be obtained from the middle of the jar (when possible), not the top, without any mixing.

Aqueous Samples

Aqueous samples must be mixed by inverting the sample several times prior to pouring off an aliquot. This inversion must be performed for each subsequent test requiring an aliquot.

Multiphasic Samples

Whenever the laboratory receives a multiphasic sample, the Katahdin Project Manager must be informed immediately prior to any subsampling. The Katahdin Project Manager will contact the client for further instructions. Depending on the end use of the data and prior knowledge of the sample, the client can provide the best information on the subsampling procedure. In some cases, the client may want the laboratory to provide their scientific judgment of which subsampling technique may be more appropriate. One of three options will typically be available:

1. Shaking the sample to form an emulsion layer so that the sample can be treated as a homogenous sample- the limitations to this method are the length of time that the emulsion will remain and how quickly a homogenous aliquot can be obtained and whether or not a complete emulsion can be formed. An analysis that cannot be performed immediately after subsampling (i.e. a subsample that may sit on an autosampler for a period of time) may not employ this technique. The biphases would likely reform if the emulsion were allowed to sit for a period of time.

2. Separating the two layers – this technique may be difficult near the interface of the two layers. This technique may involve analyzing both layers separately. The advantage is that two separate preparation methods could be employed if appropriate for the two distinct layers. However, in order to report the results properly, the layers would need to be weighted accurately.

3. Separating the two layers with analysis performed on only one layer – this technique may bias the results if there is any cross-contamination between the two layers.

In all cases, the reported data must be narrated so that the end user is aware of which subsampling method was used and which phase or phase was/were analyzed.

Note: any subsampling techniques performed on a sample for a volatile analysis would further compromise the sample by exposing the sample to the air (i.e there is the potential to lose volatile compounds) and by introducing headspace into the vial.

Other forms of subsampling may be used by the laboratory, but must be discussed with the client prior to the initiation of a project. This could include multi-icremental sampling as described in SW-846 Method 8330B.

6.5.3 Holding Time & Status Tracking

Samples are tracked through the KIMS system. Specific worklists are generated daily (at minimum) for each laboratory section. The lists contain the pertinent sample information needed to proceed with analysis including ID numbers, test codes, due dates and holding times. Each laboratory section schedules work according to these worklists. The worklists give the status for each sample batch/project within each analytical section. To update the worklist status of each sample or sample batch/project, pertinent information is entered into the KIMS once sample preparation and/or analysis has been completed. Refer to the current revision of Katahdin SOP SD-916, Tracking of Sample Status and Holding Times, for further information on worklists.

6.6 Sample Disposal

After completion of sample analysis and submission of the analytical report, unused portions of samples are retained by the laboratory for a minimum of thirty days. Unless otherwise specified by the client or analytical program, after submission of the data report, samples will be moved from the refrigerators for subsequent disposal according to the nature of the samples. The Katahdin Environmental Health and Safty Officer (EHSO) generates a KIMS sample disposal summary and uses that information to select the appropriate waste stream for the samples. Samples determined to be hazardous waste are handled by state and federally licensed hazardous waste disposal firms.

Upon disposal of samples, a record is generated by the Katahdin EHS Officer listing the sample number, inherent waste stream and date disposed. This record is maintained by the Katahdin EHS Officer. Please refer to the current revision of SOP SD-903, Sample Disposal, for further information.

6.7 Subcontracting Analytical Services

Every effort is made to perform all requested chemical analyses at Katahdin Analytical Services for our clients. There are, however, instances where subcontracting of analytical services is necessary. Currently, the following analyses most commonly require subcontracting by Katahdin Analytical Services, but are not necessarily limited to: Asbestos, Dioxins, Radiological, Perchlorate, and TOX.

When subcontracting becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. No work is subcontracted without consent from the client. Work requiring specific certifications or approvals (i.e. State of Maine, NELAC, DoD, etc.) shall only be subcontracted to laboratories that possess the required certifications or approvals. All subcontract laboratories must provide proof of certification or approval prior to any sample analyses. Katahdin shall maintain a registry of subcontract laboratories including records of compliance. This registry will be maintained by the Quality Assurance Officer.

The contact and preliminary arrangements and terms of agreement are made between the Katahdin Analytical Services Project Manager and the appropriate subcontract laboratory personnel (i.e., laboratory manager, client services contact, or the appropriate laboratory section manager). The specific terms of the subcontract laboratory agreement should include (when applicable):

- Method of analysis (including method reference, e.g., EPA SW-846, Standard Methods, etc.);
- Target analyte list required;
- Number and type of samples expected;
- Project specific QA/QC requirements (including reporting limit considerations);

- Deliverables required (including electronic deliverable if applicable);
- Applicable laboratory certification status;
- Price per analysis; and
- Turn around time requirements.

In the case of Federal Programs work, the subcontractor approval is completed as a joint effort between the prime laboratory and the Prime Contractor and through approval by the controlling Federal program office (i.e DoD, NFESC, AFCEE or ACOE). Work completed under these programs is contracted and involves a working Quality Assurance Plan. The prime laboratory and subcontracted laboratory both have a chance to review and comment on the QA Plan. The criteria listed in the QA Plan is the criteria that the laboratories are contracted to follow.

Chain-of-Custody forms shall be generated for samples that require subcontracting to other laboratories. The sample management personnel repackage the samples for shipment, create a transfer chain-of-custody form and record the following information:

- Katahdin Analytical Services Sample Number(s);
- Sample matrix;
- Requested analysis;
- Special instructions (quick turn around, required detection limits, unusual information about the samples or analytical procedure); and
- Signature in "Relinquished By".

All subcontracted sample data reports are reviewed for completeness after receipt by Katahdin Analytical Services. The subcontracted data results may be reported through Katahdin Analytical Service's KIMS or the subcontracted data report may be appended in whole to Katahdin Analytical Service's analytical data report. The final report format is determined through communication between the Katahdin Analytical Services Project Manager, Data Management Group, and the client. The contents of a subcontracted report are never altered. Please refer to Katahdin SOP, SD-900 (current revision), Subcontracting Analyses, for further details.

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES - AQUEOUS					
Acidity	SM 2310B, 305.1	100 mL	P,G	1,2	14 days
Alkalinity- Titrimetric	SM2320B, 310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1/350.2 SM4500NH3 B&H	100 mL	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, SM4500NH3 H	100 mL	P,G	1,3	28 days
Anions (F, Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (calculation from alkalinity)	SM4500-CO2 D				
Biochemical Oxygen Demand-Carbonaceous	SM 5210B, 405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	SM 5210B, 405.1	1 L	P,G	1	48 hours
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	SM4500-Cl E, 325.2	100 mL	P,G	1	28 days
Chlorine, Total Residual	SM4500-Cl G, HACH 8167	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	SM2120B, 110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	SM4500CN G, 335.1	100 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.4	100 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	SM4500-O G, 360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1,12	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F B/C, 340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	SM4500F C, 340.2	200 mL	P only	1	28 days
Free CO2	SM4500-CO2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2, SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	SM4500-NO3 F, 353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1, 3 OR 11	28 days
pH (Laboratory)	SM 4500H B 150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	SM4500-P E, 365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	SM 2540C, 160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	SM 2540D, 160.2	1 L	P,G	1	7 days

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Solids-Settleable Solids (SS)	SM2540F, 160.5	1 L	P,G	1	48 hours
Solids-Total Solids	SM 2540B, 160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	SM 2540E, 160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	SM2540C/E, 160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance	SM2510B, 120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	ASTM D516-02, 375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	SM4500-S2 F, 376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	SM4500-SO3 B, 377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	SM2130B, 180.1	100 mL	P,G	1	48 hours
Volatile Fatty Acids	SOP CA-776	(2) 40 mL	VOA vial	17	14 days
ELEMENTAL ANALYSES - AQUEOUS					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	16	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES - AQUEOUS					
EDB, DBCP & 1,2,3-TCP	8011 & 504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP EPH	(2) 1000 mL	Amber Glass	1,12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1, 18	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Petroleum Range Organics	FL-PRO	(2) 1000 mL	Amber Glass	1,12	7days/40days
Total Petroleum Hydrocarbons	TX1005	(2) 40 mL	VOA vial	12	14days/14days
Extractable Total Petroleum Hydrocarbons	CT-ETPH	(2) 1000 mL	Amber Glass	1	7days/40days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days
PCB Congeners	8082	(2) 1000 mL	Amber Glass	1	7days/40days

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2) 40 mL	VOA vial	1,11	14days
Chloropicrin	8011 Mod.	(2) 40 mL	VOA vial	1,8,9	14 days
HPLC ANALYSES – AQUEOUS					
HPLC-Explosives	8330A/B/ B Mod.	(2) 1000 mL	Amber Glass	1	7days/40days
GC/MS ORGANIC ANALYSES – AQUEOUS					
Acid Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles – Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables & (SIM)	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics & (limited SIM)	8260/8260 SIM	(3) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics	624	(3) 40 mL	VOA vial	1,8,9	14 days(~)
MICROBIOLOGICAL ANALYSES – AQUEOUS					
Coliform, Fecal (wastewater)	SM 9222D	100 mL	P,G	1,6	6 hours
Coliform, Fecal (wastewater)	Colilert-18 w/ Quantitray	100 mL	P,G	1,6	6 hours
Coliform, Total (wastewater)	SM 9222B	100 mL	P,G	1,6	6 hours
Coliform, Total (drinking water)	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total (drinking water)	SM9223B, Colitag	100 mL	P,G	1,6	30 hours
E-coli (wastewater)	SM9213D	100 mL	P,G	1,6	6 hours
E-coli (wastewater)	SM9223B Colilert w/ Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B, SIMPlate	100 mL	P,G	1,6	8 hours for compliance samples, 24 for non-compliance samples
GENERAL CHEMICAL ANALYSES – SOLID					
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1/350.2 SM4500NH3 B&H mod.	4 oz	Soil Jar	1	28 days (^)
Anions (F, Cl, Br, NO3, NO2, SO4)	9056	4 oz	Soil Jar	1	48hrs to 28 days (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/9056	4 oz	Soil Jar	1	28days (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	SM4500F B/C, 340.2 mod.	4 oz	Soil Jar	1	28 days (^)

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	9056 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Nitrite-Automated Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	28 days (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	9056 mod./365.2	4 oz	Soil Jar	1	48 hrs (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	SM2540 G, current CLP SOW	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9038	4 oz	Soil Jar	1	28 days (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Carbon	9060	4 oz	Soil Jar	1	28 days
Total Organic Carbon	Llyod Kahn	4 oz	Soil Jar	1	14 days
Total Organic Carbon	Walkley Black	4 oz	Soil Jar	1	14 days
ELEMENTAL ANALYSES – SOLID					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS Elements	6020	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES – SOLID					
Extractable Petroleum Hydrocarbons	MADEP EPH	4 oz	Soil Jar	1	14days/40days
Fuel Oil	ME HETL 4.1.25 & 8015 mod.	4 oz	Soil Jar	1	14days/40days
Petroleum Range Hydrocarbons	FL-PRO	4 oz	Soil Jar	1	14days/40days
Total Petroleum Hydrocarbons	TX1005	4 oz	Soil Jar	1	14days/14days
Extracted Total Petroleum Hydrocarbons	CT-ETPH	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17 & 8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2)40 mL	VOA vial	1,13	28days

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
HPLC ANALYSES – SOLID					
HPLC-Explosives	8330B/B Mod.	4 oz or ISM sample	Soil Jar	1	14days/40days
GC/MS ANALYSES – SOLID					
Acid Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables & (SIM)	8270/8270 SIM	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics – Low Soil (<200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics & (limited SIM)	8260/8260 SIM	(2) 40 mL	VOA Vial	1	14 days
Miscellaneous – SOLID					
Grain Size (sieve and hydrometer)	ASTM D422	8 oz	Soil jar or bag	1	none
RCRA – HAZARDOUS WASTE CHARACACTERIZATION					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311/8260	100 g	Soil Jar	1	14 days/14 days
TCLP Extraction-Semivolatiles	1311/8270	200 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Pesticides & Herbicides	1311/8081 & 8151	400 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Metals	1311/6010/6020	200 g	Soil Jar	1	28 days/180 days
TCLP Extraction-Mercury	1311/7470	200 g	Soil Jar	1	28 days/28 days
GC/MS ANALYSES – AIR					
Volatile Organics	TO-15	(1) 1.4 or 6 L	Canister	16	30 days
Volatile Organics	MA-DEP APH	(1) 1.4 or 6 L	Canister	16	30 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NznAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate
16 = None
17 = benzalkonium chloride
18 = 0.02g ammonium sulfate, 0.02g copper (II) sulfate pentahydrate

~ Hold time for unpreserved samples is 7 days.

^ Because there are no published holding times for Wet Chemistry soil methods, these are only recommended holding times. They are not regulatory.

Project-specific (i.e. CLP, NYSDEC) hold times take precedence over these hold times as appropriate.

For solid samples, please place parameters of the same analytical group (ie. wet chemistry) in the same container whenever possible. In addition, organic and inorganic parameters should be placed in separate containers. Volatile organics should always be placed in organic-free jars. Several 4 oz. soil jars may be needed when numerous parameters are required.

References:

EPA - 40 CFR 136
SW846 - Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW846, 3rd edition, Final Update III, 1986, 1997
SM - Standard Methods for the Examination of Water and Wastewater, 16th ed., 1985
ASTM - American Society for Testing and Material

FIGURE 6-1

Katahdin Analytical Services, Incorporated
Chain-of-Custody Record



Katahdin ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To

LAB USE ONLY | WORK ORDER #:

KATAHDIN PROJECT NUMBER

REMARKS:

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO.:

TEMP °C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE / PRESERVATIVE

*	Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
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COMMENTS

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THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS.

ORIGINAL

FIGURE 6-2

Katahdin Analytical Services, Incorporated
Method 5035 Collection and Preservation

Method 5035 Closed System Purge&Trap

Method 5035 details the current techniques that are required to minimize the loss of volatile organic compounds (VOC's) due to sample transport, handling, and analysis. Method 5035 provides various options for sampling including the low soil method (5.0ug/kg - 200ug/kg), which uses a hermetically sealed voa vial, and procedures for high concentration soils or wastes (> 200ug/kg). Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination.

Procedure for low level soils (<200ug/kg)

40 ml VOA vials containing either 5 ml of 20% sodium bisulfate or 5 ml of DI water, and a magnetic stirring bar are pre-weighed by the lab and shipped to the field. The field samplers collect an approximate 5 gram sample, as weighed in the field at the time of collection and place the sample in the pre-weighed and preserved vial. The vial is then returned to the laboratory for analysis. Upon receipt by the laboratory, samples that are preserved with DI water must be frozen immediately until the time of analysis. All samples must be brought to room temperature prior to analysis and analyzed within 14 days from the time of collection. Samples are analyzed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. Soil samples must be taken in accordance with the procedures outlined in the sampling plan, and may include the use of EnCore sampling devices. Alternatively, the field samplers may collect the soils in EnCore sampling devices, or equivalent and return these to the laboratory. Depending on the specifics of the project, the laboratory will extrude the soils into the 5 ml of DI water or 5 ml of 20% sodium bisulfate within 48 hours of collection. The laboratory will proceed as described above.

Procedure for high concentration soils (> 200 ug/kg)

High concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

Methanol Field Preservation- A 5 gram sample is added to a VOA vial that was charged with 5 mls of purge and trap grade methanol and weighed in the lab prior to shipment to the field. If possible the field should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field.

Considerations

1. Samples that require multiple analyses to confirm matrix interferenee's or to perform sample specific QC (MS/MSD/Dup) will require multiple vials (3-9 vials) since only one analysis is permitted per vial.
2. Samples that contain VOC's over a wide range of concentrations may require additional vials with smaller amounts of sample to come from the field.
3. Separate aliquots are required for percent solids determination for low level samples and high concentration samples that are field preserved with methanol.
4. The sodium bisulfate solution is acidic and may foam on contact with alkaline matrices or highly calcareous soils.
5. Vial weights should be measured in the field and verified in the lab. Samples must be well sealed and packaged so that vial weights are not affected by shipping conditions.
6. Unless sample history is known, Katahdin suggests the following samples be taken at minimum: one 4 oz. Soil jar for solids, one methanol field preserved sample for high concentration analysis, and two field preserved samples for low level analysis.

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7.0 CALIBRATION PROCEDURES AND FREQUENCY

All instruments and equipment used in Katahdin Analytical Services laboratories must follow a well defined calibration routine. All standards and instruments are designated by a unique identifier. This identifier shall be recorded in standards logbooks, run logs and all raw data that are pertinent. Calibration may be accomplished by laboratory personnel using certified reference materials traceable to NIST or EPA certified materials or by external calibration agencies or equipment manufacturers. The discussion presented here is general in nature because the requirements for calibration are instrument (or equipment) and method specific. Details of calibrations can be found in Katahdin Analytical Services Standard Operating Procedures, analytical methods, and operations manuals.

7.1 Standards and Traceability

Analytical standards are prepared from pure compounds or are purchased prepared from reputable vendors. These standards provide the stock used to prepare serial dilutions for calibration and spiking standards. Each laboratory section is responsible for the preparation, storage and disposal of its standards. Pertinent standards preparation information is recorded into laboratory specific standards logbooks in order to document traceability of prepared standards to their source material(s).

Each standard is given an internal identification number. The preparation of all stock standards shall be documented in a standards notebook which is used to record the date of preparation, analyst's initials, source of the reference material, standard components, amounts used, final volume, final concentration(s), solvent used, expiration date of prepared standard, and the assigned serial reference number (internal identification number) of the stock solution. All standards shall be labeled, at minimum, with a unique identifier for the prepared standard, preparation date and expiration date, and, if space permits, the reference number(s) of the stock standard(s), the name of the standard, concentration, and initials of the preparer. All diluted working standards not consumed during an analytical session shall be labeled fully, including the serial reference number of any stock standard used in its preparation. Working standards, which are consumed the same day that they are prepared, do not have to be labeled on the container itself.

If no expiration date has been assigned by the manufacturer, then an expiration date of one year from the date of preparation (or the date first opened in the case of sealed ampules) is generally reported unless degradation prior to this date is observed. The expiration date assigned to a prepared standard shall not exceed the expiration date of any individual component in the solution. To help determine if a standard has degraded, one must note inconsistencies. For instance, very poor recoveries from newly prepared quality control spikes or abnormally low instrument response to a specific standard are indications of possible standard degradation. However, for some standards, degradation is more easily noted. For instance, DDT breaks down to form DDD and DDE. Here one can visually note, on a chromatogram, the degradation of DDT by the increased concentrations of DDD and DDE. If degradation is observed before the default expiration date, it should be noted in the standards notebook for that standard entry and the standard removed from service. Refer to the current revision of Katahdin

Analytical Services SOP CA-106, Standard Preparation, Documentation and Traceability for more information.

Reference standards must be traceable to national standards of measurement (e.g. NIST) whenever possible. Standards used for calibration must be traceable, when possible, to national standards of measurement, either directly through supplier documentation or by verification against a second source traceable reference standard. Lists of reference standards used are contained within individual analytical method SOPs.

7.2 Support Equipment Calibration Procedures

7.2.1 Analytical Balances

Every twelve months, calibration of the entire analytical range shall be checked by a qualified service technician. The calibration of each balance is also checked each day of use by the analyst. Each department will be equipped with ASTM Class 2, or less, weights (when supplies permit) to be stored in a desiccator. The ASTM Class 2 (or less) weights are used for daily calibration so as to prevent damage to the ASTM Class 1 weights. The ASTM Class 1 weights will used to verify the weights of the ASTM Class 2 weights annually. The QAO will verify the weights and adjust the true values of the class 2 weights as necessary. The QAO will arrange for outside verification (NIST traceable) of the class 1 weights every five years. All information pertaining to balance maintenance and calibration is found in the individual balance logbook and/or is maintained by the QA Department. Please refer to the current revision of Katahdin SOP CA-102, Balance Calibration.

7.2.2 Thermometers

Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and recertified by expiration date on Traceable Certificate of Calibration (Digital) or every five years (Mercury) with equipment directly traceable to the NIST.

Each thermometer is individually numbered and tagged with the identification number. All working thermometers are compared with the reference thermometers on a quarterly, semi-annually or an annual basis. The timing is based on thermometer type and program requirements. In addition, working thermometers are visually inspected by laboratory personnel prior to use. Calibration temperatures and acceptance criteria are based upon the working range of the thermometer and the accuracy required for its use. An inventory of thermometers, their identification, calibration status and due date of next calibration is maintained by the QA Department or designated area. Refer to the current revision of Katahdin SOP QA-809, Working Thermometer Verification, for further information.

7.2.3 pH/Electrometers

These meters are calibrated using buffer solutions before use each day, and once after each four hours of use. Refer to the current revision of Katahdin Analytical Services SOP CA-708, Hydrogen Ion Concentration Measurement (pH) in Aqueous Samples and Solutions.

7.2.4 Spectrophotometers

During use, spectrophotometer performance is checked against initial calibration verification standards (ICVs) and continuing calibration verification standards (CCVs). The instrument operating capability is also evaluated every year by an outside service. Wavelength verification occurs every 12 months.

7.2.5 Ovens

Oven temperatures are monitored using a high temperature range digital thermometer intended for oven monitoring. Oven temperature is checked every day of use at the beginning of the analysis and at the end of the analysis and recorded in the appropriate logbook. Oven temperatures are also verified every 12 months by an outside source. Specific oven temperature recording requirements are described within each analytical SOP.

7.2.6 Incubators

Incubators temperatures are monitored using thermometers intended for incubator monitoring. Incubator temperature is checked every day of use at the beginning of the analysis and at the end of the analysis and recorded in the appropriate logbook. Specific incubator temperature recording requirements are described within each analytical SOP.

Refer to Table 5-1, Support Equipment Requirements, for additional support equipment requirements.

7.3 Instrument Calibration Procedures

Calibration procedures and acceptance criteria are method specific. Refer to the individual methods (see Tables 6-1 and 8-2) or Katahdin Analytical Services SOPs for method specific requirements in addition to the generic procedures outlined here.

Calibration of the analytical system is a critical step in the generation of quality data. Calibration of an analytical instrument involves a graphical representation of instrument response versus concentration of an analyte. The tabulation is referred to as the calibration curve.

Calibration approaches may be external standard calibration, internal standard calibration or isotope dilution calibration. Within these approaches the calibration may be linear or non-linear.

The initial calibration must be established before analysis of any samples. All calibrations are initially verified using a standard that is independent from the calibration standard. The calibration must then be verified on each day of use. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. An initial calibration must

The initial calibration must be used to quantitate samples.

When the initial calibration, the independent calibration verification or the continuing calibration verification are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible.

Please refer to the individual methods (see Tables 6-1 and 8-2) or Katahdin Analytical Services SOPs for method specific requirements.

7.4 Manual Integration Policy

The GC/MS and some of the GC group both use the data processing system, Target. The data processing system, Turbochrom is used by the rest of the GC group and Chromeleon is used for IC analysis. Target, Turbochrom and Chromeleon are designed to perform automatic identification and integration of peaks. Automatic integration is preferred because it is fast, and in most cases, very consistent. Proper instrument and integration parameter set-up facilitates automatic integration.

Consistency in integration between standards and samples is one of the most important considerations in quantitative chromatographic analysis. Split peaks, peak tailing, noisy baseline, coelution, or incomplete resolution of isomeric pairs can sometimes complicate automatic integration. In addition, the data system may incorrectly identify a peak. In these cases, analyst intervention, or manual integration, is necessary. Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern.

Please refer to the current revision of Katahdin SOP QA-812, Manual Integration on GC/MS, GC, HPLC And IC Datasystems, for more information.

8.0 ANALYTICAL PROCEDURES

Katahdin Analytical Services is capable of analyzing a broad range of environmental samples from diverse media, including surface and groundwater, soil, sediment, tissue, and waste. Refer to Table 8-2 for a listing of specific analytical capabilities. Methodologies are typically used from national or regional sources such as EPA, ASTM, USGS, APHA, AOAC, BAM and in certain instances, state regulatory agencies. In some situations, Katahdin Analytical Services develops and validates methodologies that are more applicable to a specific problem or objective. In cases where specific methods are required by regulation, the most recent and approved version of the method must be used. When not required by regulation, the laboratory shall consult with the client as to which approved version of a published method shall be used. Any modifications to these published methods must be approved by the client or regulatory (i.e. DoD) agency.

Note: These modifications must be explained in the modifications table for each SOP when these modifications are performed regularly or in the case narrative for modifications for a specific project only.

In cases where the client does not specify a method or analyte list, the laboratory will propose an appropriate method and list to the client for approval. In cases where an outdated or inappropriate method may be proposed by the client, the laboratory shall inform the client of this and propose a suitable method.

Analytical procedures are detailed descriptions of any and all processing, preparation and analysis of samples in the laboratory. In some instances, data format, presentation and delivery are also described. All analytical procedures shall be conducted in strict adherence to the QA Manual and written Standard Operating Procedures that have been reviewed and approved by the President, the QA Officer, and department managers/group supervisors. Documents from which SOPs are developed include the references listed in Table 8-1. Additional SOPs may be adapted from other sources or generated in-house as project needs require.

8.1 Project/Contract Review

To ensure that client commitments are met, a review of project documentation and contracts is performed. These documents are reviewed for technical, QC and business requirements to determine Katahdin's ability to meet them. The review process may involve the Sales & Marketing Department, Project Managers, Department Managers, Group Supervisors the Quality Assurance Officer, and/or the Katahdin President. For many client projects the specific requirements may not necessarily "fit" into a standard laboratory service or product. Katahdin is committed to working with clients to customize services as needed. This may involve modification of a method, reporting limit or report format.

The review of potential projects and bids must involve a complete review and understanding of all reporting levels, precision and accuracy (P&A) requirements, corrective actions and compound lists. Refer to the current revision of Katahdin SOP QA-810, Project Management: Review and Communication of Client Bids, Contracts and Project Specific Information & Service to the Client for further details.
Incoming projects may require the work be performed in accordance with one of the following:

- Client specific Quality Assuance Project Plan (QAPP)
- Client specific Minimum Standards
- The Department of Defense Quality System Manual (DoDQSM)
- State specific requirements

For projects that are to be performed in accordance with any of the above, the laboratory limits and P&A data must be compared to the appropriate documents. In all cases, any discrepancies must be noted in the bid specifications or in the quote so that the client can make informed decisions.

In many cases, our clients will approve the use of laboratory limits and P&A, in place of other QAPP limits. It is imperative to always be sure that the client is aware of any requirements that the laboratory cannot meet before acceptance or reward of a project.

8.2 Analytical Methods

Numerous sources of information are available to offer guidance in analytical methods. Selection of the appropriate method is dependent upon data usage and/or regulatory, client or program requirements. Table 8-1 describes the analytical references routinely used by Katahdin Analytical Services. Katahdin Analytical Services may modify existing methods based on the following considerations: 1) in order to meet project specific objectives; 2) in order to incorporate modifications or improvements in analytical technology; 3) in order to comply with changing regulations and requirements; 4) in order to address unusual matrices not covered in available methods.

Katahdin Analytical Services will make every effort to disclose to its clients any instances in which modified methods are being used in the analysis of samples.

8.3 Method Validation

The laboratory demonstrates its capability of performing an analytical method through method validation. Please refer to the current revision of Katahdin SOP QA-807, Method Performance / Precision and Accuracy Requirements, for details on specific method validation. When non-promulgated methods (i.e., methods other than EPA, APHA, ASTM, AOAC, etc.) are required for specific projects or analytes of interest, or when the laboratory develops a method, the laboratory establishes the validity of the method prior to applying it to client samples. Method validity is established by meeting specified criteria for precision and accuracy or ascertaining the precision and accuracy, which is achievable.

8.4 Method Revisions

Revisions to existing methods are handled in the same manner as a new method. After review of the published method, Katahdin determines whether or not to incorporate the new version as a product offered by the laboratory. A number of issues must be considered in making this decision. The following questions may be asked:

- Do we have the resources to perform this method?

- Can we easily obtain the resources to perform this method?
- Is this method promulgated?
- Does this method replace an existing promulgated method?
- Is the laboratory getting requests for this method?
- How long will it take to bring the method on-line?

Sales and Marketing Representatives, Project Managers, QAO, department managers, group supervisors and/or the Katahdin President may be consulted in deciding whether or not to adopt a revision of a method. Once the decision is made to adopt a method revision, a new product code is initiated. The SOP is written or revised, and training is initiated.

For revisions, which replace an existing Katahdin product, training may be documented on a Katahdin Retraining form. For revisions that are new to Katahdin, training is initiated as described in Katahdin SOP CA-805, Laboratory Technical Personnel Training. In both cases, Initial Demonstration of Method Proficiency shall be documented.

It is the responsibility of the QAO and the Department Managers to inform MIS personnel, Project Managers, Sales and Marketing Representatives and analysts of any changes in methodology. MIS personnel will either retire obsolete product codes (i.e. obsolete methods) and/or add additional product codes (i.e. new versions of existing methods) dependent upon each individual case. Project Managers and Sales will begin to sell the new product code. It is very important to ascertain which version of the method that clients are requesting. When clients request delisted methods, it is Katahdin's policy to inform them of this and the correct method that they should be requesting.

8.5 Method Detection Limits, Limits of Detection, and Limits of Quantitation

Method detection limit (MDL) studies, Limits of Detection (LOD) verifications and Limits of Quantitation (LOQ) verifications are performed in accoradnace with specific state, federal, method or program requirements. Refer to Katahdin Analytical Services, Inc. SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

Method detection limits are determined at Katahdin Analytical Services using replicate spiked analyte-free water samples or an appropriate soil matrix, if available. A minimum of seven replicates of a sample spiked with the analyte of interest is processed through the entire analytical method. The concentration of the detection limit sample should be between one and five times the anticipated detection limit.

The laboratory calculates the detection limit as the student's t (n-1, $1-\alpha = 0.99$) times the standard deviation (n-1) of the replicate spiked sample measurements. Refer to 40 CFR Part 136, Appendix B for further discussion.

Detailed procedures and acceptance criteria for MDL studies and LOD/LOQ verifications are described in the current revision of Katahdin Analytical Services SOP SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications. Current MDLs are filed in the QA Department.

IT IS IMPERATIVE TO NOTE THAT METHOD DETECTION LIMITS ARE HIGHLY MATRIX DEPENDENT. LIMITS DETERMINED BY KATAHDIN ANALYTICAL SERVICES MAY NOT BE ACHIEVABLE IN ALL MATRICES.

8.6 Compliance

8.6.1 Definition - Compliance is the proper execution of recognized, documented procedures that are either approved or required. Adherence to these procedures is required in order to provide data products acceptable to a regulatory body of competent jurisdiction in a specific regulatory context. Compliance is separate from, but not inconsistent with, technical scientific quality. Katahdin Analytical Services understands that the expectations of our clients commonly include the assumption that data and reports will satisfy a regulatory purpose and will be found acceptable *and compliant* with regulatory requirements for the performance of tests and generation of data.

8.6.2 Understanding the Regulatory Framework - Compliance is not likely to be achieved in the absence of an understanding of the regulatory framework. Katahdin Analytical Services will attempt to ascertain, prior to the initiation of a project, what regulatory jurisdiction (USEPA, state, etc.) pertains to a project; within the regulatory jurisdiction, what body of regulation is meant to be satisfied (RCRA, SDWA, NPDES, CERCLA, MCP, etc.); and finally, within this context, what protocols/programs are required/expected (DoD, NELAC, CLP, AFCEE, NFESC, etc.). Katahdin Analytical Services will work with its clients to come to a mutual understanding of all requirements, based on our best understanding of the information available.

8.6.3 Commitment - Clients may, but often do not, fully understand their compliance needs. Clients may sometimes fail to communicate their compliance requirements to Katahdin Analytical Services. Nevertheless, Katahdin Analytical Services, in defining quality as in 8.6.1 above, accepts some responsibility for compliance.

Katahdin Analytical Services makes the following commitments to its clients:

- Katahdin will proactively attempt to identify, understand, and execute the regulatory requirements for compliance.

- Katahdin will identify and disclose to clients instances of non-compliance in a forthright fashion.

8.6.4 Resolving Compliance Contradictions and Hierarchies - It is a common occurrence that multiple regulatory jurisdictions overlap in a specific case. This causes uncertainty or even contradictions to arise in a work plan. Katahdin Analytical Services will make every effort to detect such inconsistencies, and will

communicate them to clients so that the client can make an informed decision regarding execution of the project. Similarly, methods and protocols will often be prescribed in a scope of work or QAPP and may not achieve stated or implied data quality objectives (DQOs) or which are in conflict with the regulatory requirements. Katahdin Analytical Services will attempt to detect these inconsistencies, and upon detection, disclose them to our client. Katahdin Analytical Services voluntarily accepts a responsibility to provide advice to clients; however, **the primary responsibility for this issue remains with the client.**

8.6.5 Disclosure of Noncompliance - As stated previously, it is Katahdin Analytical Services' policy to disclose in a forthright manner any detected noncompliance that may affect the usability of data produced by Katahdin Analytical Services. It is not within our expertise to predict the manner in which a specific regulator or regulatory body will interpret the rules governing analysis; Katahdin Analytical Services is unable to guarantee compliance. It is Katahdin Analytical Services' policy that our responsibility begins with a bona fide and competent attempt to evaluate potential compliance issues and ends with disclosure of any findings that may be useful to our client in their making the final judgment.

TABLE 8-1

ANALYTICAL PROTOCOLS

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule, Federal Register, 40 CFR Parts 122, 136, 141, 143, 430, 455, & 465, March 12, 2007.

- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846. 3rd edition, 1986, Final Updates I(7/92), II(9/94), IIA(8/93), IIB(1/95), III(12/96), IIIA(4/98), and IIIB(11/04), IV(02/07), V(07/14) and “on-line methods”, Office of Solid Waste and Emergency Response, U.S. EPA.

- "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, 1979 Revised 1983, U.S. EPA.

- “Methods for the Determination of Inorganic Substances in Environmental Samples”, EPA 600/R-93/100, August 1993.

- "Standard Methods for the Examination of Water and Wastewater", 15th, 16th, 17th, 18th, 19th, 20th, 21st and 22nd editions, 1980, 1985, 1989, 1992, 1995, 1999 2005, 2012. APHA-AWWA-WPCF.

- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society for Testing and Materials, current revisions.

- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society for Testing and Materials, current revisions.

- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.

- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati (September 1986, & Supplements I, II, & III).

- New York State Department of Environmental Conservation. Analytical Services Protocol, June, 2000.

- Official Methods of Analysis of AOAC International, 17th Edition

- FDA Bacteriological Analytical Manual (BAM) Edition 8, Revision A, 1998

TABLE 8-2

ANALYTICAL CAPABILITIES

ORGANIC ANALYSES		
	METHODS	
ANALYTE	**WATER**	**SOIL**
Alcohols by GC-FID	SW 8015 modified	SW 8015 modified
Chlorinated Phenoxy Herbicides	SW 8151	SW 8151
Diesel Range Organics (DRO)	SW 8015 modified	SW 8015 mod/8100
EDB & DBCP by GC	EPA 504.1, SW 8011	
Explosives	SW 8330	SW 8330
Formaldehyde	SW 8015 modified	
Gas Range Organics (GRO)	SW 8015 modified	SW 8015 modified
Drinking Water Volatile Organics	EPA 524.2	
Volatile Organics	EPA 624, SW 8260	SW 8260
Volatile Organics - Air		TO-15 (air)
Volatile Organics - Air		MA-DEP APH (air)
Glycols	SW 8015 modified	
GRO/DRO - Maine HETL	HETL 4.2.17, 4.1.25	HETL4.2.17, 4.1.25
GRO/DRO - Massachusetts Risk Assessment	MA DEP VPH/EPH	MA DEP VPH/EPH
Low Level Select Volatiles	8260 SIM	8260 SIM
Methane, Ethane & Ethene	RSK 175	
Oil & Grease (Gravimetric)	EPA 1664, SW 9070	SW 9071 modified
Organochlorine Pesticides & PCBs	EPA 608, SW 8081/8082	SW 8081/8082
Organochlorine Pesticides & PCBs (Tissue)		SW 8081/8082
PCB Congeners	SW 8082 modified, SOP CA-334	SW 8082 modified, SOP CA-334
PCBs in oils		EPA 3580/SW 8082
PCBs wipes/filters		EPA 3580/SW 8082
Polynuclear Aromatics (PAH)	EPA 625, SW 8270	SW 8270
Polynuclear Aromatics (PAH) & select Semivolatile Organics	SW 8270 SIM	SW 8270 SIM
Semivolatile Organics - Extractables	EPA 625, SW 8270	SW 8270
SPLP Extraction	SW 1312	SW 1312
Total Petroleum Hydrocarbons	FLO-PRO, TX1005, CT-ETPH	FLO-PRO, TX 1005, CT-ETPH
Grain Size (sieve & hydrometer)		ASTM D422

METALS ANALYSES		
ANALYTE	**REQUIRED DIGEST**	**METHODS**
ICP ANALYSES		
Aluminum	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Antimony	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Arsenic	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Barium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Beryllium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020

TABLE 8-2

ANALYTICAL CAPABILITIES

ANALYTE	REQUIRED DIGEST	METHODS
ICP ANALYSES		
Boron	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Cadmium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Calcium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Chromium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Cobalt	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Copper	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Iron	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Lead	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Magnesium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Manganese	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Molybdenum	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Nickel	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Potassium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Selenium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Silica as SiO2	EPA 200.7	EPA 200.7
Silver	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Sodium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Strontium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Thallium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Tin	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Titanium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Vanadium	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
Uranium	EPA 200.8	EPA 200.8
Zinc	EPA 200.7, 200.8, SW 3010, 3050	EPA 200.7, 200.8, SW 6010, 6020
COLD VAPOR ATOMIC ABSORPTION (CVAA)		
Mercury	EPA 245.1, SW 7470, 7471	EPA 245.1, SW 7470, 7471
Mercury (Tissue)	SW 7470, 7471	SW 7470, 7471
COLD VAPOR ATOMIC FLUORESCENCE (CVAF)		
Mercury - Low Level	EPA 1631	EPA 1631
CALCULATION		
Hardness	EPA 200.7/SM 2340B, SOP CA-628	

INORGANIC ANALYSES	
ANALYTE	**METHODS**
Acidity	EPA 305.1, SM2310B
Acid Volatile Sulfides	EPA 376.3
Alkalinity	EPA 310.1, SM 2320B
Bicarbonate & Carbonate	SM 4500-CO2
Biochemical Oxygen Demand-total	EPA 405.1, SM5210B

TABLE 8-2

ANALYTICAL CAPABILITIES

INORGANIC ANALYSES	
ANALYTE	**METHODS**
Biochemical Oxygen Demand-carbonaceous	SM5210B
Bromide	EPA 300.0, SW 9056
Carbon - Total Inorganic (water)	EPA 415.1 modified, SW 9060 modified, SM5310B
Carbon - Total Organic (water)	EPA 415.1, SW 9060 modified, SM5310B
Carbon - Total (soil)	modified Lloyd Kahn, Walkley Black
Cation Exchange Capacity	SW 9081
Chemical Oxygen Demand	EPA 410.4, HACH 8000
Chloride	EPA 325.2, EPA 300.0, SW 9251, SM 4500 Cl E, SW 9056
Chromium - Hexavalent	SW 7196, SM3500Cr-D
Color	EPA 110.2, SM 2120B
Cyanide - Total	SM 4500CN⁻C Mod, 335.4, SW 9012 Mod
Cyanide - Total & Amenable	SM 4500CN⁻ -C/G Mod, EPA 335.1 Mod, SW 9012 Mod
Ferrous Iron	SM 3500Fe D
Fluoride	EPA 340.2, SM 4500F⁻B/C
Free Liquid Content (Paint Filter Test)	SW 9095
Hardness (Titrimetric)	EPA 130.2, SM 2340C
Lime Equivalence	EPA 310.1 modified
Nitrogen - Ammonia	EPA 350.1, EPA 350.2, SM 4500NH3 B & H
Nitrogen - Nitrate plus Nitrite (combined)	EPA 300.0, 353.2, SM4500-NO3 F, SW 9056
Nitrogen - Nitrate	EPA 300.0, 353.2, SM4500-NO3 F, SW 9056
Nitrogen - Nitrite	EPA 300.0, 353.2, SM4500-NO3 F, SW 9056
Nitrogen - Total Kjeldahl	EPA 351.2
Nitrogen - Total Organic - TKN minus NH_3	EPA 351.2/350.1
Odor	SM2150 modified
Paint Filter	SW 9095
pH	EPA 150.1, SW 9040, 9045, SM 4500H B
Phenols - Total	EPA 420.1 SW 9065
Phosphorus - Total	EPA 365.4, SM 4500P-E
Phosphorus - Ortho	EPA 365.2, EPA 365.1, SM 4500-P-E, EPA 300.0, SW 9056
Practical Salinity	SM 2520B
Residual Chlorine	SM4500 Cl G
Sample Prep - Compositing	
Sample Prep - Filtration	SM 3030B
Solids - Total	EPA 160.3, SM2540 B
Solids - Suspended	EPA 160.2, SM2540 D
Solids - Dissolved	EPA 160.1, SM2540 C
Solids - Volatile Aqueous	EPA 160.4, SM2540 E
Solids - Suspended Volatile	EPA 160.2, 160.4

TABLE 8-2

ANALYTICAL CAPABILITIES

INORGANIC ANALYSES	
ANALYTE	**METHODS**
Solids - Settleable	EPA 160.5, SM2540 F
Specific Conductance	EPA 120.1, SM 2510B
Specific Gravity	EPA/COE 1981
SPLP Extraction	SW 1312
Sulfate	EPA 375.4, 300.0, SW 9056, 9038, SM 4500SO4 E, ASTM D516-02
Sulfide	EPA 376.1, SW 9034
Sulfite	EPA 377.1, SM4500-SO3 B
Surfactants - MBAS	SM5540C
Tannins and Lignins (as tannic acid)	SM 5550B
Turbidity	EPA 180.1, SM 2130B
Volatile Fatty Acids	SOP CA-776
% Moisture	SM 2540G

TCLP ANALYSES	
ANALYTE	**METHODS**
Extraction for Volatiles (ZHE)	SW 1311
Extraction for Metals, Semivolatiles,Pesticides & Herbicides	SW 1311
Volatiles - TCLP List	
Benzene	SW 8260
Carbon Tetrachloride	SW 8260
Chlorobenzene	SW 8260
Chloroform	SW 8260
1,2-Dichloroethane	SW 8260
1,1-Dichloroethylene	SW 8260
Methyl Ethyl Ketone	SW 8260
Tetrachloroethylene	SW 8260
Trichloroethylene	SW 8260
Vinyl Chloride	SW 8260
Semi-Volatiles - TCLP LIST	
Pyridine	SW 8270
M-Cresol (3-Methyl Phenol)	SW 8270
O-Cresol (2-Methyl Phenol)	SW 8270
P-Cresol (4-Methyl Phenol)	SW 8270
1,4-Dichlorobenzene	SW 8270
2,4-Dinitrotoluene	SW 8270
Hexachloro-1,3-Butadiene	SW 8270
Hexachlorobenzene	SW 8270
Hexachloroethane	SW 8270
Nitrobenzene	SW 8270
Pentachlorophenol	SW 8270

TABLE 8-2

ANALYTICAL CAPABILITIES

Semi-Volatiles - TCLP LIST	
2,4,5-Trichlorophenol	SW 8270
2,4,6-Trichlorophenol	SW 8270
Pesticides - TCLP LIST	
Chlordane	SW 8081
Endrin	SW 8081
Heptachlor	SW 8081
Heptachlor Epoxide	SW 8081
Lindane	SW 8081
Methoxychlor	SW 8081
Toxaphene	SW 8081
Herbicides - TCLP LIST	
2,4-Dichlorophenoxyacetic Acid	SW 8151
2,4-5-Trichlorophenoxypropionic Acid	SW 8151
Metals - TCLP LIST	
Arsenic	SW 6010
Barium	SW 6010
Cadmium	SW 6010
Chromium	SW 6010
Lead	SW 6010
Mercury	SW 6010
Selenium	SW 6010
Silver	SW 6010

WASTE CHARACTERIZATION	
ANALYTE	**METHODS**
Corrosivity - pH	SW 9040, 9045
Reactivity – Releasable Cyanide	SW 7.3.3.2
- Releasable Sulfide	SW 7.3.4.2
Ignitablitity	SW 1010

APPENDIX NINE ANALYSES	
Analyte	**Methods**
Volatile Organics (extended list 19 Compounds)	SW 8260, 5030, 5035
Acid and Base Neutrals (extended list 48 Compounds)	SW 3510, 3520, 3540, 3545, 3550, 8270
Pesticides and PCBs	SW 3510, 3520, 3540, 3545, 3550, 8081, 8082
Herbicides	SW 8151
Total Cyanide	SW 9012
pH	EPA 150.1, SW 9040/9045
Metals: Arsenic	SW 6010, 6020

TABLE 8-2

ANALYTICAL CAPABILITIES

APPENDIX NINE ANALYSES	
Analyte	**Methods**
Antimony	SW 6010, 6020
Barium	SW 6010, 6020
Beryllium	SW 6010, 6020
Cadmium	SW 6010, 6020
Cobalt	SW 6010, 6020
Chromium	SW 6010, 6020
Copper	SW 6010, 6020
Mercury	SW 7470, 7471
Nickel	SW 6010, 6020
Lead	SW 6010, 6020
Selenium	SW 6010, 6020
Silver	SW 6010, 6020
Tin	SW 6010, 6020
Thallium	SW 6010, 6020
Vanadium	SW 6010, 6020
Zinc	SW 6010, 6020
Metals Digestion	SW 3010, 3020, 7470, 7471

Appendix IX is derived from Appendix VIII Hazardous Constituents. These parameter lists are intended to apply to groundwater monitoring at hazardous waste storage and disposal sites and are applied to uncontrolled site investigations and remediations.

MICROBIOLOGY	
ANALYSIS	**METHOD**
Total Coliform	SM9222B
Fecal Coliform	SM9222D, Colilert 18 w/Quantitray
Fecal Coliform	m-tec modified
Total Coliform and E-coli	SM9223B, Colitag
E-coli	SM9213D
E-coli	SM9223B Colilert w/ Quantitray
Heterotrophic Plate Count	SM9215B, SIMPlate

FOOD ANALYSES	
TEST	**METHODS**
Aerobic Plate Count (APC)	AOAC 986.33/990.12
Total Colifroms	AOAC 991.14
e-Coli	AOAC 991.14
Listeria Species - Vidas	AOAC RIN 100501
Listeria Species/Listeria Monocytogenes - Biorad	AOAC 030406

TABLE 8-2

ANALYTICAL CAPABILITIES

FOOD ANALYSES	
TEST	**METHODS**
Yeast & Mold	AOAC 997.02
Staphylococcus	AOAC 2003.07
Salmonella	AOAC 2001.09
Shigella	MPN Screen
Water Phase Salt	AOAC 937.09
Water Activity	AOAC 978.18
Clostridium Botulinum	AOAC 977.30 Mod.
Clostridium Perfringens	Screen AOAC 974.38 Mod.
Lactobacillus	SM 9215B Mod - Anaerobic
Histamine	Screen
Deoxynivalenol (DON)	USDA GIPSA 2002-106
Nitrofuran	ELISA by Ridascreen
Chloramphenical	ELISA by Ridascreen
Malachite Green	ELISA
Gentian Violet	ELISA
Bacillus Cereus	AOAC 980.31
Trans Fatty Acids	CLA by HPLC
Caffeine Content	AOAC 980.14 Mod.
BRIX	ICUMSA SPS-3
Specific Gravity	AOAC 955.37 Mod.
Campylobactor	BAM 8th Edition
Vibrio - Parahaemolyticus	BAM 8th Edition Chapter 9
Vibrio - Vulnificus	BAM 8th Edition Chapter 9
Shelf Life Studies and Challenge Studies	Various
Lactic Acid Bacteria	AOAC 990.12 anaerobic
Anaerobic Bacteria	SM9215 Mod - Anaerobic
Various Allergens – soybean, hazelnut, almond, milk, peanut, shellfish	Alert

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All analytical data generated within Katahdin Analytical Services laboratories undergo a well-defined, well-documented multi-tier review process before being reported to the client. (Refer to Figure 9-1)

Each department shall have written procedures incorporated into the working SOPs for data validation which incorporate the quality assurance goals of traceability, accountability, completeness, precision and accuracy.

No written reports shall be issued which have not undergone the data validation process. Refer to the current revision of Katahdin Analytical Services SOP SD-904, Data Reduction, Review and Reporting, for more information.

9.1 Data Reduction

Primary analytical data, otherwise known as "raw data", may be manually generated or captured in electronic format. When raw data are manually generated, they are recorded either in bound logbooks with numbered pages or on preprinted forms. Records of analysis indicate the method used, raw data, calculations, and final results. Entries are made in black ink and are initialed and dated by the individual making the entry. It is acceptable to initial and date once for an entire page. Errors are corrected by drawing a single line through the entry; corrections are initialed and dated by the individual making the change. An explanation for the change must be indicated on the raw data when possible. If space is limited, errors may be coded using the descriptive codes listed in Figure 9-2. Raw data may not be obscured in any way. The use of white-out is prohibited on all raw data, including instrumental hardcopy.

The analyst who completes the analysis assembles all relevant raw data and results together with chromatograms, strip chart recordings, instrument settings and other information essential to data interpretation. For data that are reduced by manual calculations, the calculations are documented in a laboratory notebook or in an Excel spreadsheet. All raw results are manually entered or imported into the Laboratory Information Management System (KIMS). Electronic batch sheets and all pertinent reporting forms (refer to Figure 9-3) are generated.

9.2 Data Validation

Data validation is the internal process of review by which data are shown to be valid as evidenced by the soundness of the analytical system and successful meeting of the DQOs (not to be confused with data validation by an outside independent source). In this process, the Laboratory makes no judgment as to the usability of the data by the end-user except with respect to the methodology applied.

Analysts performing an analysis and subsequent data reduction have the primary responsibility for the quality of the data produced. The primary analyst initiates the data validation process by reviewing and accepting the data, provided the associated quality control criteria have been met for the samples being reported. All reduced data must be

evaluated using the QA Acceptance Criteria tables found within individual analytical method SOPs (please refer to Appendix B of Section 11 of this manual for examples of these tables. These tables give acceptance criteria and corrective actions for criteria that are not met. Data review checklists may be used to document the data validation process (these checklists, if applicable, may be found within individual method SOPs).

Depending on the corrective action taken, data may or may not be flagged with the appropriate qualifier. Quality control excursions and any analytical discrepancies and/or matrix-related problems discovered during sample preparation or analysis, are documented in the Report of Analysis through the use of data flags.

The initial reviewer drafts any narrative comments. The narrative briefly describes any issues or discrepancies relating to the condition of the samples upon receipt, sample holding time performance, instrument calibration information, or quality control results. Any corrective actions pertaining to the samples are also addressed. Any items noted during technical and QC review are included in the narrative if deemed to impact the quality of the data. Narratives are always required for all level 3 and level 4 reports (see Figure 9-3, Levels of Data Deliverables) and only when necessary to document an issue for level 1 and level 2 reports.

The completed data package is then sent to the Department Manager or designated senior technical data reviewer. The Department Manager or designated data reviewer provides a technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. The review includes a thorough evaluation of manual calculations. For data that are reduced via computer, calculations are checked by the analyst (or designee) assigned to this task at a frequency designed to assure that the final data generation is valid. This data validation step is documented by the analyst's initials on the hardcopy of the raw data. The narrative is also checked during this stage of review.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise dating and initialing the data.

All analytical data segments and other relevant paperwork pertaining to a particular Katahdin Analytical Services data report are channeled to the Data Management Department for assembly into the final report format and compilation of the analytical narrative.

9.3 Data Reporting

The sample results are tabulated by test method and department. Katahdin Analytical Services number, client identification, method number, and dates of sample preparation and analysis are presented along with the concentrations detected for each parameter analyzed and corresponding reporting limits and units. Katahdin Analytical Services is also able to provide alternate report formats if requested and additional deliverables (e.g., quality control reports, electronic diskette deliverable). Please refer to

Figure 9-3 for a list of Katahdin data deliverables. All reports are processed through the data validation and reporting steps described in Sections 9.2 and 9.3.

Some states or programs may have more strict reporting requirements. Some may require a copy of the certification parameter list and certification ID for work originating in their state, i.e. Massachusetts. For drinking water samples, a copy of the MCL from the state where the samples originated must be included in the report. It is Katahdin's policy to indicate on the report when drinking water parameters are above the MCL. Some states (MA) also require verbal notification to the client within 24 hours when MCLs are exceeded.

The Data Management Department assembles all data from a sample set, generates the final report, checks for completion, and checks that all client requests have been met.

The final review and signing of reports is completed by a qualified individual as necessary. The final reviewer may be the Quality Assurance Officer or their designee.

The final reviewer may examine the report for method appropriateness, detection limits, completeness and accuracy and whether or not QC criteria were satisfied, as deemed necessary. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution. The final reviewer signs the cover letter, authorization line within the report and/or the narrative, indicating acceptance of the report prior to its release to the client.

When an amendment to a report is required after the issue of the report, a separate document or electronic data deliverable (EDD) is provided. The revised report must be clearly identified as reissued. A "reissued" stamp is used in the lower right-hand corner of the report page. Reissued reports may involve the entire report or just specific pages. In both cases, the pages shall be stamped as "reissued", either manually, or through pagination on the copier/scanner. A cover letter indicating the date of revision, the revised pages, and the reason for revision must accompany every revised report. The original version of the report must be kept intact and the revisions and cover letter included in the work order project file.

Refer to the current revision of Katahdin Analytical Services SOP's SD-914, Assembly of Level I and Level II Reports and SD-915, Assembly of Level III and Level IV Reports, for more information.

9.4 Subcontractor Reports

Subcontracted data must be clearly identified as such. The name of the subcontractor laboratory must be documented in the report. All subcontracted sample data reports are reviewed to ensure all analyses requested have been received by Katahdin Analytical Services. The subcontracted data report may be appended in whole to Katahdin Analytical Services' analytical data report. The final report format is determined through communication between the Katahdin Analytical Services Project

Manager, Data Management/Reporting Group, and the client. Please refer to Katahdin SOP, SD-900 (current revision), Subcontracting Analyses, for further details.

9.5 Electronic Data Deliverables

Electronic Data Deliverables (EDDs) are routinely offered as part of Katahdin's services. Katahdin offers a variety of standard formats including Excel, Dbase, GISKEY, EQUIS and Text files. Katahdin will work exclusively with clients to produce client-specific EDD formats. EDD specifications must be submitted to the Management Information Systems (MIS) Department by Katahdin project managers for review. Katahdin management and the MIS department will determine whether the EDD format can be provided.

Data Management personnel and MIS are responsible for creating any EDDs requested by the client. Once an EDD is created, at least 10% (typically 50%) is reviewed to ensure its accuracy and completeness. Any hand entered data must be checked by a secondary reviewer.

9.6 Data Archive

Once the final report has been approved and signed it is submitted to the client via email, upload or hardcopy mail. Hardcopies of the supporting data and review checklists associated with each report are stored in the data archive under the control of the Office Manager. Additionally, all raw data and supporting documents, that are not included with the client report, are scanned and saved as a pdf file on the server along with the electronic copies of the preliminary and final reports. The purpose of the data archive is to ensure the continued integrity of all documentation generated in support of laboratory analyses.

Once the scanned reports and supporting documentation have been saved to CD ROM (twice), and confirmed, any hardcopy data is disposed of. CD ROM records are maintained indefinitely. All other records including bound logbooks and QA/QC records are saved on-site (either an electronic copy or hard copy) for a minimum of five years. Any electronic data retained by the laboratory is saved on 2 CD's. One is stored on site for laboratory use and one is stored off site. Some states, clients or specific program regulations require longer retention times than five years. These records will be noted with specific storage instructions. Records maintained off-site are protected against fire, theft, loss, deterioration, and vermin. These archived records may only be retrieved by designated personnel. A member of management may designate an employee to retrieve data files from the archive storage facility.

9.7 Quality Assurance Review

The Quality Assurance Officer or designee shall review at a minimum 10% of all data packages for technical completeness and accuracy. This review is part of the oversight program and does not have to be completed in "real time". Documentation of this review will be recorded in a designated logbook.

FIGURE 9-1.

MULTI-TIER REVIEW PROCESS

DATA FLOW CHART

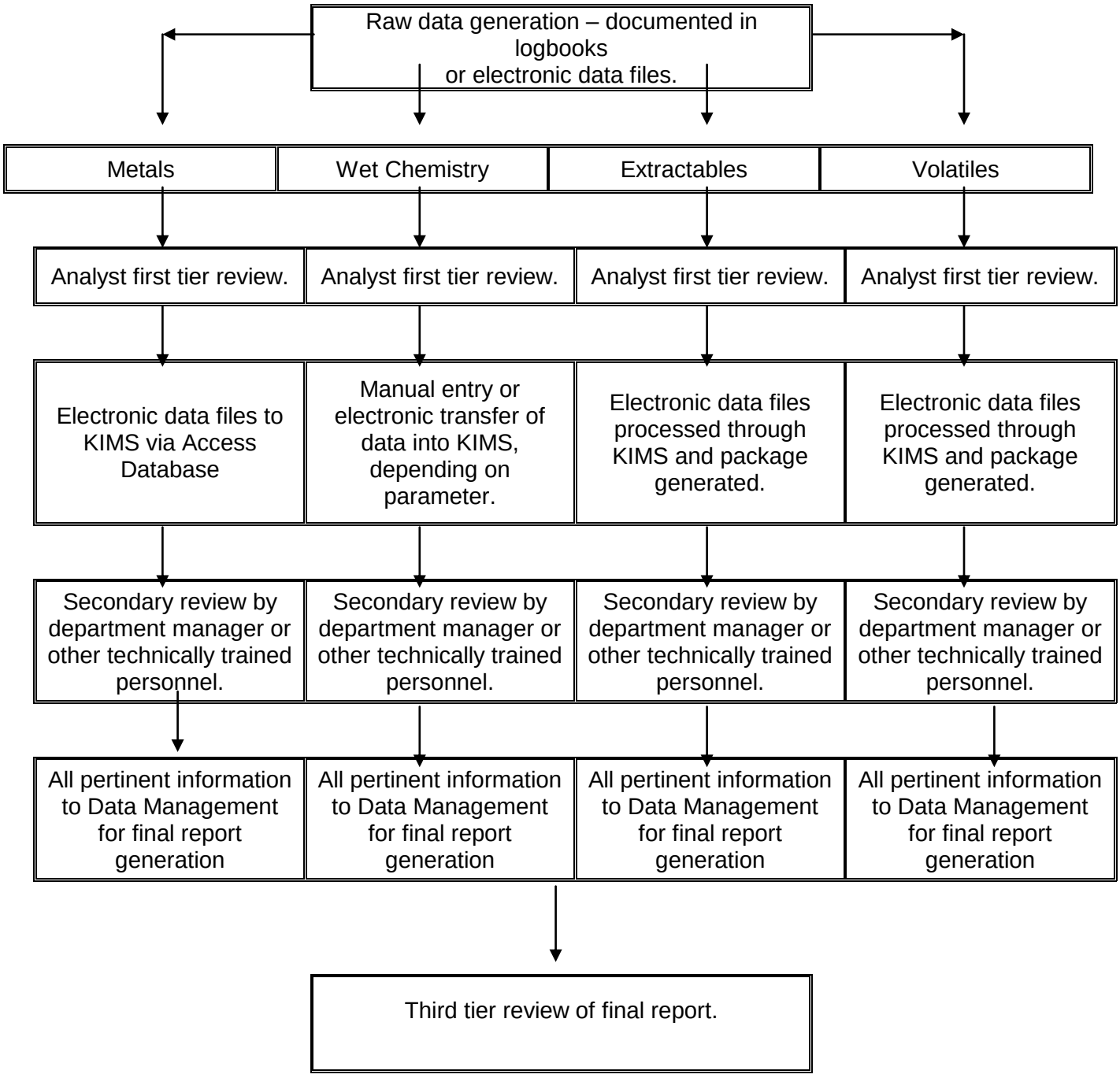

FIGURE 9-2

Error Codes

1	(or E1)	Misspelled
2	(or E2)	Mathematical Error
3	(or E3)	Wrong Entry
4	(or E4)	Transposition or Sequencing Error
5	(or E5)	Transcription Change (copy error)
6	(or E6)	Procedural Change
7	(or E7)	Wrong Conclusion
8	(or E8)	Illegible Entry
9	(or E9)	Unnecessary Entry
10	(or E10)	Footnoted Explanation
11	(or E11)	Additional Comment
12	(or E12)	Instrumentation Error/Failure

FIGURE 9-3

LEVELS OF DATA DELIVERABLES

Deliverable	Data Reporting Level								
	I				II(+)	III	IV		
	FM	HO	DW Comp	STD	STD	STD	STD	DoD	CLP
Footnotes and Definitions Pages		√	√	√	√	√	√	√	√
Narrative (if needed)	√	√	√	√	√	√	√	√	√
Report of Analysis (Form 1 or equiv – TICs optional)	√	√	√	√	√	√	√	√	√
External chains of custody	√	√	√	√	√	√	√	√	√
Drinking Water MCL Information		√	√						
State Forms			√						
E-mail Client Correspondence Regarding Data Issues						√	√	√	√
Blank Results (Org Form 1/Ino Form 3 or equiv)				√*	√	√	√	√	√
Surrogate Recoveries (Org Form 2 or equiv)						√	√	√	√
Laboratory Control Sample Recovery (Org Form 3/Ino Form 7 or equiv)					√	√	√	√	√
Dup/MS/MSD if performed on client sample (Org Form 3/Ino Form SA&6 or equiv)					√(+)	√	√	√	√
Blank Summary (Org Form 4 or equiv)				√*	√	√	√	√	√
Tune Summary (Org Form 5 or equiv)						√	√	√	√
Initial Calibration (Org Form 6/Ino Form 2A&3 or equiv)						√	√	√	√
Continuing Calibration (Org Form 7/Ino Form 2A&3 or equiv)						√	√	√	√
Internal Standard Area Summary (Org Form 8 or equiv)						√	√	√	√
Run Logs						√	√	√	√
Sample Preparation logs						√	√	√	√
Raw data							√	√	√
Before & After Manual Integration Raw Data								√	√

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FIGURE 9-3, CONT"D

LEVELS OF DATA DELIVERABLES

Deliverable	Data Reporting Level								
	I				II(*)	III	IV		
	FM	HO	DW	STD	STD	STD	STD	DoD	CLP
Florisil cartridge check (Org Form 9A or equiv) - (CLP only)									√
GPC calibration if performed (Org Form 9B or equiv)							√	√	√
Dual column ID summary (Pesticide Form 10 or equiv) - (if requested)						√	√	√	√
Instrument Sensitivity Check (Ino Form 2B or equiv)						√	√	√	√
Interference Check Sample (Ino Form 4 or equiv)						√	√	√	√
Post Digest Spike Sample – if performed (Ino Form 5B)						√	√	√	√
Standard Addition Results Summary – if performed (Ino Form 8 or equiv)						√	√	√	√
ICP Serial Dilutions – if performed (Ino Form 9 or equiv)						√	√	√	√
IDLs (Ino Form 10 or equiv)						√	√	√	√
Interelement Correction Factors (Ino Form 11A&B or equiv)						√	√	√	√
ICP Linear Ranges (Ino Form 12 or equiv)						√	√	√	√
Standard Preparation logs - (if requested)						√	√	√	√
Internal chains of custody - (if requested)						√	√	√	√

/ Data included in reporting level.
* Blank results provided for organics data.
+ Dup/MS/MSD provided if requested on client sample.
FM Food Microbiology Report
HO Homeowner Report
DW Drinking Water Compliance Report
STD Standard Report
DoD Report in accordance with the DoD QSM

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10.0 DOCUMENTATION & RECORDS MANAGEMENT

Records are the means by which an organization documents its operations and activities. They are an integral part of the Quality Assurance program since they provide documented evidence for program functionality and necessary information for performance evaluation and quality assurance audits. All information related to the administration and operations of the laboratory and quality assurance practices outlined in this manual shall be contained in records. This shall include, but is not limited to, standard operating procedures, results of instrument calibrations, analysis of quality control samples, analysis of samples, sample custody and disposal, preparation of standards, COC documentation, analytical reports, corrective action reports, audits and inspections. Refer to Table 10-2 for a more complete list of records.

All laboratory records are the property of Katahdin Analytical and shall not be removed from the premises without the permission of a member of management. All records are confidential and must be handled in that manner. Unauthorized distribution, changes, loss or destruction of records can be grounds for dismissal from the laboratory. All records are filed, maintained, stored, archived and disposed of as described in this SOP. All records shall only be made available to the original client or to a third party designated by the client. Refer to the current revision of Katahdin SOP AD-004, Laboratory Facility Security and Confidentiality, for further information. All records shall be made available to regulators, auditors or accrediting authorities as required by regulation.

Records shall be stored to be readily retrievable in environments that prevent damage to the records. All records shall be stored to allow historical reconstruction of all laboratory activities that produced the analytical data. The historical reconstruction must be understood and must include information on receipt, chain-of-custody, preparation, analysis, corrective actions and client communication, report content and reissues. Stored electronic files shall be named to facilitate the retrieval of working and archived files.

10.1 General Recordkeeping

- All documentation must be accurate, legible, complete and recorded in a timely manner using indelible ink. No measurements are to be filled in ahead of time even if the measurement is always the same. During the actual analysis it may be determined that a different quantity is needed.
- All data and/or results that are recorded and/or entered must be a true and accurate representation of the measured values and must be validly quantitated.
- If an error is made, a single line is used to cross out the incorrect entry. The original entry must remain readable. The correction must be initialed and dated and may be given an error code (see Figure 1) or explanation for the change. The use of white out is prohibited on all raw data, including instrumental hardcopy.
- If a change is made to a record, a single line is used to cross out the incorrect entry. The original entry must remain readable. The correction must be initialed and dated and may be given an error code (see Figure 1) or explanation for the change. The use of white out is prohibited on all raw data, including instrumental hardcopy. For electronic changes, all audit trails must be maintained.

- When blank space is left after all information has been recorded on a logbook page or on other forms of documentation, the blank space must be "Z'd" out. Draw a line in the shape of a "Z" starting at the top of the blank space and ending at the bottom of the blank space; initial and date the "Z" out.

- All blocks must be filled in on pre-printed forms. Header information must be complete. All columns, signatures and units of measure must be identified.

10.2 Standard Operating Procedures

Standard Operating Procedures (SOPs) are written for specific procedures or operations. Complex tasks of inspection, testing, calibration, monitoring, maintenance, data handling, and quality control as well as methods utilized in the laboratory are specified and documented by SOPs.

As a minimum requirement, each SOP must include a title, the purpose or application, list of materials or references, and detailed procedures. SOPs are reviewed and/or updated annually. If annual review is not possible for all SOP's, the QA Officer shall prioritize the SOP's for a timely review. Each has a Katahdin Analytical Services reference number and revision date at the upper right corner of each page. More detailed information regarding SOPs can be found in Section 12.0 and in the current revision of Katahdin Analytical Services SOP QA-800, Preparation of SOPs.

All personnel are required to follow SOPs when a specific operation or method is being utilized. It is the responsibility of the Department Managers/Group Supervisors to make sure that employees are aware of and follow the SOPs. Any suggestions for additional SOPs or changes to existing SOPs should be directed to the appropriate Department Manager or the QA Department.

10.3 QA Records

QA Records shall include, but are not necessarily limits to, SOPs (described above), the QA Manual, audits and responses, corrective action forms, training forms and demonstrations of capability, employee qualifications and/or transcripts, logbooks, PT results, internal COCs, a record of all employee initials and signatures responsible for signing laboratory records and MDL/IDL studies/verifications. All hardcopy current and past QA records are filed with the QA Officer for 1-3 years, depending on space. Hardcopy QA records are logged and filed off site after this for at least five years. QA records are maintained and backed up on the company server indefinitely.

10.4 Sample Tracking

Samples are tracked from the time they are received, through storage, preparation, analysis, and final disposition. More information on the following discussion can be found in the current revision of Katahdin Analytical Services SOP SD-902, Sample Receipt and Internal Control.

Proper sample identification must be established during sample collection. This information must be clearly and permanently written on a label and attached to the sample. In addition, a Chain-of-Custody must be initiated with the appropriate information recorded. Samples should also be properly preserved and stored.

Sample Management personnel verify the samples' integrity, as they are unpacked. The Katahdin Analytical Services Project Manager and/or the client are notified of samples that are received broken or have not been properly stored or preserved. The sample identification label must also be checked against Chain-of-Custody identification. Any discrepancies must be verified by the client or sampler. All these checks and any discrepancies or changes must be documented on the Sample Receipt Condition Report.

Sample Management Personnel assigns each group of samples, or job, a Work Order number and each sample within the job a sequential laboratory identification number, which is placed on the sample container. Samples are stored in the appropriate locations. A laboratory data file is initiated for the entire job.

Upon receipt the Sample Management Personnel also initiate an internal custody record for the sample set. These forms are used to document sample removal from and return to sample storage. The final disposition of a sample is documented on the hazardous waste disposal spreadsheet.

10.4.1 Chain-of-Custody

The Chain-of-Custody (COC) Form traces the possession of a sample from the time the sample is obtained in the field through receipt by and analysis in the laboratory. To initiate a Chain-of-Custody, the field sampler must fill in the appropriate information: Client or Project Name, Signature of Sampler, Sample Identification, Date and Time Sampled, Type of Sample, and Analysis Requested. After the sample is brought into the laboratory, sample integrity, preservation, and identification are checked. Any inconsistencies are noted in the sample management documentation. For samples accepted into the laboratory, the Sample Custodian verifies that the sample label IDs are consistent with the sample IDs provided on the Chain-of-Custody, signs in the space marked "Received for Laboratory" and records the date and time received.

10.4.2 Internal Sample Custody Record

The internal custody record is a mechanism for tracking samples from sample management (sample storage) to sample preparation and/or analysis and back to the appropriate storage location, unless the aliquot is depleted. When laboratory personnel remove samples from sample storage, the sample transfer is recorded electronically using bar codes. Upon returning any remaining sample, the individual electronically scans the bar code for the appropriate samples as returned. For analyses that have an immediate analytical holding time (48 hours or less) or for rapid turn around work, the sample custodian will enter this information into a Google docs internal web page that is constantly monitored in the lab. Upon

completion, the original internal custody records are archived by the QA Department.

10.5 Standards

Standards preparation is documented in the Katahdin Analytical Services laboratories in standards logbooks maintained by each laboratory section. All information needed to maintain proper traceability of standards is recorded in the appropriate standards logbook by the individual preparing the standard. More complete information regarding standards is provided in Section 7.1 and in the current revision of Katahdin Analytical Services SOP CA-106, Standard Preparation and Documentation.

10.6 Maintenance Logbooks

Maintenance logbooks are kept for each instrument. Each instrument has a unique maintenance logbook. In the logbook, an analyst records initial instrument setup, routine preventive maintenance, outside contractor services, instrumental malfunctions and repair performed, dates taken in and out of service, and resolutions. Instrument logs not only describe the instrument's history, but also can be helpful when troubleshooting. Alternatively, instrument runlogs may be used to document problems noted, routine maintenance performed and return to control. Refer to the current revision of Katahdin Analytical Services SOP CA-101, Equipment Maintenance, for more information.

10.7 Preparation Logbooks

All data pertinent to sample preparation shall be recorded by the laboratory staff in bound notebooks with numbered pages and/or in the Katahdin Information Management System (KIMS). During the sample preparation process, a preparation record shall be prepared for the project by the preparation analyst. It shall contain the following information:

- Sample identification numbers
- Date and time of preparation
- Method reference
- Analyst’s initials
- Preparation weights and/or volumes (initial and final)
- Reagent/solvents used including manufacturer and lot number
- Relevant blank
- Spike and surrogate data including the serial reference number
- Clean-up performed on sample extracts
- Instrumental analysis to be performed on each extract
- Notable observations

10.8 Run Logbooks/Instrument Logbooks

A run logbook (or computer-generated run sequence) or an instrument logbook is maintained for all instruments and/or analytical procedures. Each run log contains the entire sequence of samples, including the calibration curve. All data documented in the run log shall include reference to the person(s) who performed the analysis, the date of

analysis, the method used, the identification of the instrument, instrument operating conditions/parameters or an analytical method file to reference these and the acceptability of the results in the context of the QC system.

At the time of sample analysis, the laboratory identification number, amount injected or otherwise analyzed, any dilution of the original sample and/or extract and other relevant sample data shall be entered into the run log and/or instrument logbook, or if possible into the instrument header. All standard and reagent unique IDs must be included for traceability purposes.

All data relevant to the calculations should, where possible, be entered onto the instrument header including sample weight or volume, final volume, dilution, and spike level.

10.9 Raw Data

Raw data includes any information needed to reconstruct how analytical results are obtained. This includes standards logbooks, preparation logbooks and instrument runlogs as described above. Raw data also includes all instrument printouts, calibrations, quantitation reports, chromatograms, strip charts, summary reports, and KIMS batch sheets. Raw data shall contain all original observations that would have indicated all applicable analytical conditions that may have affected the outcome of the test. These conditions may include temperature, sample receipt condition, instrument ID's, etc. Raw data must also include original observations including dates, analyst/prep initials, any volumes or weights not already recorded in the appropriate logbooks, instrument identification and instrument operating conditions/parameters, manual calculations and manual integrations, standard and reagent information, calibration criteria and acceptability, QC sample batch identification and QC criteria and data interpretation, flags and review information.

The raw data is included with the data package as described below, when requested by the client. Not all raw data is filed with a data package if not requested by the client. Refer to Table 10-1 for a summary of raw data filed with the data report. However, all raw data is stored and retained by the laboratory for a specified amount of time. Refer to Table 10-2 for a summary of data retention times and means of retention.

10.10 Data Report/Raw Data Package

A Data Report contains the results of analyses as presented to the client. The Data Report also includes Chain-of-Custody documentation and a narrative and/or cover letter describing the general condition of the samples and a summary of any discrepancies encountered during sample analysis. For more information see Section 9.3.

The Raw Data Package contains information needed to reconstruct how the results of the analysis of a batch of samples including QA/QC samples were derived. Information such as inorganic or organic preparation, chromatograms or strip chart recording, and regression analysis data are usually included.

10.11 Electronic Data Management

Katahdin maintains security procedures in order to ensure the integrity of its network and computer systems. Katahdin maintains its database and reporting systems on dedicated resources within the company system.

All Katahdin personnel are assigned login names and passwords in order to access the computer systems. As described in the current revision of the Katahdin QA Manual and in Katahdin SOP, QA-811, Ethics in the Laboratory, all employees are required to sign a Code of Ethics. This agreement requires the proper use and maintenance of computers and software at Katahdin. Katahdin utilizes dedicated computer work stations, which are used only by a group of cross-trained employees. This prevents the accidental "changing" of data. Additionally, Katahdin's policy is for a software user to log-in under their unique log-in so that all software audit trails are accurate. Using a software program for which another user is already logged on is not acceptable.

The Katahdin Information Management System (KIMS) and the organics data acquisition system are designed with audit trail functions to track the changing and/or manipulation of records. Additionally, individual accounts for the KIMS system are set-up so that certain functions (i.e. the ability to edit data) are only given to certain authorized individuals. Coupled with unique login, these safe guards ensure the integrity and confidentiality of data entry, collection, processing and storage. Passwords for both the KIMS and organics data acquisition systems are changed annually.

Changes to electronic files are allowed in order to correct mistakes or inappropriate manual integrations that are found during the tiered review process. In these cases, the original files are edited and saved as the same file name. Many of these changes can be tracked through the system audit trails. When samples are reanalyzed for organics, the original files are never overwritten. The data system will always automatically create a new file. When changes are made electronically in the metals data system, the system will always prompt the user to save the changes as a new file.

All spreadsheet calculation cells must be locked to prevent unauthorized amendment.

Electronic records are maintained so that historical reconstruction of analytical data can be accomplished. All instrument file naming is standardized and described within each individual analytical SOP. Refer to the current revision of Katahdin SOP SD-913, Data Back-up, Archival and Restoration, for more information.

10.12 Software Quality Assurance

An integral part of the Katahdin Quality Assurance Program is the documented procurement, development, validation, verification, configuration control, modification control and maintenance of all software used in the generation, compilation, reduction and reporting of analytical measurements. The requirements of this program apply to purchased software as well as software that is developed or modified internally. All software and spreadsheets used for data reduction will be managed under Katahdin Analytical Services Information Systems Life Cycle Process (Figure 10-1) prior to use. This includes the validation and verification of that software. Further information is provided in the current revision of Katahdin SOP SD-906, Software Quality Assurance.

10.13 Records Retention

Refer to the current revision of Katahdin SOP, SD-913, Data Back-up, Archival and Restoration and to Table 10-2 of this section for further details.

Client reports are archived onto CDs and kept indefinitely. One copy is kept on site for lab use and one copy is kept off site.

Quality Control records including controlled documents are kept archived for a minimum of 5 years.

Raw data and logbooks are scanned and archived onto CD and kept indefinitely. One copy is kept on site for lab use and one copy is kept off site.

Project records are maintained by Project Management and the Sales Department. Generally, bids are retained for 1 year, client correspondence is retained for 5 years, BOAs and MSA's are retained for the defined lifespan.

Administration Records are retained for a period of 3 to 6 years.

Some states, clients or specific program regulations require longer retention times than five years. These records will be noted with specific storage instructions.

Records pertaining to compliance drinking water samples must be retained for 10 years.

Records maintained off-site are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. These archived records may only be retrieved by designated personnel. A member of management may designate an employee to retrieve data files from the archive storage facility.

In the event that the laboratory goes out of business, all clients will be contacted and given the opportunity to claim their pertinent records. In the event that the laboratory transfers ownership, record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining archives will be clearly established.

TABLE 10-1

SUMMARY OF RAW DATA HANDED IN WITH DATA PACKAGE

	Raw Data Handed into Data Management (HC and/or electronic)	
	Sent to Client with Data Package	Retained by Laboratory
Organics – Level 1/2	Sample results form, QC forms	Sample raw data, QC raw data, runlogs, review checklist
Organics – Level 3	Sample results form, QC forms, calibration forms, runlogs	Sample raw data, QC raw data, calibration raw data, review checklist.
Organics – Level 4	Sample results form, QC forms, calibration forms, runlogs, Sample raw data, QC raw data, calibration raw data	Review checklist
Metals – Level 1/2	Sample results form, QC forms	Review checklist
Metals – Level 3	Sample results form, QC forms, calibration forms, runlogs	Review checklist
Metals – Level 4	Sample results form, QC forms, calibration forms, runlogs, Sample raw data, QC raw data, calibration raw data	Review checklist
Wet Chemistry – Level 1/2	Sample results form, QC forms	Review checklist
Wet Chemistry – Level 3	Sample results form, QC forms, Sample raw data, QC raw data, calibration raw data	Review checklist
Wet Chemistry – Level 4	Sample results form, QC forms, Sample raw data, QC raw data, calibration raw data	Review checklist
Food/Microbiology- all levels	Sample results form	Review checklist

TABLE 10-2

SUMMARY OF RECORDS & RETENTION TIME/MEANS

Records	Hardcopy Retention	Electronic Retention
QA Manual	At least 5 years	Server or PC, refer to SOP SD-913
SOPs	At least 5 years	Server or PC, refer to SOP SD-913
Audits & Responses	At least 5 years	Server or PC, refer to SOP SD-913
Certifications	At least 5 years	NA
Corrective Action Reports	At least 5 years	NA
Logbooks	1-2 years or until scanned	Indefinitely once scanned and archived onto CD (2 copies) - refer to SOP SD-913
PT Data/Results	At least 5 years	NA
MDL/IDL Studies & Verifications	At least 5 years	NA
Internal COCs	At least 5 years	NA
Raw Data to Data Mgt.	Current year or longer until scanned	Indefinitely once scanned and archived onto CD (2 copies) - refer to SOP SD-913
Raw Data not to Data Mgt.	At least 5 years	Indefinitely once scanned and archived onto CD (2 copies) - refer to SOP SD-913
Instrument Files	At least 5 years	Indefinitely once scanned and archived onto CD (2 copies) - refer to SOP SD-913
Data Reports & Reissues	Current year or longer until scanned	Indefinitely once scanned
Field Records	At least 5 years	NA
Contracts, BOAs, MSAs & Amendments	At least 5 years or through the term of the contract	PC - refer to SOP SD-913
Bids	At least one year	PC - refer to SOP SD-913
Client Correspondence	At least 5 years	PC - refer to SOP SD-913
Client QAPPs/tables	At least 5 years	Server or PC - refer to SOP SD-913
PM Telephone Logs	At least 5 years	NA
EH&S manuals, records, disposal logs	At least 5 years	Server or PC - refer to SOP SD-913
Employee transcripts & training files	At least 5 years from when employee ends employment	NA
Administrative Records	At least 5 years	PC - refer to SOP SD-913
Compliance drinking water records	At least 10 years	Indefinitely once scanned and archived onto CD (2 copies) - refer to SOP SD-913

Two copies of all CDs or DVDs are burned, one to be stored on-site and one to be stored off-site.

FIGURE 10-1

INFORMATION SYSTEMS LIFE CYCLE PROCESS

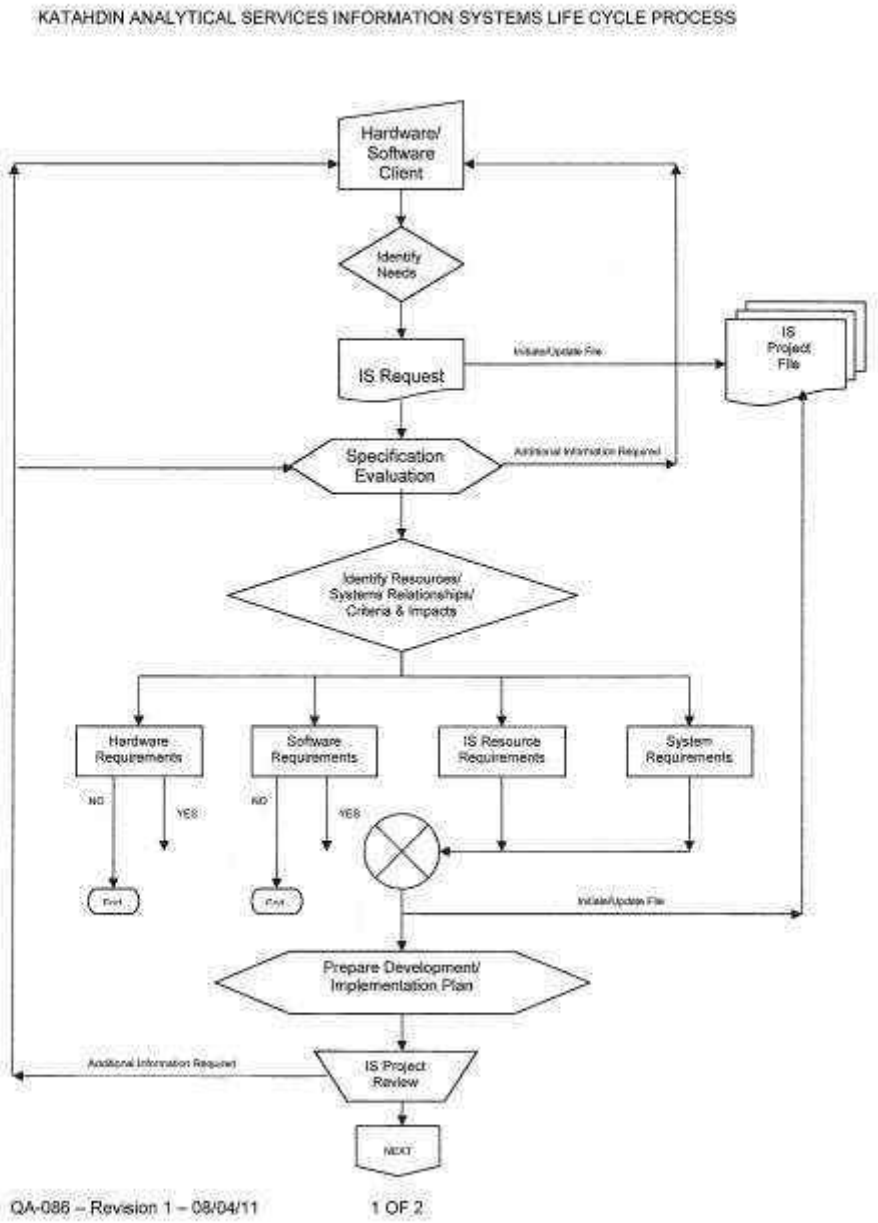
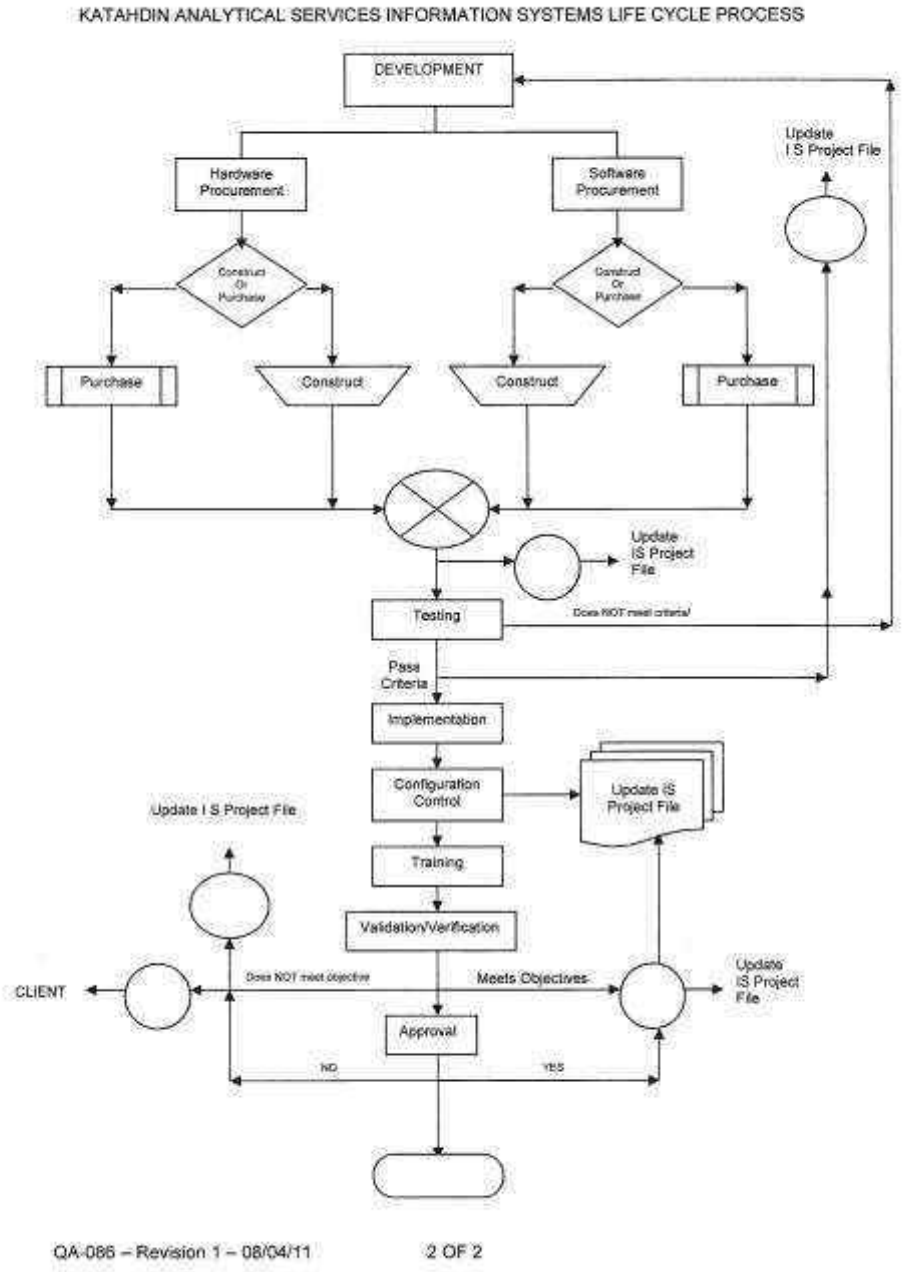


FIGURE 10-1, continued

INFORMATION SYSTEMS LIFE CYCLE PROCESS



11.0 QUALITY CONTROL

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action techniques to prevent or minimize the recurrence of these events.

Individual method SOPs contain the specific quality control criteria required for each method. In most cases, these criteria are method, program or regulatory requirements. Individual client or project required quality control criteria are handled through client driven Quality Assurance Project Plans. Refer to section 3.0 of this manual. When no method or other quality control criteria exists, the laboratory shall set nominal acceptance limits that are consistent with other similar methods. These limits shall be determined and approved by Department Managers/Group Supervisors and the Quality Assurance Officer. When a discrepancy exists between method and/or regulatory quality control acceptability, the most stringent criteria shall be used. If it is not apparent which criteria are more stringent, the regulatory criteria shall be used. All quality control samples are prepared and/or analyzed in the same manner as client field samples.

11.1 Holding Times

There is no topic that is perhaps more critical to the successful operation of any laboratory, than running samples within the accepted holding time for each analysis. The holding time for analytes reflects the allowable time span permitted before the analysis must begin. Due to the sensitive nature or volatility of certain compounds, this permitted time frame within which an analysis must begin is critical to ensuring that target compounds have been verified within a certain margin of accuracy.

Holding times are set by method and/or regulation and must be followed. Refer to Table 6-1 of this manual for a listing of required holding times. Holding times are determined from the time of collection of a grab sample or from the ending of a composite sample to the beginning of preparation and/or analysis.

11.2 Accuracy and Precision Measurements

The results of quality control samples created in the laboratory represent estimates of accuracy and precision for the preparation and analysis steps of sample handling. This section describes the quality control information provided by each of these analytical measurements. Information on the procedures to follow in preparation of the samples or spiking solutions is described for each method and matrix in the respective method Standard Operating Procedure.

Method Blank

A method blank is a volume of analyte-free matrix (e.g. deionized and/or distilled laboratory water for water analyses, or a purified solid matrix for soil/sediment analyses) carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or weight processed. A method blank is performed with each batch of samples or one with every 20 field samples whichever is

more frequent. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a method blank should contain no greater than five times (5X) the practical quantitation limit for common laboratory solvents and phthalate esters or less than the PQL for all other parameters unless otherwise specified in the method or project QA plan. Results of method blank analyses are maintained with other QC data in the respective laboratories. If requested by the client, these data will be included in the report.

Accuracy Measurements

Laboratory Control Samples (LCSs) or **Laboratory Fortified Blank** (LFB) consist of aliquots of analyte-free matrices (water, sand, etc.) spiked with all target analytes or well-characterized materials, e.g. NBS river sediment. Laboratory pure water is used to prepare most LCSs for methods for analysis of water. Highly characterized solids, where available, are used for LCSs for methods for analysis of solids. Where no such solid LCS is available, spiked laboratory pure water or spiked reagent blanks may be substituted. LCSs provide an estimate of accuracy based on recovery of the compounds from a clean, control matrix. They provide evidence that the laboratory is performing the method within accepted guidelines generally in the absence of matrix interferences. They are prepared at a rate of one per batch of twenty or fewer samples.

Matrix Spikes/Matrix Spike Duplicates are similar to Laboratory Control Samples except the analytes used for spiking are added to a second and third separate aliquot from the client samples in a batch of analyses. They incorporate sample matrix effects and field conditions. Matrix spikes are routinely prepared at a frequency of one set (MS/MSD) per twenty samples for organic analyses and one MS per twenty samples for inorganic analyses when adequate sample volume is provided.

Clients may specifiy certain samples for matrix spike and matrix spike duplicate analysis to meet the requirements of their project. In these cases, additional volume is almost always provided. If no samples are specified, the choice is left to the analyst performing the method. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate analysis. Field blank samples should never be chosen for matrix spike and matrix spike duplicate analysis.

Surrogates provide an estimate of accuracy for each sample analyzed by GC/MS and most GC analyses. The accuracy measurement incorporates sample matrix effects and field conditions and is based on recovery of compounds similar to the target analytes, but not expected to be present in the sample as received. Surrogates are added to all samples analyzed by GC/MS, certain GC, and certain HPLC analyses prior to sample preparation.

An **Internal Standard** is an analyte that has the same characteristics as the analyte(s) of interest, but is added to each sample in a batch, just prior to analysis and is used for quantitation. It corrects for bias or change in instrument performance from sample to sample, incorporating effects associated with the analytical process only.
Accuracy is expressed as Percent Recovery (%R). For LCSs and surrogates, percent recovery (**%**R) is calculated using the following equation:

$$\%R = (SR / SA) * 100$$

where: **SR** = the concentration determined
SA = the concentration spiked

For matrix spike samples, the percent recovery is calculated using the following equation:

$$\%R = (SSR-SR)/SA \times 100$$

where: **SSR** = the spiked sample determined result
SR = the original sample determined result
SA = the amount of spike added (expected)

Precision Measurements

A **Sample Duplicate** is a sample that has been homogenized and split into two equal portions before the method specified sample preparation process. It measures sample precision associated with the preparation through analysis and is prepared and analyzed at a rate of one per batch or one per twenty samples (if a batch is less than twenty samples) in the inorganic laboratories. For most organic analyses the MS/MSDs fill this function and provide a measure of overall precision.

Clients may specifiy certain samples for duplicate analysis to meet the requirements of their project. In these cases, additional volume is almost always provided. If no samples are specified, the choice is left to the analyst performing the method. The sample volumes available may restrict the choice of samples used for duplicate analysis. Field blank samples should never be chosen for duplicate analysis.

The comparison of the values determined for a sample and its duplicate (S/DUP or MS/MSD) is expressed as relative percent difference (RPD). RPD is calculated using the following equation:

$$RPD = \frac{S-D \times 100}{[(S+D)/2]}$$

where: **S** = the determined result of the original sample
D = the determined result of the duplicate sample

The vertical bars in the above equation indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

11.3 Statistical Control Limits

Statistically derived laboratory limits serve as a tool for evaluating method performance, for evaluating individual analyst performance and for monitoring the effects of changes to the analytical methods. Current lab policy states that statistically derived QC limits are to be calculated as $\pm$ 3 standard deviations from the mean recovery of a minimum of

twenty data points. It is the policy of Katahdin Analytical Services to generate statistical limits for those methods and/or programs that require their generation. This is done for Surrogates, Laboratory Control Samples and/or matrix spike/matrix spike duplicates if sufficient data is available. A minimum of twenty data points are required for a given analytical procedure and sample matrix prior to generating statistical control limits. Until twenty data points are available, recommended EPA recovery limits must be used if available. Data points shall be chosen at random, either manually or through the applicable database. All data points used in the determination must be taken from data where all routine applicable QC criteria have been met for the analysis.

The percent recovery is calculated for each spiked analyte. The average percent recovery (X) and the standard deviation (s) are calculated for the group of samples.

Refer to the current revision of Katahdin SOP, QA-808, Generation and Implementation of Statistical QC Limits and/or Control Charts, for further details.

11.3.1 Limits

Both upper and lower warning limits and upper and lower control limits are established to interpret performance. Warning limits express a narrower confidence interval and are used to warn the analyst or department manager of possible system inconsistencies or failures, before an out-of-control event occurs. Control limits express the outer limits of accepted method variability. Control limits and warning limits are reviewed periodically against performance. Based on statistical considerations, an evaluation is made to determine whether the control limits need to be revised.

Warning Limits

When not mandated by the method, Katahdin Analytical Services adopts warning limits to be the mean ±2 standard deviations or a 95% confidence interval, where:

The mean percent recovery and standard deviation are calculated as follows:

Mean(P) = $\frac{1}{n}\sum_{i=1}^{n} X_i$

Standard Deviation(s) = $S^2 = \frac{\sum_{i=1}^{n} X_i^2 - \left(1/n\right)\left(\sum_{i=1}^{n} X_i\right)^2}{n-1}$

where: X = individual values
N = total number of values

Recovery warning limits are to be calculated using the following formulas:

$$UWL = P + 2s$$
$$LWL = P - 2s$$

where: UWL = Upper Warning Limit
LWL = Lower Warning Limit

Control Limits

Unless otherwise specified by the analytical method in use or by the project QA plan, Katahdin Analytical Services uses the 99% confidence interval as the control limits, which is defined as the mean ±3 standard deviations. Where the method specific ranges have been determined, Katahdin Analytical Services' goal is for their control limits to fall within the method limits. Control limits are established as follows using the mean and standard deviation as above:

$$UCL = P + 3s$$
$$LCL = P - 3s$$

where: UCL = Upper Control Limit
LCL = Lower Control Limit
P = Mean Percent Recovery
s = Standard Deviation

The control limits and warning limits used to evaluate a sample should be those in place at the time that the sample was analyzed. Once limits are updated, the limits should apply to all subsequent analyses. It is the responsibility of the department managers to inform the QAO when new limits need to be derived. The QAO, or designated person, will derive the new limits, update the appropriate database and ensure that all associated personnel are aware of the updated limits.

11.4 Control Charts

Although data points are evaluated daily against QC limits, it is sometimes necessary to view the data. Control charts are quality control tools that graphically display the QC parameters over time. The lab shall generate control charts as a means to identify method or analyst performance issues. Control Charts are generated using Oracle Graphics, selecting the last 30 data points available for a particular Method and Matrix combination.Katahdin has chosen representative analytes from each type of technology to control chart.

Control charts may be evaluated by plotting and connecting successive data points to enable the laboratory to detect suspicious and out-of-control situations. These events can be caught by monitoring the following: outliers (out-of-control), runs (suspicious), trends (suspicious), and periodicity (suspicious). Please refer to the current revision of Katahdin SOP QA-808, Generation and Implementation of Statistical QC Limits and/or Control Charts, for further information.

11.5 Measurement Uncertainty

Once a method is validated through method development and the determination of sensitivity and precision and accuracy, the measurement uncertainty must be determined. There are many aspects of environmental measurements that contribute to the calculated uncertainty of that measurement. These aspects include field sampling, sub-sampling for analysis and the actual analysis. Unless all information is obtained, it is impossible to calculate a true uncertainty of measurement. However, given the individual SOPs, analysts can perform the methods as specified with approximately the same degree of uncertainty. This analytical uncertainty will be expressed as the measurement uncertainty of the LCS/LCSD.

The LCS/LCSD limits must be determined statistically as described in the current revision of Katahdin SOP QA-808, Generation and Implementation of Statistical QC Limits and/or Control Charts, for further information. Until enough data points can be compiled to generate statistical limits, the laboratory must use estimated or nominal limits. These limits must take into consideration other similar methods, method sensitivity, method interferences, method selectivity, analyst error, etc. Department Managers/group supervisors and the Quality Assurance Officer must reach a consensus on what estimated limits to use.

The uncertainty measurement, whether estimated or statistically derived from LCS/LCSD data must be reported to the client when requested. This would include reporting the LCS/LCSD results with the statistical or estimated acceptance limits.

11.6 Utilization of Quality Control Data

The purpose for preparing and analyzing quality control samples is to demonstrate, through the known entities, how accurate and precise the investigative sample data are. QC tables are included within each analytical method SOP summarizing typical quality control assessment criteria for the methods.

These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in the tables, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in the tables may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Managers/Group Supervisors and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in the tables may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be

different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

12.0 STANDARD OPERATING PROCEDURES

12.1 Purpose and General Provisions

Standard Operating Procedures (SOPs) are formal, revision-controlled documents that:

- provide standard methods for execution and documentation of work, to maximize consistency, uniformity and reliability of products;
- establish the basis for similar training of personnel and set a standard for assessment;
- facilitate coordination among individuals performing separate, but interdependent tasks; and
- define, to Katahdin Analytical Services' clients and to regulatory agencies, the methods used by Katahdin Analytical Services in the performance of tasks having an effect on the quality of data, findings or conclusions;

SOPs describe standard methodologies that may at times be inappropriate for a specific project. In such cases, exceptions to the SOPs are stated in the raw data or the analytical narrative with rationale.

Refer to the current revision of Katahdin SOP QA-800, Preparation of SOPs, for further detailed information on SOPs.

12.2 Responsibilities

Department Managers, Group Supervisors and analysts are responsible for determining, through consultation with the Quality Assurance Officer and Management, the activities that require SOPs. Department managers are also responsible for working with the appropriate technical personnel to develop the SOPs.

The Quality Assurance Department is responsible for obtaining technical review and approval of SOPs, for maintaining control of new SOPs and revisions, and for maintaining an up-to-date distribution list for SOPs.

Katahdin Analytical Services personnel are responsible for performing tasks in accordance with applicable SOPs, except as explicitly directed by a relevant Quality Assurance Project Plan, contract, or Health and Safety policy. Katahdin Analytical Services personnel are also responsible for assisting in designing accurate and practical SOPs and in keeping the SOPs up-to-date.

Technical reviewers of SOPs are responsible for providing review of drafts sent to them within the schedule indicated in the request.

12.3 Minimum Contents of SOPs

Each Standard Operating Procedure shall contain at a minimum, the following information:

Title - The name of the concerned task

SOP Number - The internal document control number assigned and tracked by the QA Department

Acceptance - The signature of the originator(s), Quality Assurance Officer and appropriate operations management authority to officially adopt the procedure

Date - Date of issue of most recent revision

Scope and Application - An explanation of the objectives of the procedure, typical applications and limitations

Definitions - A listing of any terms, expressions, or acronyms found in the procedure

Responsibilities - Identification of the individuals (by title or organizational position) and their responsibilities in performing and facilitating the tasks governed by the SOP

Safety Considerations - A discussion of specific Health and Safety issues that must be considered prior to and during the performance of the procedure described.

Pollution Prevention/Waste Disposal - A discussion of waste disposal and minimization procedures applicable to the method.

Summary of Method - A short synopsis of the chemistry involved in the procedure.

Interferences - Any factors that may interfere with the proper performance and/or outcome of a procedure and that could compromise the results.

Apparatus and Materials - A complete list of the equipment, apparatus, etc. needed for the procedure

Reagents and Standards - A complete list of the reagents, standard solutions, solvents, etc. needed for the performance of this procedure

Sample Collection, Preservation and Handling - Any special considerations needed to assure the integrity of the sample and, consequently, the analytical process.

Method/Procedure - A clear description of the task on a step-by-step basis. The method description should be written clearly enough, and in sufficient detail, to ensure that any two persons performing the procedure will achieve equivalent results, and to provide clients and reviewing agencies with a thorough understanding of the procedure. Acceptable and equivalent alternatives should be addressed whenever possible, and described in the same detail. Where applicable to the SOP, the procedure should include a discussion of sample preparation and calibration requirements, a discussion or reference for equipment/instrument maintenance, computer hardware and software and troubleshooting, and a summary of the automated and manual calculations performed as well as reporting requirements, including data flow charts as appropriate. The SOP should address differences between a published method and Katahdin Analytical's performance of that method if any exist.

Quality Control Requirements and Acceptance Criteria and Corrective Actions - An outline of quality control requirements, including, procedures, frequency requirements, and acceptance criteria. Corrective actions include a description of what must be done, when and by whom in instances when the QC requirements are not met. This section may be in the form of a table.

Applicable Documents/References - A listing of pertinent, supporting procedure or reference documents such as methods, manuals and/or SOPs

12.4 SOP Development and Approval

Laboratory SOPs are developed by the laboratory's technical staff, working with the QA Department. The QA Department will also assist by assigning SOP numbers and by coordinating word processing, review and approval. Laboratory SOPs must be reviewed and approved by the management of the laboratory operations to which the SOPs apply. Thus, the following people must review and approve each laboratory SOP:

- the Department Manager of the specific operation to which the SOP pertains - This signature indicates that the written SOP reflects the current practice in the laboratory and that the SOP is technically adequate to handle the analysis of environmental samples expected to be received at Katahdin Analytical Services.

- the Laboratory President - This signature signifies a management review and approval of the practices detailed in the SOP.

- the Quality Assurance Officer - This signature indicates that the SOP has been reviewed for compliance with the referenced methods and that discrepancies between the method and practice have been resolved.

12.5 Numbering

Each SOP is assigned a unique Katahdin Analytical Services number from the inventory of Katahdin Analytical Services SOPs maintained by the QA Department.

12.6 Revisions

SOP revisions may be necessitated by regulatory requirements, technological advancements or other causes, but not by the requirements of a single project alone. Contradictions between standard procedures and the requirements of a specific project are resolved in the quality assurance plan for that project.

Revisions may be proposed initially by the Quality Assurance Department or they may be recommended by users. Recommendations for revisions must be sent to the Quality Assurance Department. Revisions must not be made by an individual to only his/her personal copy. Recommendations for minor revisions will be accumulated by QA until sufficient to warrant a document revision.

Recommendations for revisions must be made on a Katahdin "SOP Request For Change" Form (Figure 12-1). All pertinent information must be filled in. Proposed changes must be indicated on the form. Alternatively, revisions can be initiated electronically. Per analyst request, the QA Officer will place an electronic copy of an SOP on the company server for revision. The revisions should be marked so that the changes can be tracked and approved electronically by the QAO or by a member of management.

Server_a\QAQC\QAQC\SOP's in Revision

The QA Officer is authorized to approve minor revisions. Revisions that affect the technical approach or content will also require review and approval of the Department manager or other technical staff. Once formally accepted, the revised document replaces the previous version and is distributed to controlled copy holders with instructions as to what document(s) it replaces.

In some situations, changes to a procedure may require immediate implementation in the lab. A formal revision must be initiated by the analyst or department manager as described above. In addition, steps must be taken to document the procedural changes in the interim while the SOP is under revision. These steps include initiating a Katahdin SOP Request for Change. The QAO will distribute controlled copies of this form to all controlled copy holders of the affected SOP. This form will act as the revision until the formal revision is completed.

Some SOPs may not require revisions if no changes have been made. At a minimum, though, SOPs should be reviewed annually to ensure that no revisions are required. A SOP No Change Form (Figure 12-3) should be filled out. This form must be signed by the Department Manager and the QAO. It is filed with the hardcopy of the SOP. Additionally,

the No Change Form should be scanned and attached as an addendum to the Adobe file of the SOP.

If annual review is not possible for all SOPs, then the QA Officer shall prioritize the SOPs for a timely review. An inventory of SOP review dates is maintained by the QAO.

The QAO will determine whether a new training form will need to be initiated for employees. This will be based on whether there are major changes to the SOP. If a new training form is not deemed necessary, training for the revised SOP may be documented on a Katahdin Retraining Form (Figure 12-2). Minor changes will not necessitate the use of either form. It is the department manager's responsibility to assure that the proper training forms are completed.

12.7 Document Control and Distribution

SOPs must reflect current operating conditions and be compliant with applicable method and program requirements. To meet these needs, all Katahdin Analytical Services SOPs and SOP revisions used within the laboratory must be controlled documents that are administered by the QA Department. Each page of a controlled copy is marked as such in contrasting ink (typically red or blue) to prevent the use of un-controlled photocopies. The QA Department maintains an inventory of all SOPs developed by the laboratory and their revision status.

The QA Department distributes SOPs to technical staff as required, maintains distribution lists to ensure that revisions and new SOPs are distributed to all appropriate individuals, periodically verifies that SOPs in use in the lab are current, controlled copies and ensures that obsolete versions are removed from use and destroyed. A Receipt Acknowledgment Form is completed for each SOP for tracking and distributing updates of the documents. Refer to Katahdin Analytical Services SOP QA-804, Document Control for Standard Operating Procedures.

12.8 SOP Archive

An archive of all laboratory SOPs, in the form of both hard-copy (Title Page only) and electronic masters of current revisions, is maintained by the Quality Assurance Department within Katahdin Analytical Services. The archive also contains a hard-copy master of all obsolete versions of each revised SOP.

SOPs are distributed from the archive. Access to originals is obtained through QA personnel.

FIGURE 12-1

SOP REQUEST FOR CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, LLC.
QAM/SOP REQUEST FOR CHANGE FORM

Name of Person Requesting Change:	Date:
SOP Number & Revision Number:	
Title:	
Reason for Change:	
Complete and Concise Description of Change (may include attachments):	
Effective Date of Change:	
Department Manager Signature:	Date:
Operations Manager Signature:	Date:
QAO Signature:	Date:

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FIGURE 12-2

KATAHDIN RETRAINING FORM

KATAHDIN ANALYTICAL SERVICES RETRAINING FORM

RETRAINING FORM #

PART 1 – RETRAINING

TRAINER _____

TRAINEE _____

PROCEDURE:

REFERENCES: Please review the following:

QA MANUAL: _____

SOP _____

PUBLISHED METHOD _____

OTHER _____

REASON FOR RETRAINING:

REFRESHER _____

NON-CONFORMANCE _____

PROCEDURAL MODIFICATIONS _____

OTHER _____

TRAINER SIGNATURE: _____ **DATE:** _____

TRAINER SIGNATURE: _____ **DATE:** _____

TRAINEE SIGNATURE: _____ **DATE:** _____

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FIGURE 12-2, CONT'D

KATAHDIN RETRAINING FORM

KATAHDIN ANALYTICAL SERVICES RETRAINING FORM

RETRAINING FORM #

PART 2 – QAO OBSERVATION OF RETRAINING

COMMENTS:

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

QAO SIGNATURE:_____ **DATE:**_____

FIGURE 12-3

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date:

SOP Number: _ _-_ _ _-_ _

SOP Title:

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** _____

QAO Signature: _____ **Date:** _____

QA-034 – Revision 1 – 01/14/2010 Updated 03/25/2016

13.0 PERFORMANCE AND SYSTEM AUDITS

Katahdin Analytical Services participates in a variety of interlaboratory and intralaboratory tests and performance checks to provide periodic assessment of the effectiveness of the overall quality control program. Refer to the current revision of Katahdin SOP, QA-803, Laboratory QA: Self Inspection System, for more detailed information.

Proficiency Test samples (PTs) are analyzed periodically to verify method accuracy. These PT samples may be external (e.g., WP, WS, S&HW, air, and food microbiology) or internal (prepared or purchased by the QAO). External Proficiency Test samples are received and analyzed twice per year for each of the following studies: Water Pollution (WP), Water Supply (WS) and Solid & Hazardous Waste (S&HW), and Air. External Proficiency Test samples are received and analyzed once per year for food microbiology. PT samples are ordered and analyzed for all NELAC required parameters and/or as required by individual state or federal regulations (State of Maine, DoD, ISO, etc). The QAO retains information regarding required PTs. External PT samples are also analyzed routinely for a number of state and federal program certifications. Internal PT samples are prepared by the laboratory QAO on a case by case basis. The samples may be identified as PT samples or they may be "blind", i.e., disguised as client samples.

13.1 Interlaboratory Performance Surveys – (including, but not limited to)

- Proficiency Test (PT) Samples - Water Supply - Semiannual
    - Trace Metals
    - Nitrate/Nitrite/Fluoride/Chloride/Sulfate/ortho-phosphate
    - Volatile Organics – Regulated, Unregulated & Trihalomethanes
    - EDB/DBCP
    - Turbidity
    - Total Dissolved Solids
    - Calcium (as $CaCO_3$)
    - pH
    - Alkalinity
    - Sodium
    - Conductivity
    - Total Cyanide
    - Total Organic Carbon
    - Uranium
    - Total Coliform, e-coli and HPC
- Proficiency Test (PT) Samples - Water Pollution - Semiannual
    - Trace Metals
    - Minerals
    - Nutrients
    - Demand
    - PCBs
    - Pesticides
    - Herbicides

- Volatile Halocarbons & Volatile Aromatics
- Semivolatile Organics and Low-Level PAH's
- Total Cyanide
- Solids
- Oil and Grease
- Total Phenolics
- Hexavalent Chromium
- Explosives
- DRO/GRO
- Nitrite
- Specific Conductance
- TPH
- Surfactants
- State Specific hydrocarbon analyses such as EPH/VPH, CT-ETPH, Fl-PRO, TX-1005
- pH
- Total Alkalinity
- Sulfide
- Turbidity
- Color
- Total Residual Chlorine
- Acidity
- Fecal Coliform and e-coli

- Proficiency Test (PT) Samples – Solid & Hazardous Waste (solids) - Semiannual

  - Metals
  - PCBs
  - Pesticides
  - Herbicides
  - Volatile Halocarbons & Volatile Aromatics & Medium Level Volatiles
  - Semivolatile Organics & Low-Level PAH's
  - Total Cyanide
  - Hexavalent Chromium
  - Explosives
  - TCLP
  - pH/corrosivity
  - DRO/GRO
  - Oil and Grease
  - Anions
  - Nutrients
  - State Specific hydrocarbon analyses such as EPH/VPH, CT-ETPH, Fl-PRO, TX-1005

- Proficiency Test (PT) Samples – Air - Semiannual

  - Volatile Organics

- Proficiency Test (PT) Samples – Microbiology (food) - Annual

  * E-coli
  * Total coliform
  * Salmonella
  * Listeria
  * Yeast & Mold
  * Staphylococcus
  * Aerobic Plate Count

PT requirements may differ from state to state or for different regulations. Generally, all states and regulations require at least one study per year. NELAC and DoD require two studies, so Katahdin participates in this more stringent program. Less stringent state requirements are filed with individual state files in the QA office.

To obtain initial certification for a parameter, in accordance with NELAC and DoD, the laboratory must successfully analyze two sets of PT studies, the analyses to be performed at least 15 calendar days apart from the closing date of one study to the shipment date of another study for the same field of proficiency testing. To maintain continuing certification for a parameter, in accordance with NELAC and DoD, the laboratory must successfully analyze two out of the last three sets of PT studies, the analyses to be performed with completion dates of successive proficiency rounds for a given field of proficiency testing being approximately six months apart. Failure to meet the semiannual schedule is regarded as a failed study.

An unacceptable PT score for two out of the last three studies for any analyte will result in decertification for that analyte.

Performance on PT samples is communicated to the appropriate laboratory staff. Any analytes demonstrating unacceptable results (i.e., falling outside the sponsoring agency's acceptance limits) require a thorough investigation of the associated analytical data, including quality control data. Causes for error are identified, if possible, and corrective measures are implemented for those parameters reported unacceptably. The laboratory QAO maintains copies of all PT results and corrective action plans in the QA files. PT results are sent directly by the PT provider to the pertinent accreditation authorities (as identified by the laboratory when PT results are submitted back to the provider). PT records and corrective action files are made available to any other accreditation authority or client upon request.

13.2 Periodic Internal Audits

Internal auditing is conducted by the QA Department or designee on an annual basis. Internal audits take two forms – Technical Audits and Systems Audits. Technical Audits verify the laboratory’s compliance with method specific requirements. Systems audits verify the laboratories compliance with this Manual, SOPs and that adequate documentation exists to satisfy the project requirements. Both types of audits are also designed to assess laboratory compliance with certification and regulatory requirements such as those for Maine, NELAC and the DoD.

Audit checklists may be used to ensure that all salient points are addressed and documented. The checklists are filled out legibly and reproducibly, in ink, by the auditor, and are signed and dated by the auditor when completed. The audit checklist is based on EPA laboratory evaluation criteria, the provisions of the Laboratory Quality Assurance Manual and Katahdin Analytical Services SOPs. Project audit checklists are drawn from the applicable QAPPs, as well as relevant provisions of the QA Manual.

13.2.1 Technical Audits

Technical Audits address the laboratories compliance with method specific requirements.

Technical Audit Procedures

Technical audits are performed by the QA Department or their designee. The audit should cover the following areas:

* Method Standard Operating Procedures
* Initial and continuing calibration procedures, including independent calibration verification.
* Demonstration of Capability
* Method Quality Control Samples – Method Blanks, Laboratory Control Spikes, Matrix Spikes, Sample Duplicates.
* Surrogates and Internal Standards
* Method Detection Limit determination.
* Corrective Action Procedure

13.2.2 Systems Audits

System audits address general laboratory operations and conformance to the Laboratory Quality Assurance Manual. System audits of laboratory operations are performed at a minimum frequency of once per year per laboratory group (i.e. wet chemistry, metals, organic extractions, GC, GC/MS). These system audits may be performed in conjunction with an outside system audit (i.e. the QAO will accompany the outside auditor and record any findings as appropriate).

Systems Audit Procedures

Systems audits are performed by the QA Department. The audit should cover the following areas:

* Personnel qualifications and training records
* Adequacy of laboratory facilities, including work space, lighting, ventilation, and supplies
* Maintenance and calibration recordkeeping for analytical equipment
* General operations, including glassware cleaning, inventory and checking of reagents and standards, and storage procedures
* Recordkeeping, including sample log-in and tracking, traceability of standards, control charts, and raw data recording and tracking.

* Corrective Action follow through from past audits

13.2.3 Internal Performance Evaluations:

Performance audits involve submittal of blind spikes to the laboratory by the Quality Assurance Department for assessment of analytical accuracy. Due to the high number of Performance Evaluation studies that Katahdin Analytical Services participates in, the Quality Assurance Department does not routinely conduct internal performance audits. However, the Quality Assurance Department may provide internal performance standards if required on specific projects or warranted by the laboratory. Performance standards may be purchased as whole sample volumes or prepared in the laboratory.

13.2.4 Project Audits:

Some quality assurance project plans, QAPPs, may require project-specific laboratory Projects Audits.

13.2.5 Audit Reports & Follow-Up

The QAO will prepare internal audit reports, within approximately 30 days of the audit, summarizing any findings and required corrective actions. Reports are disseminated to the Department Managers/Group Supervisors and the Katahdin President. The appropriate managers are responsible for immediately investigating the finding. The root cause of the finding shall be determined and the impact of the finding on a released data shall be determined. The appropriate managers are responsible for immediately notifying the Katahdin Project Manager of any data issues so that the client(s) can be contacted. Managers must submit a response to the findings, including corrective actions plans, root cause analysis and implementation dates to the QAO within 21 days of receiving the summary of findings. The QAO will review the responses and determine if the corrective actions and implementation dates are adequate.

Once the responses are accepted, the QAO shall communicate any changes in procedure or other corrective action plans to all appropriate personnel. Any additional training shall be documented on a Katahdin Retraining Form. The non-conformance, the correct procedure and the reference for the requirement shall be indicated on the form. Each employee receiving the training shall sign the form. A copy of the form will become part of the employee's training records.
It is the responsibility of section department managers to ensure that all corrective actions documented on the retraining forms are implemented. Any problems should be communicated to the QAO. The QAO shall conduct follow up audit inspections to ensure that corrective actions are implemented. This is documented on part 2 of the Katahdin Retraining Form. All Katahdin Retraining Forms shall be numbered systematically and a record kept with dates of closure. Closure is indicated by a QAO follow-up audit.

If an event is discovered which casts doubt on the validity of any test results, an internal investigation will be initiated within 24 hours of the discovery. If it is

determined the validity of the test results is uncertain, the client shall be notified within 24 business hours of the conclusion of the investigation.

13.3 External Audits

In addition to internally conducted systems audits, the laboratory is regularly audited by clients, potential clients, state and federal program regulators, and other organizations. These audits may be specific to program or project requirements, encompass a complete review of laboratory systems, or both.

External audits usually begin with an opening meeting in which the audit team states the purpose of the audit and how the audit will be conducted. The laboratory may have auditors sign a Confidentiality Agreement to protect its clients and business. All records and documentation will be made available on-site for the audit team to review as required and necessary. Auditors will tour the laboratory and interview analysts to assess the lab's compliance with the requirements. A closing meeting is usually held at the end of the audit to summarize the audit findings and to discuss a timeframe for receipt and response to the audit.

Katahdin Analytical Services uses these external audits to additionally evaluate laboratory function and performance. The QA Department distributes audit reports to department managers and management, coordinates corrective actions to any findings, formalizes and documents the responses required, and may use external audit findings as the basis for future internal audits.

13.4 QA Reporting and Corrective Action

Each systems audit (internal or external) may be followed by a debriefing, in which the auditor discusses his/her findings with the laboratory representatives. The debriefing serves a two-fold purpose. First, laboratory management is afforded an early summary of findings, which allows them to begin formulating corrective actions, and second, the auditor has a chance to test preliminary conclusions and to correct any misconceptions before drafting his/her report.

The systems audit report (which may or may not contain performance audit findings) is issued to the the appropriate Department Managers/Group Supervisors and personnel for corrective action. Responses to the findings are forwarded, in writing, to the QA Officer. The QAO then circulates the report to Katahdin President.

14.0 PREVENTIVE MAINTENANCE

The laboratory is equipped with all equipment needed for analysis and support activities. All equipment is maintained in working condition through a schedule of preventive maintenance and/or outside service contracts or appointments as needed. All maintenance, including internal maintenance and outside service calls, is documented. Any out-of-service equipment is marked as such until it can be repaired and shown by calibration or testing to perform correctly. In cases where outside equipment is rented or borrowed, any records necessary to demonstrate equipment calibration or maintenance will be requested from the provider.

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or instrument manufacturer service personnel.

All equipment must be maintained so that its performance is acceptable for its intended use. For example, an instrument that does not have sufficient sensitivity should not be used for low-level analysis or a balance not verified above 100 grams should not be used to weigh items over 100 grams.

SOPs are written for each instrument that cover basic operation and maintenance procedures. Refer to Katahdin Analytical Services SOPs CA-101, Equipment Maintenance, and CA-102, Balance Calibration for further information. Detailed logbooks documenting preventive maintenance, non-routine maintenance and repairs are also maintained for each instrument. Logbook requirements are detailed in SOP CA-101. The following are brief summaries of maintenance for each major instrument.

14.1 Preventive Maintenance - **GC/MS Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Check to ensure the pressure on the primary regulator never runs below 100 psi
- Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- Change septa weekly or as needed.
- Replace/cut GC column as needed.
- Replace GC injector glass liner weekly or as needed.
- Replace glass jet splitter as needed.
- Replace pump oil as needed.
- Change gas line dryers as needed.
- Replace electron multiplier as needed.

14.2 Preventive Maintenance – **GC Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Check to ensure the pressure on the primary regulator never runs below 100 psi.
- Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- Change septa weekly or as needed.
- Replace/cut GC column as needed.
- Replace GC injector glass liner as needed.
- Change O_2/moisture traps as needed
- Clean/replace GC detector as needed.

14.3 Preventive Maintenance – **Purge and Trap Sample Concentrator**

Regularly performed maintenance includes, but is not limited to, the following:

- Check to ensure the gas supply is sufficient for the day's activity and delivery pressures are set as described in the SOP.
- Replace trap as needed.
- Decontaminate the system after running high concentration samples or as required by blank analysis.
- Check system for leaks when problem suspected.

14.4 Preventive Maintenance – **Archons**

Regularly performed maintenance includes, but is not limited to, the following:

- Visually inspect sampler for corrosion or significant water seepage past plunger.
- Check system pressure (denotes leaks).
- Check for sufficient standard materials in standard vials.
- Recalibrate x,y,z component, i.e. robotic arm, as needed.

14.5 Preventive Maintenance – **ICP Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Clean torch assembly and spray chamber when discolored.
- Clean nebulizer as needed.
- Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Check coolant water level weekly; replenish as necessary.
- Check rinse solution level daily; replenish as necessary.
- Check waste container level daily; empty as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

14.6 Preventive Maintenance – **Mercury Analyzer**

Regularly performed maintenance includes, but is not limited to, the following:

- Replace drying tube as necessary
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Replace mercury lamp as necessary.
- Clean optical cell quarterly or as needed.
- Clean liquid/gas separator when it becomes cloudy. Replace as needed.
- Check waste container level before each use; empty as necessary.
- Check exhaust system integrity before each use; correct as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

14.7 Preventive Maintenance – **Lachat Autoanalyzer**

Regularly performed maintenance includes, but is not limited to, the following:

- Check pump tubing; replace as needed.
- Clean interference filter with Kimwipe.
- Check reagent levels and expiration dates; refill or replace as needed.

- Rinse manifolds with water after analysis.
- Check manifold board surfaces; clean as needed by running under tap water.
- Check supplies and reagents; order as needed
- Check for leaks
- Check autosampler and autosampler trays; clean and/or lubricate as needed.
- Check fittings and o-rings on boards; replace as needed.
- Check peristaltic pump rollers; clean and lubricate as needed.
- Inspect manifold tubing for kinks and/or stains; replace as needed.
- Inspect valve flares; clean or replace as needed.
- Inspect reagent and waste lines; replace as needed.
- Check flow cell; clean as needed with Kimwipe.

14.8 Preventive Maintenance - **General Laboratory Areas**

- Clean and calibrate balances annually (minimum).
- Check balance calibration each day of use.
- Clean balance pan prior to each use.
- Calibrate automatic pipettes with each use.
- Calibrate spirit thermometers yearly against an NIST traceable thermometer; calibrate digital thermometers quarterly.
- Record refrigerator, freezer, and oven temperatures each weekday.
- Clean, check, calibrate to manufacturers' specifications all pH, DO, conductivity, and Turbidity meters annually (minimum); clean, check, calibrate to manufacturers' specifications all spectrophotometers annually.
- General housekeeping: keep counter tops, hoods, and floors clean.
- Check airflow in hoods once a week.

14.9 Preventive Maintenance – **Konelab AutoAnalyzer**

Regularly performed maintenance includes, but is not limited to, the following:

- Rinse and refill distilled water container weekly.
- Check cleanness of segments weekly.
- Wash reagent tubes monthly.
- Change lamp as needed.
- Change dilutent and wash tubes as needed.
- Change mixing paddles as needed.
- Change syringes as needed.
- Change dispensing needles as needed.
- Change drain and waste tubes as needed.
- Check used cuvettes and waste daily.

14.10 Preventive Maintenance – **Accelerated Solvent Extractor (ASE)**

Regularly performed maintenance includes, but is not limited to, the following:

- Check for leaks at the pump solvent reservoir, valves and other components.
- Inspect needle alignment of source needle.
- Check alignment of autoseal arms.
- Change peek seals and o-rings on cell caps after about 50 extractions per cell.
- Inspect cell edges for nicks and gouges on cell body.
- Inspect stainless steel frits and sonicate in solvent if needed.

14.11 Preventive Maintenance – **ICP/MS Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Clean torch assembly and spray chamber when discolored.
- Clean nebulizer as needed.

- Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Check coolant water level weekly; replenish as necessary.
- Check rinse solution level daily; replenish as necessary.
- Check waste container level daily; empty as necessary.
- Check cleanliness of instrument air filters weekly; clean or replace as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

14.12 Preventive Maintenance - **Horizon SPE Automated Extractor System**

Regularly performed maintenance includes, but is not limited to, the following:

- Check and clean sensors with a KIMWipe.
- Change sensors as needed.
- Purge system with solvent before use and after use.
- Clean system with hot water by running method 15 after samples are analyzed and before purging system.

14.13 Preventive Maintenance – **HPLC Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Check and sonicate pump valves as needed.
- Backflush column as needed.
- Replace analytical column or guard column as needed.
- Sonicate and replace solvent with every use.
- Replace the UV lamp as needed.
- Check and replace seal-pak as needed

14.14 Preventive Maintenance – **GPC Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Fill solvent reservoir as needed.
- Empty waste container as needed.

14.15 Preventive Maintenance - **TOC Combustion Analyzer**

Regularly performed maintenance includes, but is not limited to, the following:

- Check level of dilution water, drain vessel water, humidifier water, autosampler rinse water, and phosphoric acid vessel and fill as needed.
- Replace oxygen cylinder as needed.

14.16 Preventive Maintenance – **IC Instrumentation**

Regularly performed maintenance includes, but is not limited to, the following:

- Check regenerate pump tubing and replace as needed.
- Clean or regenerate column as needed.
- Replace analytical column or guard column as needed.
- Change suppressor as needed.

14.17 Preventive Maintenance - **Miscellaneous Instrumentation (including field testing equipment)**

Regularly performed maintenance includes, but is not limited to, the following:

- Replace spectrophotometer lamps as needed.
- Inspect DO probe membrane for tears and check for air bubbles under the membrane.
- Replace the DO probe membrane cap and electrolyte solution as needed.
- Clean pH electrode as needed.
- Change the autotitrator filling solution as needed.
- Clean, check, calibrate to manufacturers' specifications all pH, DO, conductivity, and turbidity meters annually (minimum); clean, check, calibrate to manufacturers' specifications all spectrophotometers and balances annually. All calibrations must be NIST traceable.

14.18 Preventive Maintenance - **Milestone Ethos EX Microwave Extraction System**

Regularly performed maintenance includes, but is not limited to, the following:

- Reshape caps after each use.
- Check probe vessel for cracks.
- Check seals for disfigurement. Replace as necessary.
- When vessels are dirty, run through methylene chloride cycle.

14.19 Unscheduled Maintenance & Troubleshooting

In addition to preventive maintenance, analysts are often required to perform unscheduled maintenance due to sporadic instrument problems. The instrument problems could be obvious, such as those cause by “nasty” samples, or not so obvious. The problem could be more mechanical, such as an autosampler jamming or a needle clogging, or the problems could be more technical such as failing QC for no apparent reason. In all cases, analysts shall proceed through a series of troubleshooting steps to investigate the problem. Analysts should consult the manufacturer’s guidelines for troubleshooting certain equipment problems. Changing only one parameter and/or part at a time is critical to ascertain which change may improve or correct a problem.

All troubleshooting steps and outcomes must be recorded in the applicable instrument maintenance logbook for future reference and guidance. The steps taken, which correct the problem should be noted as such. If it is determined that a problem with the equipment exists, and that the problem has had an impact on the quality of past completed data, an immediate Corrective Action Report (CAR) must be initiated. A thorough investigation of all impacted data must be completed and corrective action must be implemented. Refer to the current revision of Katahdin SOP, QA-803, Laboratory Quality Assurance Self Inspection System, for further details.

14.20 Service Contracts

Katahdin will maintain service contracts for major instrumentation as needed. The need for a service contract will depend on factors such as the cost, the estimated down time, the availability of back-up instrumentation, and the availability of skilled analysts to perform non-routine maintenance. The decision to purchase a service contract is made by the management team of the President and the Department Managers.

In cases where a service contract is not in place, and a problem with an instrument or piece of support equipment cannot be corrected in-house, Katahdin will use reputable service companies to perform the repair and/or troubleshooting work. Relationships are maintained with several companies to perform these services.

Occasionally, equipment may need to be sent out to the service company for repair. All equipment must be packaged in large enough boxes to leave room for at least four inches of bubble wrap on every side, including the top and bottom. Any small movable parts must be removed and wrapped separately. When available, a manufacturer’s box with appropriate Styrofoam inserts should be used. All equipment returning to the laboratory must be checked and recalibrated to be sure all instrument capabilities and conditions have been maintained.

A preventive maintenance contract is in place for all support equipment and thermometers for cleaning, calibrating to manufacturer's specifications and verifying against NIST traceable sources. The support equipment includes all pH meters, conductivity meters, turbidity meters, balances, spectrophotometers, autoclaves, ovens, and incubators. Field equipment meters are included in this annual verification. All equipment supported under this contract are labeled by the outside calibration company with the last calibration date, and when the next calibration is due. All additional comments of the outside calibration are maintained in files with the QA Officer.

Records of all service contract visits, third party services and preventive maintenance services are maintained to document support equipment performance. All equipment not meeting manufacturer's specifications or those required by the test are removed from service until appropriate corrective actions can be taken.

15.0 NON-CONFORMANCE & CORRECTIVE ACTION

When problems are identified, whether at the bench level before results have been issued, or at some point after the results have been issued, certain steps must be taken to ensure the consistent control of non-conforming work or test results. Investigation of the non-conformances must be initiated by the affected Department Manager/Group Supervisor and/or the Quality Assurance Officer. When corrective action can be taken without affecting the data quality, immediate action must be taken to implement the corrective action. After reviewing the impact to data quality, if it is determined that the quality will be impacted (i.e. missed hold time, failing QC), the client must be contacted for consultation. All joint decisions must be documented and/or narrated as appropriate. If results have been sent or are ready to be sent when a non-conformance is discovered, the affected clients must also be notified, and any reports held or resubmitted as appropriate. If the cause of a problem cannot be immediately identified, and the probability of other work being affected exists, the QAO has the authority to halt work until the non-conformance can be corrected.

For most laboratory situations, problem identification, corrective action, and resumption of operation and/or return to control occurs at the bench, with documentation written directly in the appropriate logbooks. These occurrences include events where laboratory quality control criteria have been exceeded but which can be corrected without compromising the analytical results or delaying the preparation or analytical process.

For other situations, non-conformances may affect data quality and will require client notification. These situations may involve narration of data, re-analysis and/or re-preparation, or flagging of data. Non-conformances that must be documented on a Non-Conformance Report (NCR) (Forms QA-006, QA-007, and QA-008) (Figure 15-1) include, but are not limited to the following:

1. **When Quality Control criteria are not met** - These QC criteria include, but are not limited to, blanks, LCSs, surrogates, spikes, ICVs/CCVs. CARs do not necessarily have to be initiated in each case, especially if data is rejected, but the corrective action should be documented on the raw data, logbook or other analyst records.

2. **When laboratory SOPs are not followed** - This includes all aspects of laboratory operations from receipt to reporting. If SOPs are in the draft format, or if they are not written, it should be understood that lab operations will proceed at the direction of your department manager or Operations Manager, rather than what may be written down (since this is in draft form and may not be adequate information). For this reason, communication is vital.

3. **When we do not meet client expectations** - Some aspects of this may be narrated, Other aspects will come in the form of client complaints.

The underlying purpose of this process is to identify instances that may adversely affect the data. This process may also help to:

1. **To help standardize the laboratory's procedure for handling events that require corrective action** - Every situation should be evaluated individually, but there are some basic guidelines that should be followed.

2. **To record actions taken when SOPs are not followed so that the data produced is supported with a documented sequence of events.**

3. **To document occurrences in the lab that may affect the integrity of laboratory records.**

4. **To provide a learning tool for individuals involved in the problem investigation and corrective action plan.**

5. **To provide a means for tracking recurring problems that may need further investigation into the root cause of the problem.**

15.1 Problem Identification

15.1.1 Laboratory Identification

The analyst generating the data is responsible for reviewing all results against the established limits. Any deviations are immediately evaluated as potential out-of-control events. Specific examples of some out-of-control events may be: LCS failures, blank contamination, poor precision, prep errors, missed holding times, login errors, calibration failures, retention time window problems, matrix spike failures and surrogate failures. The review process may include the application of statistics. Please refer to the current revision of Katahdin SOP SD-904, Data Reduction and Validation. If data are outside accepted limits, the analyst should review and evaluate the data and all associated Quality Control elements together before making a decision as to the acceptability of the data. Each individual method SOP contains corrective action tables to help guide analysts in making these decisions. Once all QC items have been considered, the analyst should immediately take the appropriate actions including documenting the problem on an NCR (Figure 15-1).

15.1.2 Client Complaints

In the event that problems are not evident or identified prior to reaching the client, the Katahdin Project Manager would receive notification. All conversations must be recorded in the project manager phone log. Events that would require non-conformance reports are: inaccurate reports, incomplete reports or that the client is requesting additional information. At Katahdin, such inquiries are handled as client “complaints”. The Project Manager shall obtain all of the pertinent information from the client.

When the client “complaint” requires a report to be reissued, it will be documented on the team viewable document titled Reissues. The reason for reissue and whether the reissue is required due to a client additional request or a laboratory issue will be recorded. Additionally, the suspected cause may be included. All reissues will be reviewed during the next daily status meeting to determine if the cause of the reissue is reoccurring and a corrective action report needs to be initiated.

When the client "complaint" concerns a data quality issue, especially one that may impact the usability of data, the President, or the QAO **must** be informed. They will help to determine what corrective action, if any, should be taken.

If a complaint or other event raises doubt concerning the laboratory's compliance with its policies, or otherwise concerning the quality of the data, the QAO, and President, shall ensure that the areas involved are promptly reviewed and/or audited to determine the appropriate course of action.

Client complaints which do not require a reissued report are filed with the QAO.

15.2 Corrective Action

In the event that an analyst is unable to achieve return to control of a non-conformance, he/she notifies their department manager. If the department manager and analyst are unable to achieve return to control, the department manager shall notify the President and/or the Quality Assurance Officer. The President, Quality Assurance Officer and the technical team are responsible for identifying the source of the problem and achieving return to control.

The QAO has the authority to halt the affected work until a non-conformance is corrected.

When evaluation of non-conforming work indicates that a problem has not been corrected, that a problem could recur, or has recurred, or if there is concern that a major problem or potential problem exists in the laboratory, a Corrective Action Report (CAR) (Form QA-009)(Figure 15-2) must be initiated. The CAR will document the issue and the investigation to attempt to determine the root cause(s) of the problem.

15.2.1 Root Cause Analysis

Root cause analysis requires an investigation into the possible causes of the problem. Katahdin uses the six "M's" to investigate the possible causes. They are: Machine, Method, Materials, Maintenance, Man and Mother Nature. These categories will lead the investigation to consider such causes as malfunctioning instrumentation, inadequate instrumentation, inadequate training, inadequate processes, or laboratory accidents. All possibilities should be considered. Once a root cause(s) has been determined, a plan for corrective action must be determined.

15.2.2 Corrective Action

Corrective actions must be well thought out and take into consideration long-term actions to reduce or eliminate the chance of recurrence. Corrective actions shall identify needed improvements or additional training. In all cases the corrective actions must be appropriate to the magnitude and the risk of the problem. Narration may be appropriate in many cases, but more involved non-conformances may require halting of work, resampling, modification of the method, etc.

CARs are initiated on the company server. The President, QA Officer, and the Department Managers have access to this system. If an employee wishes to initiate a CAR, they may take the request to the appropriate manager. The CARs are sequentially numbered for tracking purposes.

15.2.3 Monitoring of Corrective Action

All corrective action reports must be reviewed by the appropriate management personnel (President, Department Manager) and by the QAO. Consistency and compliance with Katahdin policies and the effectiveness of the corrective actions must be reviewed.

The QAO shall approve or reject corrective action reports and/or may require additional corrective actions. The QAO will follow up at a reasonable time to determine whether the process has returned to control or whether further monitoring or additional corrective action is needed. The CAR shall indicate this and any further actions needed. A new CAR may be generated at this time with further corrective actions. This should be noted so that there is a traceablility of the related CARs.

All documentation associated with the CAR, i.e. raw data or reissued reports shall be filed with the associated samples. A copy of the CAR shall also be filed with the samples. The original CAR shall be filed in the QAO's office.

15.2.4 Preventive Action

Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints. Preventive action will identify and attempt to correct a problem before it occurs. Some examples of tools used to identify potential preventive actions are control charts and internal audits. Any employee may initiate a Preventive Action Form, QA-010, with suggestions for an improvement to a process, method, system, etc. in order to avoid potential non-conformances. The form shall be reviewed by the affected managers and implemented if deemed appropriate. Once initiated, QA follow-up is necessary to document the effectiveness of the actions.

Refer to the current revision of Katahdin SOP QA-803, Laboratory QA: Self-Inspection System for further details on non-conformance and corrective action practices.

FIGURE 15-1

KATAHDIN ANALYTICAL SERVICES NON-CONFORMANCE REPORT

KATAHDIN ANALYTICAL SERVICES – ORGANICS NON-CONFORMANCE REPORT

Problem Identification (Person discovering problem) Name:					Date:
Affected Method:					
☐ LCS Failure ☐ Blank Contamination ☐ Poor Precision (RPD) ☐ Sample out of clock ☐ Retention Time Window	☐ Prep Error ☐ Hold Time Missed ☐ Surrogate Failure ☐ Tune Failure ☐ Lab Accident	☐ ICAL Failure ☐ Independent Std Failure ☐ CV Failure ☐ Matrix Spike Failure ☐ Spiking Error	☐ Discrepancy between results ☐ Chromatographic Interference ☐ Internal Standard Failure ☐ Sample Contamination ☐ Other		
Details:					
Work Orders/Samples Affected →					
Client					
Requirements and/or Procedures Not Met: Project QAPP(Q) / Method(M) / SOP(S) / DoD QSM(D) / Client (C) / Other:					
Significance of Non-Conforming Work:	---	---	---	---	---
Corrective Action can be taken within holding time					
Corrective Action cannot be taken within holding time					
Non-Conformance not related to holding time					
Approval of Significance (Dept. Mgr., Ops. Mgr. or QAO) – Initials & Date					
Client Contacted PM initials & Date					
Corrective Action:	---	---	---	---	---
Reanalyze with compliant QC					
Re-Extract/Re-Prep					
Report non-compliance w/Cover Letter/Narrative and/or flagging					
Date Corrective Action Completed					
By Whom?					
Re-analysis or Re-extraction results indicate?					
Approval of Corrective Action (Dept. Mgr., Ops. Mgr. or QAO) – Initials & Date					
Is Further Corrective Action including root cause analysis warranted (i.e recurring problems)?					
By Whom?					Date:

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FIGURE 15-2

KATAHDIN ANALYTICAL SERVICES - CORRECTIVE ACTION REPORT

CAR0XXX Page **1** of **3**

KATAHDIN ANALYTICAL SERVICES, LLC. – CORRECTIVE ACTION REPORT

Problem Identification (Person initiating CAR) Name:		Date:
☐ Discovered by Laboratory	☐ Discovered by Client (Complaint)	☐ Other
Details of Problem:		
Team	Facilitator: Lead: Members:	
Root Cause Investigation & Determination (To be completed by Department Manager, Operations Manager and/or QA Officer)		
Review the 6 "M's" below and investigate to determine whether one of them, or more than one, could be the cause of the problem.		
Possible Causes	Details	
Machine (Instrument, PC, Software, Support Equipment, Maintenance)		
Method (Process, SOPs, Informal procedures, Activities, Customs, Habits, Culture)		
Materials (Samples, Consumables, Standards, Gases)		
Metrics(Analyte lists, QC Limits, Reporting Limits, Trends in NCRs)		
Man (Activities, Capabilities, Communication, Training)		
Mother Nature (Climate, Accidents, Power issues)		

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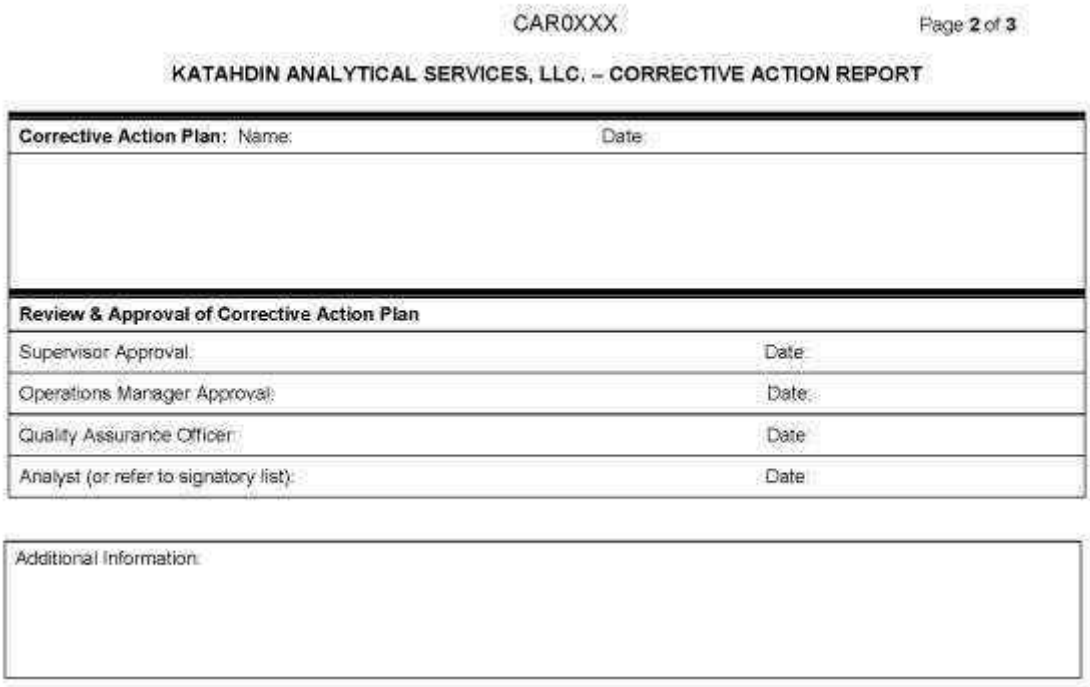
FIGURE 15-2, CONT'D

KATAHDIN ANALYTICAL SERVICES - CORRECTIVE ACTION REPORT



CAR0XXX Page **2** of **3**

KATAHDIN ANALYTICAL SERVICES, LLC. – CORRECTIVE ACTION REPORT

Corrective Action Plan: Name:	Date:
Review & Approval of Corrective Action Plan	
Supervisor Approval:	Date:
Operations Manager Approval:	Date:
Quality Assurance Officer:	Date:
Analyst (or refer to signatory list):	Date:

Additional Information:

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FIGURE 15-2, CONT'D

KATAHDIN ANALYTICAL SERVICES - CORRECTIVE ACTION REPORT

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

16.1 Management Review of the Quality Assurance Program

Review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. At a minimum, the President, Department Managers, Supervisors, Manager of Sales & Marketing Project Managers and QA Officer will meet at least annually to review the status of the laboratory. All aspects including operations, sales, marketing and quality assurance will be discussed. Any laboratory employee may present recommended changes to the Quality Assurance Officer or Department Managers at any time. Notes from these meetings and any actions taken are documented and retained by each member.

Additionally, any issues with the quality assurance program may be discussed during status meetings held daily. These meetings may be attended by the President, Department Managers, The Quality Assurance Officer, Project Managers, Sales & Marketing personnel or any other representative from the lab. The meetings focus on the status of work in house, upcoming projects, QA/QC problems, instrument problems, upcoming audits, PTs, etc.

16.2 Quality Assurance Documentation

At the start of a new year a QA & Operations Meeting is scheduled to review the previous year and to plan for the new year. The meeting shall include but is not limited to:

- Lessons learned and Areas for Improvement
- Client Complaints and/or Feedback
- Status of SOP review
- Status of LOD/LOQ Verifications and MDL studies
- PT Responses and Corrective Action
- Audit Responses and Corrective Action
    - Internal Audit
    - Upcoming Audits
- Corrective and Preventive Action recommendations
- Certifications
- New Methods
- Instrumentation
- Cross-training
- Training
    - EHSM

- Hazardous Waste
- Ethics
- Quality Control

- Outstanding Issues
- Changes that may affect certification and the notification of certifying states or LAB.
- Miscellaneous

This meeting is attended by the President, the Operations Manager, the IT/Production Manager, Department Managers, Group Supervisors, The Quality Assurance Officer, Project Managers, Sales & Marketing personnel or any other representative from the lab. Dates are set for any actions that need to take place. Subsequent meetings are attended by the pertinent management and a running memo is maintained by the Operations Manager to track the status of the issues discussed.

As with any corrective action, problem identification, investigation, and follow through must be completed and documented. These must be completed within 45 days of receiving the QA report, or as determined otherwise by the management team.

During any of these management activities changes for improvement may be suggested and reviewed. At a minimum, QA/QC procedures and policies shall be reviewed to determine their effectiveness and suitability.

17.0 REFERENCES

- "EPA Requirements for Quality Assurance Plans for Environmental Data Operations", U.S. EPA QA/R-5, August 1994
- "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", U.S. EPA, QAMS-005/80, EPA-600/4-83-004, February 1983
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, October 26, 1984.
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Act; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule" Federal Register, 40 CFR Part 122, 136, et al., March 12, 2007.
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for Biological Pollutants in Wastewater and Sewage Sludge; Final Rule", Federal Register 40 CFR Parts 136 and 503 March 26, 2007.
- "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Office of Solid Waste and Emergency Response, U.S. EPA.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, 1979 Revised 1983, U.S. EPA.
- "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA 600/R-93/100, August 1993, revision 2.
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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: Jeff C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA Soil Freezer Storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures 8 and 10. Updated Table and Figures to different ones. Added wording to Sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to calculation checks (not performed). Add IR gun usage. Reorganized section 7.C to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRC. Other minor changes. Added section concerning looking at coolers. Added more detail to 7.9 on unique container IDs. Added more detail on immediate COCs + a section on retention of samples.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
09	Added new log-in information for bottle IDs & screen attachment. Added procedures for bar code scanning for internal custody & deleted manual forms. Added new Incoming from KIMS & deleted old. Added form controlled forms to figures.	DR	9.24.10	09/10
10	Sect. 7 - Removed F9 function in printing labels, fixed how the lab ID appears on labels, and fixed how the date needs to be entered. Updated Fig.s 6 and 13.	LAD	08/13	08/13
11	Sect. 7 - Updated WC shirts and rushes from log-book to Google Docs, updated microbiological/food login process, updated bottle labeling. Updated Table 1 and Figures 1→7 & 12. Added Figure 17 - Sample Acceptance Policy	LAD	05/16	05/16
12	Sect. 7.15.4 - Added additional fields in login Info. Updated Table 1 Sect. 7.20 - Changed time held after preservation and before analyses from 16 to 24	LAB	09/17	09/17
13	Updated Figure 7 - Sample Receipt Condition Report	LAD	01/19	01/19

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-13**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-13**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., President, Department Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified, Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the QAO or President when warranted.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards,” current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS AND STANDARDS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 5). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "rush turn around" samples or "short hold-time" analyses. Notify the appropriate department Managers/Supervisors of these analyses.

7.4.1 Short hold-time analyses need to be entered into the "Wet Chemistry Shorts Spreadsheet" (Figure 6) on the company Google Docs system. Be sure to list the client, number of samples and date and time of the earliest sample.

7.4.2 GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion.

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7.4.3 Notify all applicable personnel of samples with limited hold-time remaining or rush turn around samples. Appropriate supervisors and PMs must be emailed when a client has requested rush results. The email should include the work order number, the client, the matrix, number of samples, analysis requested

and the turnaround time. Samples for microbiology lab should be brought to them immediately.

7.4.4 Parameters that we routinely analyze which have short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 7). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 8 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.11 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.

7.12 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Reports (generated from KIMS), quotes, memos, etc. These forms may provide details on specific client requests. The Incoming will provide information on specifics for log-in. Refer to Figure 11 for an example.

7.13 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.14 Samples requiring microbiological and/or food analyses are stored in the F/M laboratory walkin. For environmental tests, samples are logged in by the sample receipt department and a copy of the chain of custody is brought with the samples. For non-environmental microbiological tests, a workorder number is assigned by

sample receipt but the samples are not logged in. The workorder number, the chain of custody and a copy of the chain of custody are delivered with the samples. The samples are then logged in by the F/M staff. Sample that require both environmental and non-environmental microbiological analyses are usually processed the same as non-environmental samples

7.15 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 12) is generated for the samples received:

7.15.1 Log onto KIMS by entering employee ID under “Username”, employee specific password under “Password” and KIMS under “Database”.

7.15.2 Once logged onto KIMS select “Sample Management” and then “Login”.

7.15.3 Select “New” and the next available Login ID number will automatically be entered. Select “OK” and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select “change” and type in the appropriate number to access the information.

7.15.4 In the Sample Definition Screen, enter the following information.

Top Section of Screen:

Client ID - Enter client sample description.

ReceiveDate - Enter in date that samples were received in the lab in the format Day-Month-Year (ex. August 23, 2013 is 23-AUG-13).

CollectDate - Enter in date that samples were collected in the format Day-Month-YearTime (ex. 8:30am August 23, 2013 is 23-AUG-13).

TAT - Enter TAT for hardcopy report.

DueDate - Due date will automatically be calculated based on calendar days.

VerbalDate - Manually type in verbal due date.
QuoteRef - Enter quote number if applicable.

Project - Enter project number if applicable.

Account - Enter client specific account number.

Account Name - Account name will automatically be entered.

Collected By - Enter name/initials of sampler listed on COC. If unknown, enter “Client”.

Locator - May be used for client ID information when requested by the project manager.

Site - Enter project site name.

Description - May be used for food descriptions.

Discount - No entry-not currently used.

Priority - No entry-not currently used.

Fact. - No entry-not currently used.

Expected - No entry-not currently used.

Mailed - Data Management will enter the mailed date of the report or SDG right after the report is mailed.

Comments - Enter MS/MSD, verbal due date and any sample irregularities if applicable. Also may be used for long client IDs when requested by the project manager.

OrderDate - Current date is automatically entered.

Middle Section of Screen:

Highlight the first sample in the top section of the screen and then proceed with entries in the middle section of the screen.

Matrix - Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline
NOAQ = NonAqueous	TIS = Tissue
DW = Drinking Water	

Product Code -	Enter analysis code per test requested on COC. Log-in personnel should refer to Project Incomings, quotes or past Work Orders to aid in the entry of correct product codes.
Type -	Product code type will automatically be entered where S = Stand alone P = Parent C = Children
Fact. -	No entry-default is 1.
Price -	This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.
Cost -	No entry needed.
Lev -	No entry needed.
Container Type -	Container type will automatically be entered. Please change from the various choices if the automatic entry is not correct. This is especially important for volatiles in soil since there are many types of preservations.
Container Key-	Make sure “Container Type” is populated. Determine how many bottles there are for each container type. Assign bottles by entering sequential letters for each bottle. For example, sample 1 has six containers, one for metals which we’ll assign container ID, “A”, two for PCBs which we’ll assign container IDs, “B” and “C”, and three for volatiles which we’ll assign container IDs, “D”, “E”, and “F”. The letters should be typed in all in a row with no commas or spaces in between. If 26 bottles per samplenum are exceeded the next ‘key’ would be, ‘A1’, ‘B1’ etc. If no container IDs are needed (i.e. for food or field) it is okay to leave the container key field blank. After the Container Keys are entered click ‘SAVE’. This will create the containers section in the bottom section of the screen. This will also initiate the creation of container labels.

Bottom Section of Screen:

Container # - The container ID numbers will automatically fill in for each analysis from the container key information above.

Container Type - The container types will automatically fill in for each analysis from the container key information above.

Current Location - The current location is automatically entered based on the analysis.

Cooler - Currently not used.

pH - Currently not used.

Temperature - Currently not used.

Seal - Currently not used.

Properly Preserved - Currently not used.

Comments - Comments on individual containers may be entered here, i.e. bubble in VOA vial. Comments regarding problems or breaks with internal custody scanning of bar codes are also automatically entered here.

Select Login Info tab at top of screen and proceed with entry:

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager - Initials of Katahdin person overseeing the project.

Client Project Manager – Name of client manager

Contract – Name or number of contract for the job.

Client PO# - Client purchase order.

Project - Project name.
Cooler Temperature - Temperature blanks or cooler temps.

Delivery Services - Method of delivery to the lab.

QC Level - QC Level of report

SDG ID - Sample Delivery Group ID if applicable.

SDG Status - Begin, Continue or End.

Analysis Instructions - PM will enter special instructions regarding project.

Report Instructions - PM will enter special instructions regarding project.

Regulatory List - Not used.

EDD Format - Specific KAS EDD format.

Login Initials – Initials of person that logged the work order in.

Check – Check number when client pays at sample delivery

Select “SAVE” and then “CANCEL”.

Addresses - Select “Addresses” and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under “Project/Account” and select the report to contact under “Address Type”. Select the appropriate boxes for report, report CC and invoice CC. Select “SAVE” and then “CLOSE”.

Refer to Figure 13 for a screen snapshot of the log-in process in KIMS. Log-in personnel should also refer to the current revision of Katahdin SOP, SD-918, KIMS Work Order Approval & Dispatching, for further hints on log-in.

7.15.5 To print the login report, select “Reports”, “Login” and “Login COC”. Enter login number under “Login Number”. Select “OK”, “Run Report” and then “Print”.

7.16 To print labels, select “Reports”, “Login” and “Labels”. Enter login number under “Login/Prelogin”, select “Background (IDXL) (this is the default)”. Select “OK” and then “Print”. After labels print out select “Cancel”.

Note: As stated in "container key" above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-1A, SC9001-1B, SC9001-1C

Example: One job containing two client samples with 2 different containers for each:

SC9002-1A, SC9002-1B, SC9002-2A, SC9002-2B

7.17 Print the Label Bottle Reference report (under reports tab) for a cross reference to use during labeling. This report will list the bottle type and products related to each Container ID.

7.18 Remove samples from cooler and place them on the counter. Organize them by site ID, in the order of the chain and then by sample analysis.

7.19 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.20 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation

discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 24 hours after preservation before sample preparation can begin.

7.21 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 9). The samples are filtered by the Metals Group or the Wet Chemistry Group depending on which group requires the filtered samples.

7.21.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.21.2 A vacuum pump is attached.

7.21.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.21.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.21.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.22 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.23 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 10).

7.24 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

40 mL vial, 125 ml plastic bottle and 4 oz jar labels will have to be placed vertically on the sample container instead of the standard horizontal placement. Additionally, label for 2 oz jars must be placed on the cover.

7.25 Scan the containers into the appropriate storage locations using the following steps. Note that non-environmental food samples are not scanned and are taken immediately to the food/microbiology lab for storage.

7.25.1 In KIMS, click on “containers”. This can also be done at the walk-in computer or on the “D” instrument computer in the VOA lab, depending on where you are storing samples.

7.25.2 Click on “transfer/update” then “transfer” and select. This will bring you to the screen where you scan your badge. **NOTE: make sure you keep your badge available for this**. Alternatively, at the walk-in computer, click on the check-in/check-out ICON. This will also bring you to the screen where you scan your badge.

7.25.3 Scan the barcode on your badge.

7.25.4 Pick “log-in”.

7.25.5 Pick “check-in”.

7.25.6 Select the location you are checking into, i.e. walk-in, VOA Walkin, etc.

7.25.7 The sample screen will now be open. Scan each sample, so that you hear a beep and the sample pops up on the screen. The program is set so that you can continuously scan each sample without having to click anything on the screen. The samples do not have to be scanned in numerical order.

7.25.8 Hit “done/save”.

7.25.9 Hit “close/cancel”. This will return you to the badge scanning screen.

Note: An internal custody report may currently be printed, per client request, by the MIS department.

7.26 Place samples in their designated storage locations. Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.26.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.26.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.26.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in “VOA Refrigerator 2” located in the Volatiles Laboratory.

7.26.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.26.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous and soil samples in VOA vials (except those which are preserved with D.I. water) are stored in “VOA walkin” in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a “paint can” and stored in the sample receipt walk-in.

7.26.6 Soil samples for volatile organics analyses (VOA) that are preserved with Laboratory Reagent Grade Water are stored in “VOA Freezer 1” in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented through the bar code system with respect to native samples. Internal chain-of-custody for extracts and digestates is documented on hardcopy batch sheets. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

7.27 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.28 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 14).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.29 When removing or returning a sample from its storage location, it must be scanned in or out using the bar code on the container.

7.29.1 In KIMS, click on “containers”.

7.29.2 Click on “transfer/update” then “transfer” and select.

7.29.3 This will bring you to the screen where you scan your badge. Alternatively, at the walk-in computer, click on the check-in/check-out ICON. This will also bring you to the screen where you scan your badge.

7.29.4 Scan the barcode on your badge.

7.29.5 Pick the department that you are bringing samples to or from.

7.29.6 Pick “check-in” or “check-out”.

7.29.7 For check-in, select the location you are checking into.

7.29.8 The sample screen will now be open. Scan each sample, so that you hear a beep and the sample pops up on the screen.

7.29.9 Hit “done/save”.

7.29.10 Hit “close/cancel”. This will return you to the badge scanning screen.

7.30 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 15) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. The Immediate Internal COC Logbook must always be consulted if there is ever a question about internal custody.

7.31 If there is an error (i.e. a sample was checked out, but not checked back, and you are trying to check it out), an error screen will pop up indicating who made the error. Take note of who made the error and click "accept bottle". This will allow you to continue, and a note will automatically be applied to the record. If you notice somebody making a lot of errors, please talk to them or let a manager know.

7.32 For samples that are consumed during analysis or preparation, i.e. extractables – either log the samples out and then rescan your badge and log them back in to "consumed" or remove the labels in the lab (when finished) and stick them to your lab coat and then return to scan them into "consumed".

7.33 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and rescan back in using the steps in 7.23.

7.34 After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated quarterly against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Environmental Health & Safety Manual, current revision.

Katahdin QA Manual, current revision

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

LIST OF TABLES & FIGURES

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TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES - AQUEOUS					
Acidity	SM 2310B, 305.1	100 mL	P,G	1,2	14 days
Alkalinity- Titrimetric	SM2320B, 310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1/350.2 SM4500NH3 B&H	100 mL	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, SM4500NH3 H	100 mL	P,G	1,3	28 days
Anions (F, Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (calculation from alkalinity)	SM4500-CO2 D				
Biochemical Oxygen Demand-Carbonaceous	SM 5210B, 405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	SM 5210B, 405.1	1 L	P,G	1	48 hours
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	SM4500-Cl E, 325.2	100 mL	P,G	1	28 days
Chlorine, Total Residual	SM4500-Cl G, HACH 8167	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	SM2120B, 110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	SM4500CN G, 335.1	100 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.4	100 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	SM4500-O G, 360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1,12	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F B/C, 340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	SM4500F C, 340.2	200 mL	P only	1	28 days
Free CO2	SM4500-CO2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2, SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	SM4500-NO3 F, 353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1, 3 OR 11	28 days
pH (Laboratory)	SM 4500H B 150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	SM4500-P E, 365.2	100 mL	P,G	1	48 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	SM 2540C, 160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	SM 2540D, 160.2	1 L	P,G	1	7 days
Solids-Settleable Solids (SS)	SM2540F, 160.5	1 L	P,G	1	48 hours
Solids-Total Solids	SM 2540B, 160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	SM 2540E, 160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	SM2540C/E, 160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance	SM2510B, 120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	ASTM D516-02, 375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	SM4500-S2 F, 376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	SM4500-SO3 B, 377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	SM2130B, 180.1	100 mL	P,G	1	48 hours
Volatile Fatty Acids	SOP CA-776	(2) 40 mL	VOA vial	17	14 days
ELEMENTAL ANALYSES - AQUEOUS					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	16	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES - AQUEOUS					
EDB, DBCP & 1,2,3-TCP	8011 & 504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP EPH	(2) 1000 mL	Amber Glass	1,12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1, 18	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Petroleum Range Organics	FL-PRO	(2) 1000 mL	Amber Glass	1,12	7days/40days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Total Petroleum Hydrocarbons	TX1005	(2) 40 mL	VOA vial	12	14days/14days
Extractable Total Petroleum Hydrocarbons	CT-ETPH	(2) 1000 mL	Amber Glass	1	7days/40days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days
PCB Congeners	8082	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2) 40 mL	VOA vial	1,11	14days
Chloropicrin	8011 Mod.	(2) 40 mL	VOA vial	1,8,9	14 days
HPLC ANALYSES – AQUEOUS					
HPLC-Explosives	8330A/B/ B Mod.	(2) 1000 mL	Amber Glass	1	7days/40days
GC/MS ORGANIC ANALYSES – AQUEOUS					
Acid Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles – Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables & (SIM)	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics & (limited SIM)	8260/8260 SIM	(3) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics	624	(3) 40 mL	VOA vial	1,8,9	14 days(~)
MICROBIOLOGICAL ANALYSES – AQUEOUS					
Coliform, Fecal (wastewater)	SM 9222D	100 mL	P,G	1,6	6 hours
Coliform, Fecal (wastewater)	Colilert-18 w/ Quantitray	100 mL	P,G	1,6	6 hours
Coliform, Total (wastewater)	SM 9222B	100 mL	P,G	1,6	6 hours
Coliform, Total (drinking water)	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total (drinking water)	SM9223B, Colitag	100 mL	P,G	1,6	30 hours
E-coli (wastewater)	SM9213D	100 mL	P,G	1,6	6 hours
E-coli (wastewater)	SM9223B Colilert w/ Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B, SIMPlate	100 mL	P,G	1,6	8 hours for compliance samples, 24 for non-compliance samples

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES – SOLID					
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1/350.2 SM4500NH3 B&H mod.	4 oz	Soil Jar	1	28 days (^)
Anions (F, Cl, Br, NO3, NO2, SO4)	9056	4 oz	Soil Jar	1	48hrs to 28 days (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/9056	4 oz	Soil Jar	1	28days (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	SM4500F B/C, 340.2 mod.	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	9056 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Nitrite-Automated Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	28 days (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	9056 mod./365.2	4 oz	Soil Jar	1	48 hrs (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	SM2540 G, current CLP SOW	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9038	4 oz	Soil Jar	1	28 days (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Carbon	9060	4 oz	Soil Jar	1	28 days
Total Organic Carbon	Llyod Kahn	4 oz	Soil Jar	1	14 days
Total Organic Carbon	Walkley Black	4 oz	Soil Jar	1	14 days
ELEMENTAL ANALYSES – SOLID					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS Elements	6020	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES – SOLID					

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Extractable Petroleum Hydrocarbons	MADEP EPH	4 oz	Soil Jar	1	14days/40days
Fuel Oil	ME HETL 4.1.25 & 8015 mod.	4 oz	Soil Jar	1	14days/40days
Petroleum Range Hydrocarbons	FL-PRO	4 oz	Soil Jar	1	14days/40days
Total Petroleum Hydrocarbons	TX1005	4 oz	Soil Jar	1	14days/14days
Extracted Total Petroleum Hydrocarbons	CT-ETPH	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17 & 8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2)40 mL	VOA vial	1,13	28days
HPLC ANALYSES – SOLID					
HPLC-Explosives	8330B/B Mod.	4 oz or ISM sample	Soil Jar	1	14days/40days
GC/MS ANALYSES – SOLID					
Acid Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables & (SIM)	8270/8270 SIM	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics – Low Soil (<200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics & (limited SIM)	8260/8260 SIM	(2) 40 mL	VOA Vial	1	14 days
Miscellaneous – SOLID					
Grain Size (sieve and hydrometer)	ASTM D422	8 oz	Soil jar or bag	1	none
RCRA – HAZARDOUS WASTE CHARACACTERIZATION					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311/8260	100 g	Soil Jar	1	14 days/14 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
TCLP Extraction-Semivolatiles	1311/8270	200 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Pesticides & Herbicides	1311/8081 & 8151	400 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Metals	1311/6010/6020	200 g	Soil Jar	1	28 days/180 days
TCLP Extraction-Mercury	1311/7470	200 g	Soil Jar	1	28 days/28 days
GC/MS ANALYSES – AIR					
Volatile Organics	TO-15	(1) 1.4 or 6 L	Canister	16	30 days
Volatile Organics	MA-DEP APH	(1) 1.4 or 6 L	Canister	16	30 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NznAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate
16 = None
17 = benzalkonium chloride
18 = 0.02g ammonium sulfate, 0.02g copper (II) sulfate pentahydrate

~ Hold time for unpreserved samples is 7 days.

^ Because there are no published holding times for Wet Chemistry soil methods, these are only recommended holding times. They are not regulatory.

Project-specific (i.e. CLP, NYSDEC) hold times take precedence over these hold times as appropriate.

For solid samples, please place parameters of the same analytical group (ie. wet chemistry) in the same container whenever possible. In addition, organic and inorganic parameters should be placed in separate containers. Volatile organics should always be placed in organic-free jars. Several 4 oz. soil jars may be needed when numerous parameters are required.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin ANALYTICAL SERVICES
400 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client	Contact	Phone # ( )	Fax # ( )
Address	City	State	Zip Code
Purchase Order #	Proj. Name / No.		Katahdin Quote #
Bill (if different than above)	Address		
Sampler (Print / Sign)			Copies To:

LAB USE ONLY | WORK ORDER #:
KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: _____

TEMP °C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE / PRESERVATIVES

*	Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF KATAHDIN HOMEOWNER CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Drinking Water Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

***Test results are for compliance and will be reported to the state (see statement below) yes no Compliance samples may need to be received on ice

Lab Use Only | Work Order #: | KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Requested Services

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs	Standard Homeowner	Arsenic	Total Coliforms & E-coli	Lead (1st draw)	Safety Test – coliform & Nitrate	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test
													Standard Homeowner: Total Coliform/e-coli, Nitrate, Nitrite, Chloride, pH, Hardness, Uranium, Copper, Iron, Lead, Manganese, Sodium, Arsenic
													FHA/MSH: Standard plus Lead(1st draw), Turbidity, Color, Odor

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at temperatures specified in the regulations. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements. All services shall be governed by Katahdin's standard terms and conditions.

QA-059 – Revision 2 – 03/31/2016

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF KATAHDIN FOOD/MICROBIOLOGY CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #:	KAS Project Manager:	Food & Microbiological Services
Shipping: UPS Fed-Ex	Airbill No.:		
Temperature:			

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (APHA)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

The terms and conditions on the following page hereof shall govern services, except when a signed contractual agreement exists.

QA-058 – Revision 1 – 09/22/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN AIR CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Air Analysis Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #	KAS Project Manager:	Requested Services
Shipping:	UPS	Fed-Ex	Mail	Drop-Off	

Sample Description (Sample Identification and/or Lot #)	Collection					Matrix	Sampler	Can Size	Can ID	Flow Controller ID				Comments
	Date	Start Time	End Time	Initial Vac	Final Vac									

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Katahdin inspects and verifies all equipment including, but not limited to, canisters and flow controllers before being sent to the client. As the client you have agreed to pay a rental fee for use of this equipment, which is the sole property of Katahdin. All equipment will be inspected for damage and completeness upon return to Katahdin. In the event that rental equipment is missing and/or damaged, by signing this COC, you (the client) agrees to pay Katahdin for replacement of any unuseable, damaged or missing equipment.

QA-132 – Revision 1 – 02/06/2014

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBOOK

KATAHDIN ANALYTICAL SERVICES, LLC.
SAMPLE LOG IN

pH Paper Lot #: HC 564756

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
5/13/16	1345	5-13-16	1400	SJ 3311	Harmon's	
5/13/16	1350	5-13-16	15:00	SJ 3312	Bristol Seafood	
↓	↓			SJ 3313	Camp Sunshine	↓
5-13-16	14:00			SJ 3314	DEP-B	
				SJ 3315	↓	
				SJ 3316	DEP-A	
				SJ 3317		
			15:30	SJ 3318	FGS	
				SJ 3319	CES	
				SJ 3320		
				SJ 3321		
				SJ 3322	SWCole	
		5/13/16	16:00	SJ 3323	Clearwater	
				SJ 3324		
				SJ 3325		
				SJ 3326		
				SJ 3327		
				SJ 3328		
				SJ 3329		
				SJ 3330		
				SJ 3331		
				SJ 3332		
				SJ 3333		
				SJ 3334	PWD	open
		5-13-16	16:40	SJ 3335	Cape Elizabeth Tax	
5-13-16	15:00			SJ 3336	MEL	
				SJ 3337		
5-13-16	15:35			SJ 3338	PWD	
5/13/16	1540			SJ 3339	Maine Medical	
				SJ 3340	COLM	
↓	↓			SJ 3341	↓	↓

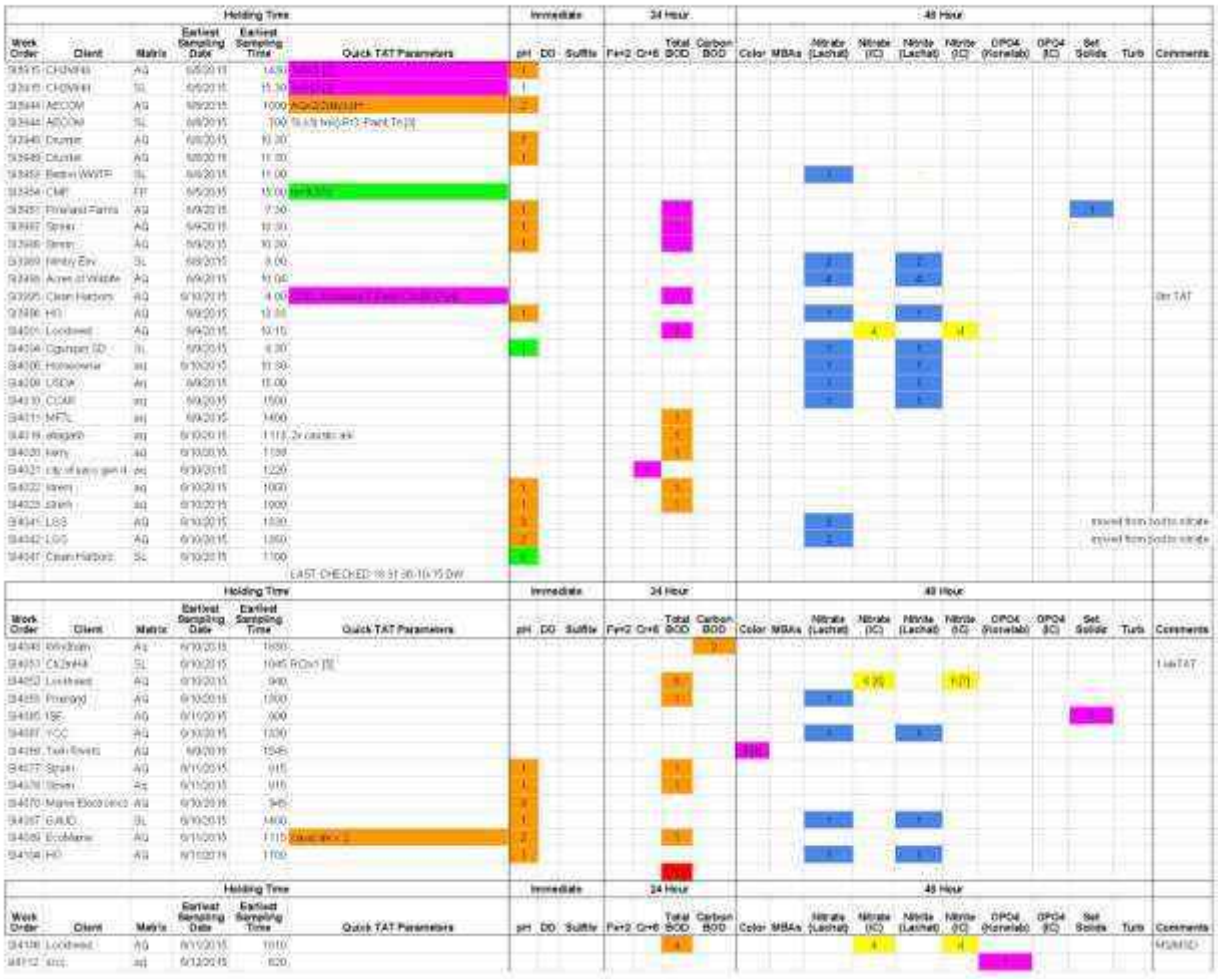
QA-032 - Revision 1 - 12/30/2009 Updated: 04/26/2016

QAQC793 0000011

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES SCREEN SHOT



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, LLC. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C): Thermometer ID: IR-1
Samples received at <6 °C w/o freezing?					Note: Not required for metals (except Hg soil) analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) or insufficient ice may not meet certain regulatory requirements and may invalidate certain data.
If yes, was there sufficient ice to meet temperature requirements?					
If temp. out, has the cooling process begun (i.e. ice or packs present) and sample collection times <6hrs., but samples are not yet cool?					Note: No cooling process required for metals (except Hg soil) analysis.
6. Volatiles:					
Aqueous: No bubble larger than a pea?					
Soil/Sediment:					
Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
D.I. Water - Received within 48 hour HT?					
Air: Refer to KAS COC for canister/flow controller requirements	√ if air included				
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2					
Sulfide - >9					
Cyanide – pH >12					
11. Bottleware Prepped on:					

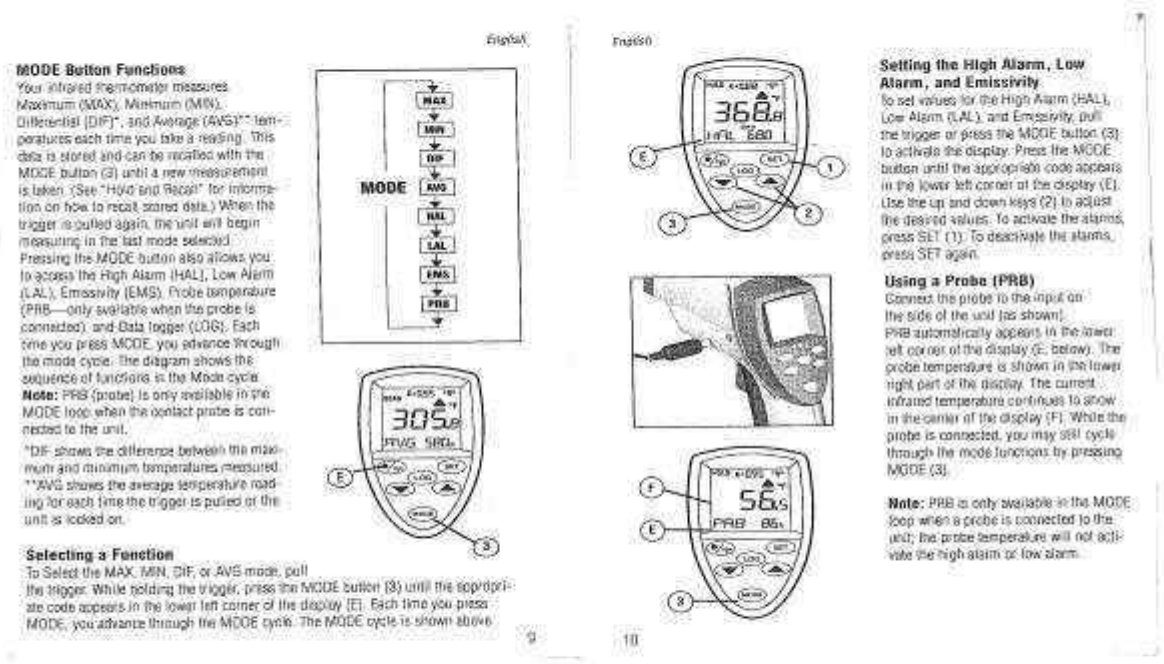
* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments.

QA-048 – Revision 7 – 09/26/2018

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY



English

MODE Button Functions

Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.

Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

MODE
MAX
MIN
DIF
AVG
HAL
LAL
EMS
PRB

Selecting a Function

To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.

9

English

Setting the High Alarm, Low Alarm, and Emissivity

To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)

Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

10

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No. List Individually	Site ID (Optional)	Filtration Requested By:		Filtered and Preserved By:		
		Initials	Date	Initials	Date	Time

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KIMS LABORATORY INCOMING SAMPLE REPORT

Katahdin
ANALYTICAL SERVICES

INCOMING SAMPLE REPORT

Quote: ELM001 Account: ELM001 Project:
Company: [redacted]
Name: [redacted]
Address: [redacted]
Quote Date: 29-MAR-10 Expires:
Date Expected: 28-SEP-40
Email: [redacted]

Notes: 8-aqueous VOA, 4-MBE,22-TOC, 44-metals (Total & Dissolved), 22-alkalinity, 12-Chloride,Nitrate, Nitrite, Sulfate

Analysis Notes: Merge Results for EDD

Report Notes: Email pdf and EDD to maltmayer@elmllc.com and tmarr@elmllc.com, Mail rpt and EDD on CD, no NC, metals need to be rpt ug/l. Down load rpt to FTP site for ELM. Hold Rpt Till Payment See Daphne

Description:
Project Name: Pilot Test [redacted] Client PO:
QCLevel: II Tat: 10 Terms: Reg List: Edd: KAS064-XLS

Product	Matrix	Quant	STD or Special Lists	Short	Unit Price	Total Price
8325.2-CHLORIDE	AQ	1	STD		40	40
8353.2-NITRATE	AQ	1	STD	SHORT	0	0
8353.2-NITRITE	AQ	1	STD	SHORT	0	0
8375.4-SULFATE	AQ	1	STD		0	0
RSKSOP175-MEE	AQ	1	STD		85	85
SM5310B-TOC	AQ	1	STD		25	25
SW3010-SREP	AQ	1	STD		0	0
SW6010-ARSENIC	AQ	1	STD		60	60
SW6010-ARSENIC-DIS	AQ	1	STD		60	60
SW6010-CALCIUM	AQ	1	STD		0	0
SW6010-CALCIUM-DIS	AQ	1	STD		0	0
SW6010-IRON	AQ	1	STD		0	0
SW6010-IRON-DIS	AQ	1	STD		0	0
SW6010-MAGNESIUM	AQ	1	STD		0	0
SW6010-MAGNESIUM-DIS	AQ	1	STD		0	0
SW6010-MANGANESE	AQ	1	STD		0	0
SW6010-MANGANESE-DIS	AQ	1	STD		0	0
SW6010-POTASSIUM	AQ	1	STD		0	0
SW6010-POTASSIUM-DIS	AQ	1	STD		0	0
SW6010-SODIUM	AQ	1	STD		0	0
SW6010-SODIUM-DIS	AQ	1	STD		0	0
SW8260FULL_10	AQ	1	STD		115	115
					8	385.00

History: _____
Other: _____

Printed: 21-SEP-10 1 of 1

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Login Chain of Custody Report (lno1)

Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services
Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: 1
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

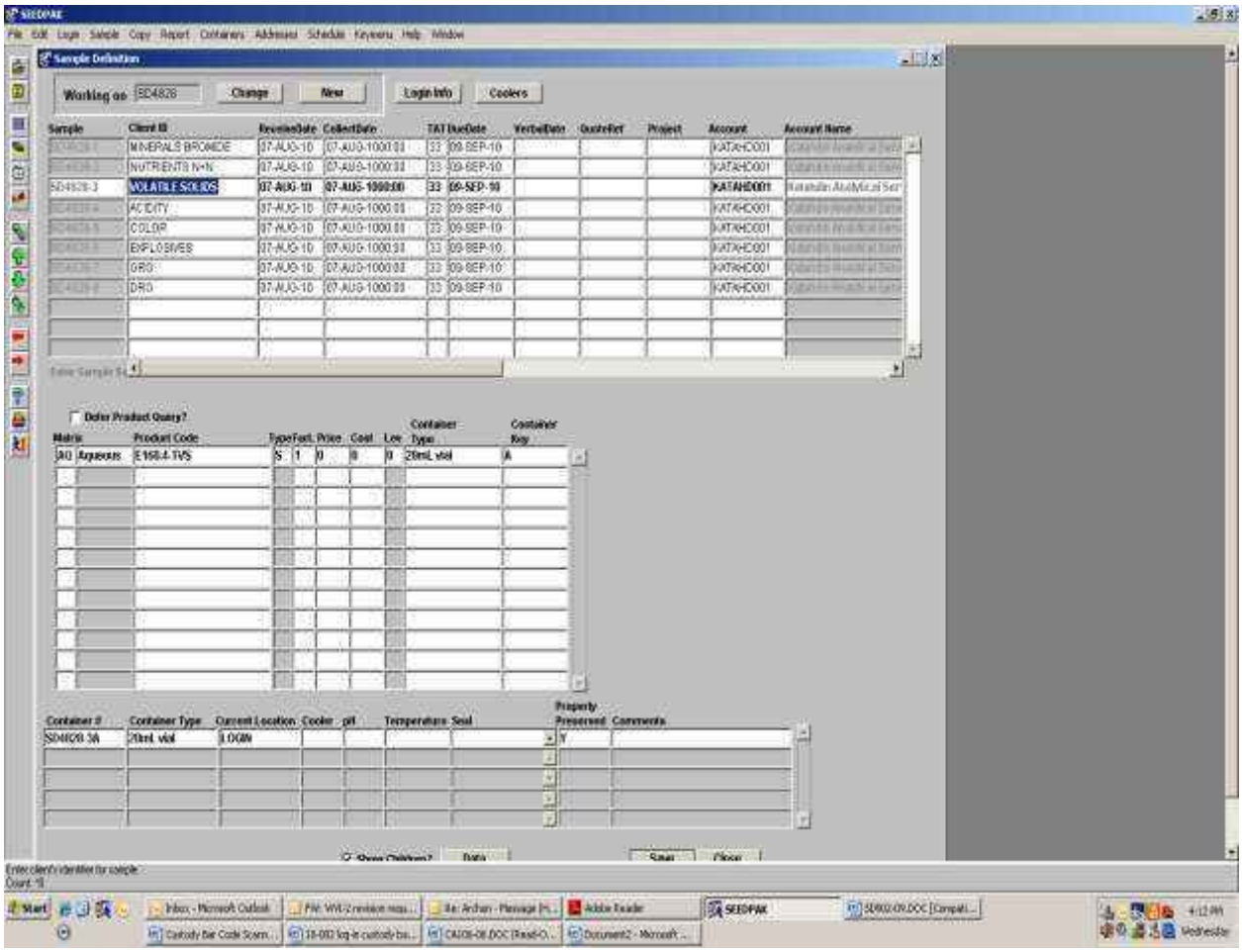
Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			09-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260C-L LML	09-FEB-07			3		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			09-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260C-L LML	09-FEB-07			3		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF LOGIN SCREEN IN KIMS



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

Sample Receipt Refrigerator and Freezer Temperature Logbook

Corrective Action: If acceptance criteria are not met, notify the QAO or your supervisor immediately to determine corrective action to be taken. Document the corrective action in the Comments section.

Thermometer Location		Sample Receipt Refrigerator 1	Sample Receipt Freezer 1	Comments
Acceptance Criteria		Above 0 to 6 °C	< -10 °C	
Date	Initials	Temp (°C)	Temp (°C)	

QA-069 – Revision 1 – 06/12/2013

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 15

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

QA-046 – Revision 1 – 04/15/2010

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no

0000001

FIGURE 16

SAMPLE ACCEPTANCE POLICY

Katahdin Analytical Services Sample Acceptance Policy

Katahdin Analytical Services reserves the right to refuse any samples due to any anomalies, discrepancies or non-compliances concerning the receipt and/or analysis of samples. These may include but are not limited to:

- Insufficient sample volume
- Insufficient remaining holding time
- Health or safety risks the samples may pose, including radioactivity
- Insufficient experience to handle sample or analysis
- Improper or illegible labeling of samples
- Improper sample containers
- Insufficient documentation including sample identification, location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample
- Damaged, contaminated or inadequately preserved samples

Any decisions to reject samples are made with the client's input.

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Joel C. Benton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution~~ prevention, added updated log-book and girater detail on dis-posal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regu-lations & to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509
05	Sect. 7 - Added non-hazardous samples are recycled, added PCB information, changed elementary neutralization target pH to 5-~~8~~9. Added wording for clarification. Updated Figures 1, 3 and 5.	CMD	06/13	06/13

TITLE: SAMPLE DISPOSAL

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Corrected typos, updated Figure 3	LAD	09/17	09/17

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-06**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-06**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A “Solid Waste” which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined,

to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory President.

It is the responsibility of the Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory President.

It is the responsibility of the Laboratory President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS AND STANDARDS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handling samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles (VOA)
VOA (SL)	Solid Volatiles (VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous liquid samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples and their containers are disposed of with the recycling trash, which is picked up by commercial trash collectors and ultimately turned into construction material.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream “K”. Containers are disposed of with general trash. Solid samples are placed into waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream “A”. Containers are disposed of with general trash. Solid samples are placed in waste stream “L” with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)
Refer to section 7.10 below.

7.9.5 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “Q”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “F” with their containers. The disposal date is recorded in the SDL. Any PCB samples with PCB content 50 ppm or greater, solid or aqueous, are set aside for TCSA regulated disposal.

7.9.6 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “O”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “NHi”. Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.8 Miscellaneous Disposal (as determined by the preservative)

7.9.8.1 Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream “A”. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

7.9.8.2 Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the “O” waste stream. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. Lastly, samples marked “free product” on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium

hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 5 and 9. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Dumping Basic samples (as determined by the basic preservative or by laboratory testing). If the samples have been to be hazardous due solely to the corrosivity characteristic, they are included in the neutralization process above.

7.12 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a “lab pack” of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.13 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Environmental Compliance Officer and the Katahdin President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

Figure 1 Example of Sample Disposal Logbook
Figure 2 Example of KIMS Generated Waste Disposal Report
Figure 3 Example Of Hazardous Waste Area Daily Check Documentation
Figure 4 Characteristic Toxic Hazardous Waste and TCLP concentrations
Figure 5 Example of Elementary Neutralization Logbook

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

QA-022 - Revision 1 - 11/09/2009

QAQC645

0000012

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
56-1969-1	M	4-10-13		✓					✓				5-20-13	5-20-13
56-1970-1		4-9-13		✓										
56-1977-1		4-4-13	✓											
56-1978-1-22		4-18-13	1-10, 12-14 16-22		11,15									
56-1979-2-10		4-10-13	✓											
56-1990-1		4-4-13				✓								
56-1991-1	↓	4-4-13				✓							↓	↓
56-1002-1	WC	2-27-13	✓										5-21-13	5-21-13
56-1010-1		2-26-13	✓											
56-1017-1		2-21-13	✓											
56-1021-1		2-26-13	✓											
56-1022-1,2	↓	2-28-13	2	1									↓	↓

TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value	
SA6605-1		NEED	12/02/07			
SA6606-1		NEED	12/02/07			
SA6607-1		NEED	11/15/07			
SA6608-1		NEED	12/06/07	ORG	1.17	MG/L (HIGH)
SA6608-1		NEED	12/06/07			
SA6608-2		NEED	12/06/07	AA	13	MG/KG (HIGH)
SA6609-1		NEED	11/26/07			
SA6609-1		NEED	11/26/07			
SA6610-1		NEED	11/30/07			
SA6611-1	FCS-020	NEED	12/07/07			
SA6611-2	FCS-020	NEED	12/07/07			
SA6611-3	FCS-020	NEED	12/07/07			
SA6611-4	FCS-020	NEED	12/07/07			
SA6611-5	FCS-020	NEED	12/07/07			
SA6611-6	FCS-020	NEED	12/07/07			
SA6611-7	FCS-020	NEED	12/07/07			
SA6611-8	FCS-020	NEED	12/07/07			
SA6612-1	NSA-030	NEED	12/07/07			
SA6612-2	NSA-030	NEED	12/07/07			
SA6612-3	NSA-030	NEED	12/07/07			
SA6612-4	NSA-030	NEED	12/07/07	ORG	1.70735	MG/L (HIGH)
SA6612-5	NSA-030	NEED	12/07/07	ORG	1.0481	MG/L (HIGH)

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TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Month: _____, 20___

Item / Date:					
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" aisle?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No Initials)					
9. Time of Inspection					
10. Verification of Inspection (Name/Date)					

Deficiency noted:					
Corrective action:					
By (Name/Date):					

HW-007 – Revision 2 – 08/30/2017

0000029

TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 5-9-13		Time: 16:30	Analyst: GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	7		
5	6		
5	5		
5	7		

Date: 5-16-13		Time: 12:00	Analyst: GN/WS
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	7	good	
5	5		
5	7		
5	7		
6	5		
6	8		
5	7		
5	7		
4	6		

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date: 1.10.19

SOP Number: SD-903-06

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-11-19

QAO Signature: Leslie Dimond **Date:** 01.24.19

APPENDIX H
Community Air Monitoring Plan

SERES Engineering & Services, LLC
ARCADIS
a joint venture

COMMUNITY AIR MONITORING PLAN

Former Nike Anti-Aircraft Missile Battery BU-51/52
Launch Area Remedial Investigation
Hamburg, Erie County, New York
FUDS Project Number: C02NY0079

August 2020

COMMUNITY AIR MONITORING PLAN

Former Nike Anti-Aircraft Missile Battery BU-51/52
Launch Area Remedial Investigation
Hamburg, Erie County, New York
FUDS Project Number: C02NY0079

Prepared for:

U.S. Army Corps of Engineers, New England District
696 Virginia Road
Concord, Massachusetts 01742

Prepared by:

SERES-Arcadis JV, LLC
669 Marina Drive, Suite B-7
Charleston, South Carolina 29492

Date:

August 2020

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TABLES

Table 1 Community Air Monitoring Alert and Action Levels

ATTACHMENTS

1 NYSDOH Generic Community Air Monitoring Plan

1 INTRODUCTION

This Community Air Monitoring Plan (CAMP) has been prepared to support the Remedial Investigation (RI) at the former Nike Antiaircraft Missile Battery BU-51/52 (Nike Battery), a Formerly Used Defense Site (FUDS), located in Hamburg, Erie County, New York (site). This work is being performed under Contract No.: W912WJ-19-D-0004, Task Order No.: W912WJ19F0123 with the US Army Corps of Engineers – New England District (USACE). The Nike Battery includes the non-contiguous former Launch Area and former Control Area. The focus of the RI effort is the former Launch Area.

The purpose of this CAMP is to describe the monitoring activities that will be conducted by the SERES-Arcadis JV, LLC (JV) to monitor for potential airborne releases of constituents of concern (COCs) during ground-intrusive activities (i.e., drilling) conducted at the site. This CAMP specifies the air emission action levels, air monitoring procedures, monitoring schedule, and data collection and reporting to be performed during the implementation of the field activities.

1.1 Site Location and Description

The Nike Battery, located in Hamburg, New York, operated from 1956 until its closure in 1961 (USACE 1991). During that period, the Battery consisted of a Launch Area, a Control Area, and an Easement Area. The Launch Area contained the facilities and equipment required to assemble, test, and maintain the missiles and launchers. The Control Area contained the electronic equipment for target tracking, missile guidance, and missile fire control. In 1968, a 57-acre plot, which contained the former Nike BU-51/52 Battery area, was turned over to the Town of Hamburg, who is the current owner of the Nike Battery (USACE 1991). The former Launch Area is currently used by the Town of Hamburg's Highway Department for office space, truck garage space, and storage of gravel piles and road salt. The Town of Hamburg police department uses the former enlisted men's barracks along the southwestern property boundary for the special weapons and tactics (SWAT) team and a firearm training center with earthen backstop berm in the northwestern corner of the former Launch Area for target practice.

1.2 Summary of Activities

In general, the work will include the drilling of soil borings and the installation of monitoring wells at the site.

2 ODOR, VAPOR, AND DUST CONTROL

As defined in the New York State Department of Health (NYSDOH) Generic CAMP (included as Attachment 1), intrusive remedial activities to be performed at the site have the potential to generate localized impacts to air quality. Field activities that have the potential to generate air emissions include, but may not be limited to, the following:

- Drilling.
- Material handling (e.g., offloading of materials, stockpiling of materials, and loading of materials for transport to an off-site disposal facility).
- Site restoration.
- Other ancillary intrusive activities.

Odor, vapor, and dust emissions resulting from these activities will be controlled using a combination of the following:

- Water spray.
- Polyethylene sheeting (for covering material stockpiles).

Odor, vapor, and dust controls will be proactively utilized by the contractor during the work to:

- Prevent exceedances of the total volatile organic compounds (VOCs) and Particulate Matter of 10 microns in diameter or smaller (PM_{10}) action levels specified Section 3.4 of this CAMP.
- Mitigate odor emissions to the extent practicable and to the satisfaction of the USACE, JV, New York State Department of Environmental Conservation, and NYSDOH.

3 AIR MONITORING PROCEDURES

The community air monitoring program is intended to be a discrete program that will be operated in conjunction with the exclusion zone (i.e., work zone) air monitoring program. JV will conduct real-time community air monitoring throughout the RI. Monitoring will be conducted at representative locations at the perimeter of the work zone for VOCs and PM_{10}. However, particulate monitoring will not be performed during precipitation events.

Additional information regarding the monitoring locations, equipment, and action levels is presented below.

3.1 Monitoring Location Selection and Deployment

Monitoring will be conducted upwind and downwind of each work zone during work activities. Monitoring locations will be determined daily based the nature of the anticipated work activities.

3.2 Volatile Organic Compound Monitoring

Real-time monitoring for VOCs will be conducted at the site during investigation activities. A real-time VOC monitor equipped with a photoionization detector and calibrated per manufacturer's specifications,

will be used to monitor for VOCs. All measurements will be recorded using an electronic data logger and/or in the field logbook.

3.3 Total Suspended Particulate Monitoring

Real-time monitoring for PM_{10} will be conducted during investigation activities. A real-time PM_{10} monitor will be used for PM_{10} monitoring. All measurements will be recorded using an electronic data logger and/or in the field logbook.

Fugitive dust migration will be visually assessed during all work activities, and reasonable dust-suppression techniques will be used during any site activities that may generate fugitive dust (as discussed in Section 2).

3.4 Alert and Action Levels

Alert and action levels for VOCs and ambient air PM_{10} concentrations are provided below. Alert and action levels are to be used to initiate corrective actions, if necessary, based on real-time monitoring. Each piece of monitoring equipment will have alarm capabilities (audible and/or visual) to indicate exceedances of the alert levels. Table 1 summarizes the alert and action levels for total VOCs and PM_{10}. Additional details are provided in the following subsections.

Table 1. Community Air Monitoring Alert and Action Levels

Parameter	Alert Level	Action Level
Total VOCs	2.5 ppm	5.0 ppm
PM_{10}	100 µg/m^3	150 µg/m^3

Notes:

1. ppm – parts per million
2. µg/m3 – micrograms per cubic meter

3.4.1 Alert and Action Levels for VOCs

As outlined in the NYSDOH Generic CAMP (Attachment 1) if the ambient air concentration for VOCs exceeds 2.5 parts per million (ppm) above background (i.e., upwind location) work may continue. The contractor will attempt to identify the potential source of the exceedance and employ additional vapor, and/or dust controls, as necessary, to abate emissions.

If the ambient air concentrations for VOCs persist at levels in excess of 5 ppm but less than 25 ppm above background, all work activities will be halted, USACE will be immediately notified, the source of the elevated VOC concentrations identified, corrective actions to reduce or abate the emissions will be completed or modify construction techniques, as necessary, and continue air monitoring. Work activities may resume provided that the 15-minute average VOC concentration remains below the alert levels.

If the ambient air concentrations for VOCs exceed 25 ppm above background, work shall not resume until authorized by USACE.

3.4.2 Alert and Action Levels for PM_{10}

As outlined in the NYSDOH Generic CAMP (Attachment 1), if the ambient air concentration for PM_{10} exceeds 100 micrograms per cubic meter ($\mu g/m^3$) above average background (i.e., upwind location), or visible dust is observed leaving the work area, work may continue if dust suppression techniques are implemented. The contractor will attempt to identify the potential source of the exceedance and shall employ additional dust controls, or modify construction techniques, to abate emissions.

If the ambient air concentration for PM_{10} exceeds 150 $\mu g/m^3$ above average background, or visible dust is observed leaving the work area, the contractor shall stop all work activities, immediately notify USACE, identify the source of elevated PM_{10} concentrations, complete corrective actions to reduce or abate the emissions or modify construction techniques, as necessary, and continue air monitoring. Work activities may resume provided that the 15-minute TWA concentration remains below the action levels.

3.5 Odor Monitoring

During working hours, JV will conduct periodic walks around the perimeter of the work area(s) to monitor for odors originating from the intrusive activities. These perimeter checks will be performed more frequently, as necessary, depending on the work being performed and meteorological factors such as change in wind direction. Meteorological factors that can influence odor generation and dissemination generally include temperature, humidity, precipitation, atmospheric pressure, wind direction, and wind speed. These factors can work synergistically with a positive or negative impact on odor generation and transport/dispersion. For example, odors generally tend to be less prevalent with lower temperatures, precipitation, or high humidity. Additionally, odor dissemination is greatly influenced by wind direction and wind speed. Meteorological factors, including wind direction, will be monitored during the remedial construction activities.

If odors are noticed along the perimeter of the work area, work will continue and odor-, vapor-, and dust-suppression techniques employed to abate emissions.

Odor complaints (if any) will be directed to JV. The legitimacy of the complaint will be verified based on the work activities being performed, the predominant wind direction, and other meteorological factors. In response to verified odor complaints, perimeter monitoring will continue, and additional odor, vapor, and dust controls will be employed to abate emissions. Additionally, construction techniques will be evaluated and modified, if necessary and appropriate.

If odors continue to be noticed at the perimeter of the work area, work will be stopped while activities are reevaluated. The source or cause of the odors will be identified and additional modifications of construction techniques or additional methods to abate emissions will be implemented. Work will resume provided the measures are successful at abating the odors noticed along the work area perimeter

3.6 Instrument Calibration

JV shall calibrate air monitoring equipment daily (at a minimum), or other frequency recommended by the manufacturer. All instrument readings, field reference checks, and calibrations will be recorded in the field logbook.

4 MONITORING SCHEDULE AND REPORTING

The following subsections identify the monitoring schedule and data collection/reporting requirements.

4.1 Monitoring Schedule

Air monitoring will be conducted prior to initiation of the field activities/intrusive work to establish adequate baseline data and until such time that significant material handling activities are complete (i.e., removal of stockpiled materials for off-site transportation and disposal). As previously indicated, real-time VOC and PM_{10} monitoring will be performed during intrusive and/or potential dust-generating activities (e.g., excavation, backfilling, and material handling activities).

The frequency of air monitoring will be relative to the level of site work activities being conducted and may be adjusted as the work proceeds and in consideration of the monitoring results. Air monitoring for VOCs and PM_{10} may be discontinued during periods of heavy precipitation that would otherwise result in unreliable data or damage to the monitoring equipment.

ATTACHMENT 1

NYSDOH Generic Community Air Monitoring Plan

Appendix 1A
New York State Department of Health
Generic Community Air Monitoring Plan

Overview

A Community Air Monitoring Plan (CAMP) requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the downwind perimeter of each designated work area when certain activities are in progress at contaminated sites. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, the CAMP helps to confirm that work activities did not spread contamination off-site through the air.

The generic CAMP presented below will be sufficient to cover many, if not most, sites. Specific requirements should be reviewed for each situation in consultation with NYSDOH to ensure proper applicability. In some cases, a separate site-specific CAMP or supplement may be required. Depending upon the nature of contamination, chemical- specific monitoring with appropriately-sensitive methods may be required. Depending upon the proximity of potentially exposed individuals, more stringent monitoring or response levels than those presented below may be required. Special requirements will be necessary for work within 20 feet of potentially exposed individuals or structures and for indoor work with co-located residences or facilities. These requirements should be determined in consultation with NYSDOH.

Reliance on the CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

Community Air Monitoring Plan

Depending upon the nature of known or potential contaminants at each site, real-time air monitoring for VOCs and/or particulate levels at the perimeter of the exclusion zone or work area will be necessary. Most sites will involve VOC and particulate monitoring; sites known to be contaminated with heavy metals alone may only require particulate monitoring. If radiological contamination is a concern, additional monitoring requirements may be necessary per consultation with appropriate DEC/NYSDOH staff.

Continuous monitoring will be required for all ground intrusive activities and during the demolition of contaminated or potentially contaminated structures. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pitting or trenching, and the installation of soil borings or monitoring wells.

Periodic monitoring for VOCs will be required during non-intrusive activities such as the collection of soil and sediment samples or the collection of groundwater samples from existing monitoring wells. “Periodic” monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or

overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. In some instances, depending upon the proximity of potentially exposed individuals, continuous monitoring may be required during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

VOC Monitoring, Response Levels, and Actions

Volatile organic compounds (VOCs) must be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions, particularly if wind direction changes. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

1. If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

2. If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities must be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15-minute average.

3. If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.

4. All 15-minute readings must be recorded and be available for State (DEC and NYSDOH) personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

Particulate Monitoring, Response Levels, and Actions

Particulate concentrations should be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

1. If the downwind PM-10 particulate level is 100 micrograms per cubic meter (mcg/m^3) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed 150 mcg/m^3 above the upwind level and provided that no visible dust is migrating from the work area.

2. If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 mcg/m^3 above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 mcg/m^3 of the upwind level and in preventing visible dust migration.

3. All readings must be recorded and be available for State (DEC and NYSDOH) and County Health personnel to review.

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Appendix 1B
Fugitive Dust and Particulate Monitoring

A program for suppressing fugitive dust and particulate matter monitoring at hazardous waste sites is a responsibility on the remedial party performing the work. These procedures must be incorporated into appropriate intrusive work plans. The following fugitive dust suppression and particulate monitoring program should be employed at sites during construction and other intrusive activities which warrant its use:

1. Reasonable fugitive dust suppression techniques must be employed during all site activities which may generate fugitive dust.

2. Particulate monitoring must be employed during the handling of waste or contaminated soil or when activities on site may generate fugitive dust from exposed waste or contaminated soil. Remedial activities may also include the excavation, grading, or placement of clean fill. These control measures should not be considered necessary for these activities.

3. Particulate monitoring must be performed using real-time particulate monitors and shall monitor particulate matter less than ten microns (PM10) with the following minimum performance standards:

(a) Objects to be measured: Dust, mists or aerosols;
(b) Measurement Ranges: 0.001 to 400 mg/m3 (1 to 400,000 :ug/m3);
(c) Precision (2-sigma) at constant temperature: +/- 10 :g/m3 for one second averaging; and +/- 1.5 g/m3 for sixty second averaging;
(d) Accuracy: +/- 5% of reading +/- precision (Referred to gravimetric calibration with SAE fine test dust (mmd= 2 to 3 :m, g= 2.5, as aerosolized);
(e) Resolution: 0.1% of reading or 1g/m3, whichever is larger;
(f) Particle Size Range of Maximum Response: 0.1-10;
(g) Total Number of Data Points in Memory: 10,000;
(h) Logged Data: Each data point with average concentration, time/date and data point number
(i) Run Summary: overall average, maximum concentrations, time/date of maximum, total number of logged points, start time/date, total elapsed time (run duration), STEL concentration and time/date occurrence, averaging (logging) period, calibration factor, and tag number;
(j) Alarm Averaging Time (user selectable): real-time (1-60 seconds) or STEL (15 minutes), alarms required;
(k) Operating Time: 48 hours (fully charged NiCd battery); continuously with charger;
(l) Operating Temperature: -10 to 50° C (14 to 122° F);
(m) Particulate levels will be monitored upwind and immediately downwind at the working site and integrated over a period not to exceed 15 minutes.

4. In order to ensure the validity of the fugitive dust measurements performed, there must be appropriate Quality Assurance/Quality Control (QA/QC). It is the responsibility of the remedial party to adequately supplement QA/QC Plans to include the following critical features: periodic instrument calibration, operator training, daily instrument performance (span) checks, and a record keeping plan.

5. The action level will be established at 150 ug/m3 (15 minutes average). While conservative,

this short-term interval will provide a real-time assessment of on-site air quality to assure both health and safety. If particulate levels are detected in excess of 150 ug/m3, the upwind background level must be confirmed immediately. If the working site particulate measurement is greater than 100 ug/m3 above the background level, additional dust suppression techniques must be implemented to reduce the generation of fugitive dust and corrective action taken to protect site personnel and reduce the potential for contaminant migration. Corrective measures may include increasing the level of personal protection for on-site personnel and implementing additional dust suppression techniques (see paragraph 7). Should the action level of 150 ug/m3 continue to be exceeded work must stop and DER must be notified as provided in the site design or remedial work plan. The notification shall include a description of the control measures implemented to prevent further exceedances.

6. It must be recognized that the generation of dust from waste or contaminated soil that migrates off-site, has the potential for transporting contaminants off-site. There may be situations when dust is being generated and leaving the site and the monitoring equipment does not measure PM10 at or above the action level. Since this situation has the potential to allow for the migration of contaminants off-site, it is unacceptable. While it is not practical to quantify total suspended particulates on a real-time basis, it is appropriate to rely on visual observation. If dust is observed leaving the working site, additional dust suppression techniques must be employed. Activities that have a high dusting potential--such as solidification and treatment involving materials like kiln dust and lime--will require the need for special measures to be considered.

7. The following techniques have been shown to be effective for the controlling of the generation and migration of dust during construction activities:

(a) Applying water on haul roads;
(b) Wetting equipment and excavation faces;
(c) Spraying water on buckets during excavation and dumping;
(d) Hauling materials in properly tarped or watertight containers;
(e) Restricting vehicle speeds to 10 mph;
(f) Covering excavated areas and material after excavation activity ceases; and
(g) Reducing the excavation size and/or number of excavations.

Experience has shown that the chance of exceeding the 150ug/m3 action level is remote when the above-mentioned techniques are used. When techniques involving water application are used, care must be taken not to use excess water, which can result in unacceptably wet conditions. Using atomizing sprays will prevent overly wet conditions, conserve water, and provide an effective means of suppressing the fugitive dust.

8. The evaluation of weather conditions is necessary for proper fugitive dust control. When extreme wind conditions make dust control ineffective, as a last resort remedial actions may need to be suspended. There may be situations that require fugitive dust suppression and particulate monitoring requirements with action levels more stringent than those provided above. Under some circumstances, the contaminant concentration and/or toxicity may require additional monitoring to protect site personnel and the public. Additional integrated sampling and chemical analysis of the dust may also be in order. This must be evaluated when a health and safety plan is developed and when appropriate suppression and monitoring requirements are established for protection of health and the environment.

