FINAL
QUALITY ASSURANCE PROJECT PLAN

Remedial Investigation
Former NIKE PR-79 Control Area
Foster, Rhode Island
DERP-FUDS D01RI0063/02

Version Number: 3

Prepared For:

Department of the Army
US Army Corps of Engineers
New England District
696 Virginia Road
Concord, MA 01742-2751

Contract Number: W912WJ-19-D-0003
Delivery Order Number: W912WJ19F0110

Prepared By:

AECOM

AECOM
250 Apollo Drive
Chelmsford, MA 02184-3267

September 11, 2020

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Prepared under:

Contract Number: W912WJ-19-D-0003
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Review Signature:  9/11/2020

Gregory Hencir, Task Order Manager
AECOM
Date

Approval Signature:  9/11/2020

Mark Kauffman, Program Manager
AECOM
Date

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ACRONYMS AND ABBREVIATIONS

%	percent
%RSD	percent relative standard deviation
µg/L	micrograms per liter
°C	degrees Celsius
95UTL	95% upper tolerance limit
ADR	Automated Data Review
AECOM	AECOM Technical Services Inc.
AE IDIQ	Architect-Engineering Indefinite Delivery/Indefinite Quantity
AFFF	aqueous film forming foam
AMEC	AMEC Environment & Infrastructure, Inc.
ANL	Argonne National Laboratory
AOC	Area of Concern
APP	Accident Prevention Plan
AR	Administrative Record
ARAR	Applicable or Relevant Appropriate Requirements
ATV	Acoustic Televiewer
AWQCs	Ambient Water Quality Criteria
BFB	4-Bromofluorobenze
BGS	below ground surface
BS	blank spikes
BTV	background threshold values
CB	Calibration Blank
CCV	Continuing Calibration Verification or Continuing Calibration
CDM	Camp Dresser & McKee, Inc.
CENAB	USACE, Baltimore District
CENAE	USACE, New England District
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CERCLIS	Comprehensive Environmental Response, Compensation, and Liability Information System
CFR	Code of Federal Regulations
CHMM	Certified Hazardous Materials Manager
cis-1,2-DCE	cis-1,2-dichloroethene
CLP	Contract Laboratory Program
COPC	constituent of potential concern
CoC	Chain of Custody

CPR	cardiopulmonary resuscitation
CSM	conceptual site model
CSP	Certified Safety Professional
CVOCs	chlorinated volatile organic compounds
DERP	Defense Environmental Restoration Program
DFTPP	decafluorotriphenylphosphine
DL	detection limit
DO	dissolved oxygen
DoD	Department of Defense
DoE	Department of Energy
DOT	Department of Transportation
DQI	Data Quality Indicator
DQO	data quality objective
DU	Decision Unit
EB	equipment blank
EDQW	Environmental Data Quality Workgroup
ELAP	Environmental Laboratory Accreditation Program
EMI	Environmental Measurement Information
EPC	Exposure Point Concentration
eQAPP	electronic Quality Assurance Project Plan
ER	Engineer Regulation
ft	feet
FACT	FLUTe Activated Carbon Technique
FD	field duplicate
FLUTe	Flexible Liner Underground Technologies, LLC
FS	Feasibility Study
FTL	Field Team Leader
FUDS	Formerly Used Defense Site
FUDSChem	Formerly Used Defense Sites Chemical database
GAI	Geophysical Applications, Inc.
GC/MS	gas chromatography/mass spectrometry
GPR	ground ppenetrating radar
GPS	global positioning system
GSA	General Services Administration
HAZWOPER	Hazardous Waste Operations and Emergency Response
HGI	Hager Geoscience, Inc.

HHRA	Human Health Risk Assessment
HPA	Historic Photographic Analysis
HPFM	Heat Pulse Flow Meter
HQ	Hazard Quotients
HTRW	Hazardous, Toxic and Radiologic Waste
ICAL	initial calibration
ICS	Interference Check Solution
ICV	initial calibration verification
IDW	investigation-derived waste
INPR	Inventory Project Report
IS	internal standard
J/UJ	estimated
LCS	laboratory control sample
LEP	Licensed Environmental Professional
LLC	Limited Liability Company
LOD	limit of detection
LOQ	limit of quantitation
LOR	Letter of Responsibility
MB	method blank
MCL	maximum contaminant level
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MLS	Multi-Level Sampler
MNA	monitored natural attenuation
MS	matrix spike
MSD	matrix spike duplicate
MSL	mean sea level
MTBE	methyl-tert-butyl-ether
mV	millivolts
NPL	National Priorities List
NAPL	non-aqueous phase liquid
NAVD	North American Vertical Datum
NELAP	National Environmental Laboratory Accreditation Program
NTU	nephelometric turbidity unit
ORP	oxidation-reduction potential
OSHA	Occupational Safety and Health Administration

OTV	Optical Televiewer
PA	Preliminary Assessment
PAH	polynuclear aromatic hydrocarbons
PAL	project action level
PARCC	precision, Accuracy, Representativeness, comparability and completeness
PCB	polychlorinated biphenyl
PDT	Project Delivery Team
PFAS	per- and polyfluoroalkyl substances
PG	Project Geologist
pH	potential of hydrogen
PID	Photoionization Detector
PM	Project Manager
PMP	Project Management Professional
POC	point of contact
Ppb	parts per billion
PPE	personal protective equipment
ppm	parts per million
PSQ	principal study question
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
R	rejected
RAB	Restoration Advisory Board
RAWP	Risk Assessment Work Plan
RDEC	Residential Direct Exposure Criteria
RI	Remedial Investigation
RICR	Rhode Island Code of Regulation
RIDEM	Rhode Island Department of Environmental Management
RIDOH	Rhode Island Department of Health
RI/FS	Remedial Investigation/Feasibility Study
RPD	relative percent difference
RPM	Remedial Project Manager
RRT	Relative Retention Time
RSLs	Regional Screening Levels
RTCs	response-to-comments

RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SC	specific conductivity
SDWA	Safe Drinking Water Act
SDG	sample delivery group
SEDD	Staged Electronic Data Deliverable
SHSP	Site-Specific Safety Plan
SI	Site Inspection
SIM	Selected Ion Monitoring
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
STSC	Safety Trained Supervision Construction
STS	Safety Trained Supervisor
SVOC	semivolatile organic compound
TAL	target analyte list
TAT	turn-around time
TB	trip blank
TBD	to be determined
TCE	trichloroethene
TCRA	Time Critical Removal Action
TO	Task Order
TOC	Total Organic Carbon
TOQR	Rask Order Quality Representative
TOM	Task Order Manager
TPH	Total Petroleum Hydrocarbons
TSA	Laboratory Systems Audit
UCL	upper confidence level
US	United States
USACE	United States Army Corps of Engineers
USAGC	United States Army Geospatial Center
USAPHC	United States Army Public Health Command
USEPA	United States Environmental Protection Agency
USCS	Unified Soil Classification
USGS	United States Geological Survey

UST	underground storage tank
UFP	Uniform Federal Policy
UU/UE	unlimited use and unrestricted exposure
VOC	volatile organic compound

EXECUTIVE SUMMARY

This Quality Assurance Project Plan (QAPP) was prepared to support closure of the Former NIKE PR-79 Control Area located in Foster, Rhode Island ("Property"). This document follows the format required for an Optimized Uniform Federal Policy (UFP) QAPP (DoD/USEPA/DoE, 2012) and includes the required content for a UFP-QAPP and Sampling and Analysis Plan (SAP) (DoD, 2013). The Risk Assessment Work Plan (RAWP) is provided as **Appendix G**. These plans combined under this document satisfies the requirements of a Remedial Investigation Work Plan. The area where contamination associated with the Property has come to be located is referred to, throughout this QAPP, as the "Site".

Site closure activities are being conducted by the United States Army Corps of Engineers (USACE), New England District (CENAE) under the Defense Environmental Restoration Program (DERP) for Formerly Used Defense Sites (FUDS) Program Policy for Environmental Quality Engineer Regulation (ER) ER-200-3-1 dated May 10, 2004 (USACE, 2004), the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and guidance from Rhode Island Department of Environmental Management's (RIDEM) Rules and Regulations for the Investigation and Remediation of Hazardous Material Releases, Title 250 Rhode Island Code of Regulations (RICR) Chapter-140, Subchapter 30, Part 1 (Rhode Island Remediation Regulations 250-RICR-140-30-1) codified on January 8, 2019. This QAPP presents the organization, objectives, and planned activities to complete the Remedial Investigation (RI) phase under CERCLA. Protocols for sample collection, handling, and storage, chain-of-custody, laboratory and field analyses, data validation, data evaluation, and reporting are addressed herein.

QAPP Worksheet #1 & 2: Title and Approval Page

1. Project Identifying Information

   a. Site name/project name: Former NIKE PR-79 Control Area Remedial Investigation
   b. Site location/number: Foster, Rhode Island / D01RI0063/02
   c. Contract/Work assignment number: W912WJ-19-D-0003

2. Lead Organization

   a. Lead Organization Project Manager (name/title/signature/date)

   Erin Kirby PG, LEP, Engineering Technical Leader 9/11/2020
   Name Date

   b. Lead Organization Quality Manager (name/title/signature/date)

   Project Delivery Team 9/11/2020
   Name Date

2. Investigative Organization

   a. Investigative Organization Project Manager (name/title/signature/date)

   Gregory Hencir, PMP, Task Order Manager 9/11/2020
   Name Date

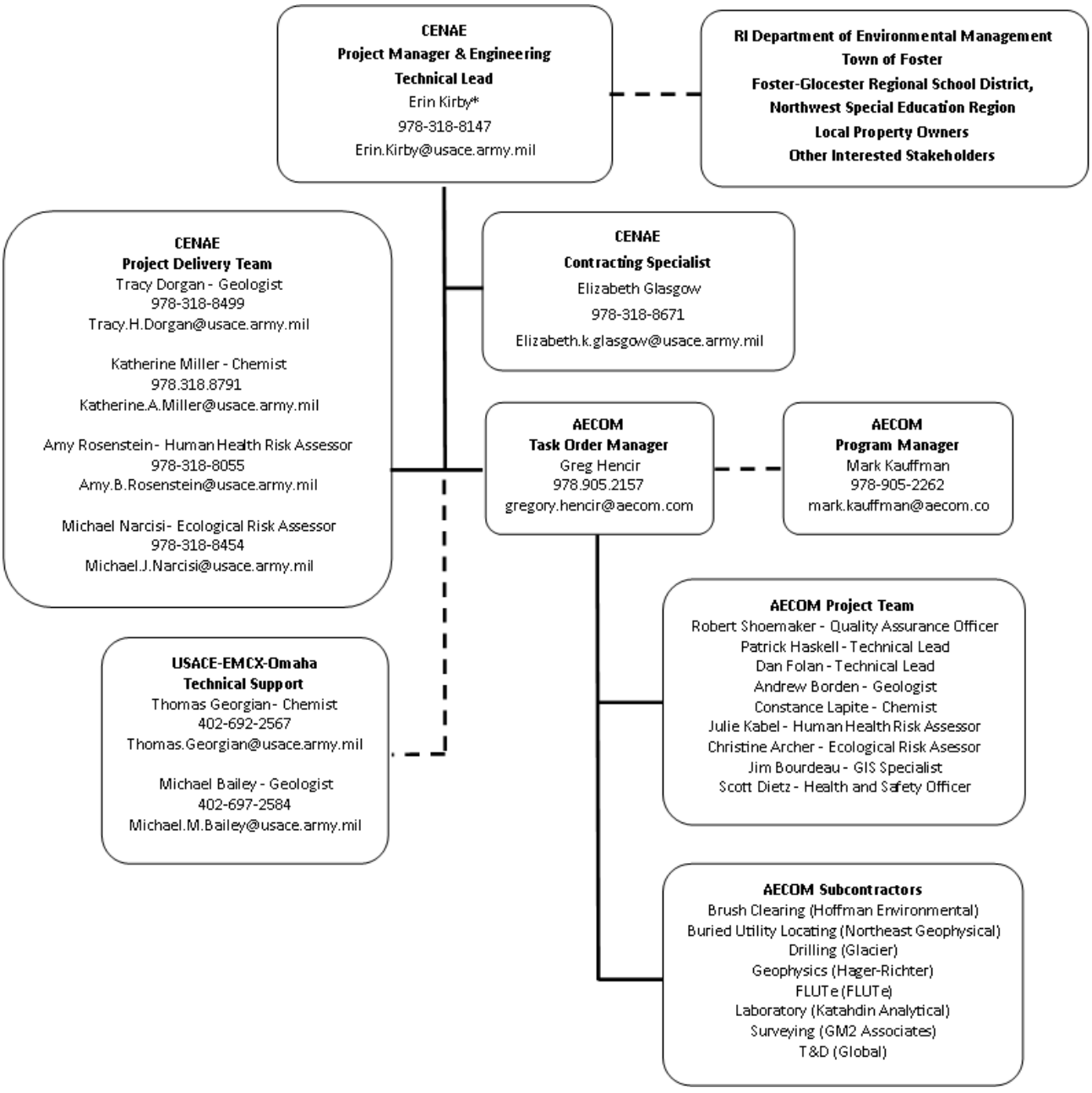
   b. Investigative Organization Quality Manager (name/title/signature/date)

   Robert Shoemaker, PMP, Task Order Quality Representative 9/11/2020
   Name Date

5. List plans and reports from previous investigations relevant to this project: Please refer to Section 10.7 of Worksheet 10.

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

* QAPP recipient Line of Authority —————— Line of Communication - - - - - -



QAPP Worksheets #4, 7 & 8: Personnel Qualifications and Sign-off Sheet

This worksheet contains a list of the key project personnel who are identified as performing the tasks that are defined in this QAPP and includes the personnel's organization, project role, education/experience, and specialized training/certifications. Key personnel will receive a copy of this project-specific QAPP and will be required to read and understand the QAPP prior to performing project tasks. This table as well as **Worksheet #1** will be completed and maintained within AECOM's files.

Project Personnel	Project Title/Role	Telephone Number	Email Address	Specialized Training/Certifications	Signature/Date
Mark Kauffman, PE	HTRW Program Manager	978-905-2262	mark.kauffman@aecom.com	PE: New York	See Project File
Greg Hencir, PMP	Task Order Manager (TOM)	978-905-2157	gregory.hencir@aecom.com	Project Management Professional (PMP) Certification OSHA 40hr HAZWOPER OSHA 10hr Construction	See Project File
Robert Shoemaker, PMP	Deputy TOM, Task Order Quality Representative (TOQR)	512-419-5576	robert.shoemaker@aecom.com	PMP Certification OSHA 40hr HAZWOPER	See Project File
Patrick Haskell, CHMM	Technical Lead	401-854-2808	patrick.haskell@aecom.com	Licensed Environmental Professional (LEP) Certified Hazardous Materials Manager (CHMM)	See Project File
Andrew Borden, PG	Junior Geologist	978-905-2405	andrew.borden@aecom.com	Project Geologist (PG) OSHA 40hr HAZWOPER OSHA 30hr Construction Safety	See Project File
Constance Lapite	Project Chemist	978-905-3131	constance.lapite@aecom.com	OSHA 40hr HAZWOPER	See Project File
Julie Kabel	Human Health Risk Assessor	603-263-2145	julie.kabel@aecom.com	Master of Public Health	See Project File
Christine Archer	Ecological Risk Assessor	603-622-1556	christine.archer@aecom.com	OSHA 40hr HAZWOPER	See Project File
Jim Bourdeau	GIS Specialist	978-905-2129	james.bourdeau@aecom.com		See Project File

Project Personnel	Project Title/Role	Telephone Number	Email Address	Specialized Training/Certifications	Signature/Date
Scott Dietz, CSP, STSC	Health and Safety Officer	240-344-5892	scott.dietz@aecom.com	Certified Safety Professional (CSP) Safety Trained Supervisor Construction (STSC) OSHA 40hr HAZWOPER OSHA 30hr Construction Safety OSHA 8hr Supervisor	See Project File
John Shannon	Field Team Lead	617-775-0123	john.shannon@aecom.com	OSHA 40hr HAZWOPER OSHA 8hr Supervisor CPR/First Aid/AED	See Project File
Richard Purdy, STS	Site Safety and Health Officer (SSHO)	978-905-3171	richard.purdy@aecom.com	OSHA 40hr HAZWOPER OSHA 30hr Construction Safety OSHA 8hr Supervisor Safety Trained Supervisor (STS) Level 1+ Hazardous Materials/Waste Shipper CPR/First Aid/AED	See Project File

ORGANIZATION: Katahdin Analytical Services

Name	Project Title/Role	Telephone Number	Email Address	Specialized Training/Certifications	Signature/Date
Heather Manz	Project Manager	207-874-2400 ext. 17	hmanz@katahdinlab.com		See Project File
Leslie Dimond	QA Manager	207-874-2400	ldimond@katahdinlab.com		See Project File

Special Training Requirements/Certification

AECOM and AECOM subcontractor personnel conducting intrusive field work will have received training in accordance with Occupational Safety and Health Administration (OSHA) requirements as stated in 29 Code of Federal Regulations (CFR) 1910.120(e). AECOM personnel will be provided a copy of the AECOM Accident Prevention Plan that complies with USACE EM 385-1-1 and developed to cover field activities and contains information on the hazards associated with the work area and the precautions that should be observed.

In addition to health and safety training, project personnel will be given copies of the project QAPP for review and a briefing on lines of communication and responsibilities. Specialized training is required of subcontract laboratory personnel and is documented in the associated laboratory quality assurance plans.

QAPP Worksheet #6: Communication Pathways

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathway To/From, etc.)
Regulatory Agency Interface	RIDEM RPM CENAE PM/ETL	Richard Gottlieb Erin Kirby	401-222-2797 ext. 7138 978-318-8147	The CENAE PM/ETL will inform regulatory agency via phone and/or email as needed during execution of the QAPP.
Field Progress Reports	AECOM FTL AECOM TOM	John Shannon Greg Hencir	617-775-0123 978-905-2157	The AECOM FTL will contact the AECOM TOM on a daily basis via phone, and every 1 to 2 days will summarize progress via email.
Gaining Site Access	AECOM TOM AECOM FTL CENAE PM/ETL	Greg Hencir John Shannon Erin Kirby	978-905-2157 617-775-0123 978-318-8147	The AECOM TOM or FTL will notify the CENAE PM/ETL and via email or verbally of planned sampling event 2 weeks prior to mobilizing to the Site.
Obtaining Utility Clearances for Intrusive Activities	AECOM TOM CENAE PM/ETL	Greg Hencir Erin Kirby	978-905-2157 978-318-8147	The AECOM TOM will coordinate verbally or via email with the CENAE PM/ETL at least 2 weeks in advance of accessing the Site to initiate the utility clearance process for intrusive sampling locations.
Stop Work due to Safety Issues	AECOM TOM AECOM FTL AECOM SSHO AECOM HSO CENAE PM/ETL	Greg Hencir John Shannon Rick Purdy Scott Dietz Erin Kirby	978-905-2157 617-775-0123 978-905-3171 240-344-5892 978-318-8398	Field team members who observe an unsafe situation have the authority to stop work. If AECOM is the responsible party for a stop work command, the AECOM SSHO will inform on-Site personnel, subcontractor(s), AECOM TOM and HSO within 1 hour (verbally or by email). The HSO will notify the CENAE PM/ETL within 24 hours of notification from the SSHO. If a subcontractor is the responsible party, the subcontractor TOM must verbally inform the AECOM SSHO within 15 minutes, and the AECOM SSHO will then follow the procedure listed above.

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathway To/From, etc.)
QAPP Changes prior to Field/ Laboratory work	AECOM TOM AECOM FTL AECOM Project Chemist CENAE PM/ETL RIDEM RPM	Greg Hencir John Shannon Constance Lapite Erin Kirby Richard Gottlieb	978-905-2157 617-775-0123 978-905-3131 978-318-8398 401-222-2797 ext. 7138	The AECOM FTL will verbally inform the AECOM TOM upon realizing a need for a QAPP modification. The FTL or TOM will document the proposed changes via a QAPP modification form within 5 days and send it to the Project Chemist. The Project Chemist will assess whether: a) The modification has the potential to affect the project's ability to achieve Data Quality Objectives (DQOs), b) The modification requires a change in field or laboratory methods, which may affect project schedule or cost, or c) The modification does not affect DQOs, schedule, or cost, and is for documentation purposes only. QAPP modifications potentially affecting DQOs will be submitted to the CENAE PM/ETL for review and consideration and may require QAPP amendments and approval. Modifications in laboratory/field methods, project schedule or cost will be submitted to the CENAE for review and consideration. The CENAE PM/ETL will inform RIDEM RPM of significant QAPP changes. Minor modifications not affecting DQOs, schedule, or cost will be documented in the project file.
QAPP Changes in the Field	AECOM TOM AECOM FTL CENAE PM/ETL RIDEM RPM	Greg Hencir John Shannon Erin Kirby Richard Gottlieb	978-905-2157 617-775-0123 978-318-8398 401-222-2797 ext. 7138	The AECOM FTL will inform the AECOM TOM verbally within same day of the need for a QAPP change in the field. The TOM will inform the CENAE PM/ETL by email within 24 hours; the PM sends a concurrence letter to the CENAE PM/ETL, if warranted, within 7 calendar days and the CENAE PM/ETL signs the letter within 5 business days of receipt Scope change is to be implemented before work is executed. Changes must be documented on the QAPP modification form (within 2 business days) or QAPP amendment (within timeframe agreed to by the AECOM TOM and CENAE PM/ETL). Changes of the approved QAPP affecting the scope or implementation of the sampling program will be made only upon authorization of CENAE PM/ETL. The CENAE PM/ETL will inform RIDEM RPM of significant QAPP field changes within 5 business days.
Field Corrective Actions	AECOM TOM AECOM FTL CENAE PM/ETL	Greg Hencir John Shannon Erin Kirby	978-905-2157 617-775-0123 978-318-8398	The AECOM FTL will inform the AECOM TOM verbally within same day of field corrective actions. The AECOM TOM will then notify the CENAE PM/ETL (verbally or by email) within 1 business day.

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathway To/From, etc.)
Recommendations to stop work and initiate work upon corrective action	AECOM FTL AECOM SSHO AECOM TOM AECOM Project Chemist CENAE PM/ETL	John Shannon Rick Purdy Greg Hencir Constance Lapite Erin Kirby	617-775-0123 978-905-3171 978-905-2157 978-905-3131 978-318-8398	Responsible party verbally informs the AECOM TOM, FTL, and subcontractors within 1 hour of recommendation to stop work and within 24 hours of recommendation to restart work. Responsible party follows verbal notification with an email to the AECOM TOM and CENAE PM/ETL within 24 hours.
Sample Receipt Variances	Laboratory PM AECOM Project Chemist AECOM TOM AECOM FTL	Heather Manz Constance Lapite Greg Hencir John Shannon	207-874-2400 ext. 17 978-905-3131 978-905-2157 617-775-0123	The analytical laboratory PM will notify verbally or by email the AECOM Project Chemist immediately upon receipt of chain-of-custody/sample receipt variances for clarification or direction from the Project Chemist. The AECOM Project Chemist will notify verbally or by email the AECOM TOM and FTL within 1 business day, if corrective action is required. The AECOM Project Chemist will notify verbally or by email the analytical laboratory PM and the AECOM FTL within 1 business day of required corrective action.
Analytical Corrective Actions	Laboratory PM AECOM Project Chemist	Heather Manz Constance Lapite	207-874-2400 ext. 17 978-905-3131	The analytical laboratory PM shall notify the AECOM Project Chemist of any analytical data anomaly within 1 business day of discovery. After the analytical laboratory receives guidance from the AECOM Project Chemist, the laboratory shall initiate corrective action to prevent further anomalies.

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathway To/From, etc.)
Analytical Data Quality Issues	Laboratory PM AECOM Project Chemist AECOM TOM CENAE PM/ETL	Heather Manz Constance Lapite Greg Hencir Erin Kirby	207-874-2400 ext. 17 978-905-3131 978-905-2157 978-318-8398	The analytical laboratory PM will notify verbally or by email the AECOM Project Chemist within 1 business day of discovering an issue related to analytical laboratory data. The AECOM Project Chemist will notify the AECOM TOM within 1 business day. The AECOM Project Chemist will notify the AECOM TOM verbally or by email within 48 hours of validation completion that a non-routine and significant analytical laboratory quality deficiency has been detected that could affect this project and/or other projects. The AECOM TOM will then verbally advise the CENAE PM/ETL within 24 hours of notification from the Project Chemist. The CENAE PM/ETL will take corrective action appropriate for the identified deficiency. If lack of significant data quality or non-useable data is at issue, the CENAE Chemist will be contacted to ensure the issues do not have the potential to impact other CENAE projects.
Reporting Data Validation Issues/Data Validation Corrective Actions	AECOM Project Chemist AECOM TOM	Constance Lapite Greg Hencir	978-905-3131 978-905-2157	The AECOM Project Chemist or Data Validator will perform validation as specified in Worksheets #34, #35, and #36, and will contact the analytical laboratory as soon as possible if issues are found that require corrective action. If the AECOM Project Chemist or Data Validator identifies non-usable data during the data validation process that requires corrective action, the AECOM TOM will coordinate with the Project Chemist to take corrective action appropriate for the identified deficiency to ensure project objectives are met. Corrective action may include resampling and/or reanalyzing the affected samples, as determined by the TOM.

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathway To/From, etc.)
Notification of Non-Usable Data	Laboratory PM AECOM Project Chemist AECOM TOM CENAE PM/ETL	Heather Manz Constance Lapite Greg Hencir Erin Kirby	207-874-2400 ext. 17 978-905-3131 978-905-2157 978-318-8398	If the analytical laboratory determines that data they have generated is non-usable, the analytical laboratory PM will notify verbally or by email the AECOM Project Chemist within 1 business day of when the issue is discovered. The AECOM Project Chemist will notify the AECOM TOM verbally or by email within 1 business day of the need for corrective action, if the non-usable data is a significant issue (i.e., critical sample data). Corrective action may include resampling and/or reanalyzing the affected samples. If a AECOM Project Chemist or Data Validator identifies non-usable data during the data validation process, the TOM will be notified verbally or via email within 48 hours of validation completion that a non-routine and significant analytical laboratory quality deficiency has resulted in non-usable data. The AECOM TOM will take corrective action appropriate for the identified deficiency to ensure the project objectives are met. The TOM will notify the CENAE PM/ETL verbally or by email of problems discovered regarding analytical laboratory results or of analyses that could significantly affect the usability of that data or of project failures that impact the ability to complete the scope of work. The CENAE PM/ETL may, at his discretion, contact the CENAE Chemist for assistance in problem resolution. Such notification will be made within one business day of when the issue is discovered.

Notes:

CENAE	=	United States Army Corps of Engineers New England District
DQO	=	Data Quality Objective
ETL	=	Engineering Technical Lead
ext.	=	Extension
FTL	=	Field Team Lead
HSO	=	Health and Safety Officer
PM	=	Project Manager
POC	=	Point of Contact
QAPP	=	Quality Assurance Project Plan
RIDEM	=	Rhode Island Department of Environmental Management
RPM	=	Remedial Project Manager
SSHO	=	Site Safety and Health Officer
TBD	=	To be determined
TOM	=	Task Order Manager
TOQR	=	Task Order Quality Representative

QAPP Worksheet #9-1: Project Scoping Session Participants Sheet

Project Name: Remedial Investigation
Projected Date(s) of Sampling: Spring 2020
Project Manager (Contractor): Gregory Hencir
Date of Session: 25 June 2019
Location of Session: CENAE, Concord, MA
Scoping Session Purpose: Kickoff Meeting

Site Name: Former NIKE PR-79 Control Area
Site Location: Foster, Rhode Island

Name	Title	Affiliation	Phone #	E–Mail Address	Project Role
Erin Kirby	Project Manager	CENAE	978-318-8147	Erin.Kirby@usace.army.mil	Project Manager/ Contract Officer Representative (COR)
Tracy Dorgan	Geologist	CENAE	978-318-8499	Tracy.H.Dorgan@usace.army.mil	Geologist
Amy Rosenstein	Human Health Risk Assessor	CENAE	978-318-8055	Amy.B.Rosenstein@usace.army.mil	Human Health Risk Assessor
Michael Narcisi	Ecological Risk Assessor	CENAE	978-318-8454	Michael.J.Narcisi@usace.army.mil	Ecological Risk Assessor
Dion Lewis	Chemistry and Risk Assessment Department Section Chief	CENAE	978.318.8785	Dion.A.Lewis@usace.army.mil	Chemist
Katherine Miller	Chemist	CENAE	978-318-8791	Katherine.A.Miller@usace.army.mil	Project Chemist
Rosemary Schmidt	Geology Department Section Chief	CENAE	978-318-8345	Rosemary.A.Schmidt@usace.army.mil	Technical Support
Thomas Georgian	Chemist	USACE-EMCX- Omaha	402-692-2567	Thomas.Georgian@usace.army.mil	Chemist
Michael Bailey	Geologist	USACE-EMCX- Omaha	402-697-2584	Michael.M.Bailey@usace.army.mil	Geologist
Gregory Hencir	Project Manager	AECOM	978-905-2157	Gregory.Hencir@aecom.com	Project Manager
Patrick Haskell	Technical Leader	AECOM	401-854-2808	Patrick.Haskell@aecom.com	Technical Lead
Patricia Shattuck	Geologist	AECOM	603-520-1238	Patricia.Shattuck@aecom.com	Senior Geologist
Robert Shoemaker	Project Manager	AECOM	512-419-5576	Robert.Shoemaker@aecom.com	Technical Support
Julie Kabel	Human Health Risk Assessor	AECOM	603-263-2145	Julie.Kable@aecom.com	Senior Human Health Risk Assessor
Constance Lapite	Chemist	AECOM	978-905-313	Constance.Lapite@aecom.com	Project Chemist

The following bullets summarize the significant topics that were discussed and decisions made during the kickoff meeting session that contribute to the Remedial Investigation QAPP. Participants included CENAE, USACE, and AECOM representatives on the Project Delivery Team (PDT).

Potential Source Areas

- Team discussed "beneficial use" of infrastructure transferred to the Town of Foster in 1965, including the Barracks, Administrative, and Mess Hall buildings and heating oil underground storage tanks (USTs) supplying each building. The three heating oil USTs currently remain in-place. The 2,000-gallon No. 2 fuel oil UST adjacent to the Mess Hall building was recently in-use and is reportedly leaking as of November 2018[1]. Team concurred that these three USTs beneficially used by the Town of Foster are ineligible for investigation and cleanup under DERP-FUDS and are excluded from this RI.

- The former Barracks and Administrative buildings were transferred to the Town of Foster in good condition and demolished by the Town of Foster in 2012. Investigation of possible hazardous building materials associated with these buildings (i.e. lead and asbestos) is ineligible for investigation and cleanup under DERP-FUDS.

- A UST adjacent to the Frequency Changer Building in the Radar Area was not beneficially used by the Town of Foster. CENAE removed that UST in 1994. There is limited documentation beyond that it was closed under RIDEM UST regulations and no additional investigation was necessary. Team agreed that this UST will be excluded from this RI because it was closed under a state program.

- Area of Concern 5 (AOC-5) ("Western Disposal Area") was used for residential and agricultural dumping by the property owner and is not attributable to former Department of Defense (DoD) activities. Team agreed that AOC-5 is ineligible for investigation and cleanup under DERP-FUDS.

- The Western Sewage Disposal Area (AOC-4) was constructed by DoD to handle excess sewage from the Southern Leach Field (AOC-3). The Southern Leach Field was beneficially used by the Town of Foster until a new septic leach field was built by the Town of Foster in the 2000s. AOC-3 and AOC-4 will be investigated in this RI. The new septic leach field constructed by the Town of Foster (located within the vicinity of AOC-3) is excluded from this RI. Potential impacts

[1] Following the meeting, the UST adjacent to the Mess Hall building was removed by the Town of Foster in September 2019.

identified in AOC-3 that are due to a release from the new septic leach field will not be further assessed because that release is not attributable to former DoD activities.

- Trichloroethylene (TCE) is the Site-related primary constituent of potential concern (COPC), the release of which to groundwater precipitated a Time Critical Removal Action (TCRA) in 2002 to install point of use carbon filtration systems on downgradient residential supply wells ROU-1, ROU-2, and ROU-3 and on-Property supply well NIKE-1. The source and/or release area of the TCE has not been identified to date. Historical soil and groundwater samples collected near a floor drain in the Frequency Changer Building reported low level TCE detections in subsurface soils. CENAE noted that according to retired US Army personnel including the former Executive Officer stationed at NIKE PR-79, solvents were reportedly used for multiple on-Property activities including deicing the roads and helipad. TCE and other solvents/chemicals may have also been used for motor pool vehicle maintenance at various locations. CENAE added that dilute TCE-containing water was leaking from the NIKE-1 water supply line over an extended period of time until it was replaced with a new water supply line installed by the Town of Foster. The exact location(s) of that pipe leak is unknown. Team discussed multiple small individual releases as a working conceptual site model (CSM). USACE-EMCX noted that the age of potential releases from former DoD activities and the unknown location of a release from the NIKE-1 water supply line will make it a challenge to identify a discrete "smoking gun" source of TCE and flow path from a point of release in overburden to bedrock to the supply wells. USACE-EMCX added that the RI may find a low-concentration diffuse source of TCE in cobble/boulder, weathered bedrock, and/or bedrock fractures. Team acknowledged that finding evidence of a diffuse source area without a discrete source is an acceptable RI conclusion. A principal study question (PSQ) is: are TCE impacts from one or multiple discrete or diffuse source(s)? Another PSQ is: are there are other Site-related COPCs besides TCE?

- Motor pool vehicle maintenance may have been conducted by DoD northwest of the former Guard Post along Theodore Foster Drive and northwest of the former water storage tank and pump house. These two areas will be investigated in the RI.

Constituents of Potential Concern

- Team discussed COPCs related to former DoD activities. USACE-EMCX suggested reference to the Environmental Conditions Assessment Guide for NIKE Missile Batteries under DERP-FUDS prepared by USACE dated July 2003 in developing a COPC list. The Team discussed the sampling design at length. The RI sampling design will consist of exposure areas that will test for COPCs

associated with former DoD activities as described in the Environmental Conditions Assessment Guide with modifications based on Site-specific knowledge.

- CENAE said that the RI sampling design should focus on filling in identified data gaps from historical analytical data. The COPC list may be reduced by using historical analytical data.

- Prior to DoD development in 1955, a portion of the Site was used as an apple & peach orchard when arsenic was commonly used as a pesticide. Neighboring properties are currently used for agricultural purposes and (more recently) solar power generation. Team discussed pesticides used for agricultural purposes (i.e. arsenic) and agricultural equipment maintenance and fueling (e.g., methyl-tert-butyl-ether [MTBE], petroleum).

- Team discussed confounding COPCs that may be encountered. USACE-EMCX recommended being selective in testing COPCs in light of more recent releases attributable to others (i.e. heating oil, MTBE). As an example, USACE-EMCX suggested focusing analysis on chlorinated volatile organic compounds (CVOCs) to identify TCE and its breakdown products instead of a full list of volatile organic compounds (VOCs). USACE-EMCX also suggested sampling the former transformer area for polychlorinated biphenyl (PCBs) only. However, only the two motor pool vehicle maintenance areas would be analyzed by a full list of VOCs because potential petroleum-related releases would be attributable to former DoD activities. CENAE recommended the full suite of analytical data being evaluated as it is not clear on the release types and locations.

- In response to USACE-EMCX's comment about the transformer area, CENAE noted that Aroclor 1260 was not detected above laboratory reporting limits (4.3 U micrograms per liter [µg/L]) in a surficial soil sample collected from 0-2 feet (ft) below ground surface (bgs) in the transformer area (AMEC Environment & Infrastructure, Inc. [AMEC], 2014a). Prior Site information should be used to refine the RI and address data gaps.

Potential Pathways

- The existing overburden monitoring wells were installed as temporary piezometers by direct-push drilling methods with pre-pack screens. Refusal was encountered at what is believed to be the top of boulders/cobbles based on 2016 surface geophysics data collection and reprocessing. It is estimated that these wells were installed between 5-10 ft above bedrock. There is currently no lithological or soil analytical data from below the refusal depths. Team understanding of overburden groundwater flow is limited. There are no weathered bedrock and bedrock monitoring wells. A PSQ is whether there is an existing COPC mass in overburden,

weathered bedrock, and/or transmissive fracture network in bedrock that may act as a continuing source of impacts at exposure points?

- The RI will address data gaps in weathered rock by advancing soil borings through boulders/cobbles and into weathered bedrock using rotosonic drilling methods. One soil sample should be collected from each overburden soil boring at the groundwater interface and another soil sample collected at a shallower depth with the highest photoionization detector (PID) reading.

- The RI will assess whether concentrations of VOCs in shallow groundwater are detected at or above levels with the potential to pose a health concern via the vapor intrusion pathway. Recommendations for further evaluation of the potential vapor intrusion pathway will be made as applicable/appropriate.

- The RI will install bedrock monitoring wells with the primary study goal of identifying transmissive fractures with higher concentrations than what has been detected in supply wells. Team discussed installing sentinel monitoring wells in the RI phase. Monitoring wells should primarily be placed to address RI study goals with dual use for future long-term monitoring as a secondary goal. A refined CSM as an output of the RI and Feasibility Study (FS) phases will assist in determining sentinel well placement as a component of the remedy, if needed. Sentinel wells should be situated to intercept groundwater that flows between the identified source and exposure points (i.e. residential supply wells, surface water, and seeps).

- AOC-4 ("Western Sewage Disposal Area") discharges to Winsor Brook. Details about drainage in the western portion of the Site are available in the *Wetland and Waters of the US Delineation Report* prepared by Woodard & Curran dated February 8, 2019.

- A subset of local seeps and intermittent streams at the base of Oak Hill, where property access was permitted, were mapped in 2018 as part of the 2019 wetland and waters delineation report. CENAE mentioned that elevations of the seeps and surface water bodies may be above the existing COPC mass in groundwater and that hydraulic connectedness to seeps is unclear. A PSQ is to determine whether seeps are a potential exposure point and what are the Site-related COPCs concentrations in seeps?

Risk Assessment

- Team discussed background soil and groundwater. A PSQ is what are the Site-related COPC concentrations in background soil and groundwater? An appropriate area for the background

study needs to be identified. The orchard north of the Property is not a good candidate since there could be impacts from recent agricultural equipment and pesticide use (i.e. arsenic). Arsenic may not need to be included in the background study if none of the former DoD activities had the potential to release arsenic. Team discussed several background study options including one or two background study areas for identification of anthropogenic and non-anthropologic concentrations of commonly occurring metals and polynuclear aromatic hydrocarbons (PAHs). CENAE suggested background sampling on another hilltop. Team discussed that the easternmost portion of the Site that was not developed by DoD may be the best location for background samples; however, it was unclear at the meeting whether this portion of the Site was historically used as an apple orchard or not.[2] The location of background study area(s) will be discussed in a follow-up meeting. USACE-EMCX recommended that the background study consist of a representative dataset of at least 25 grab samples in order to do statistical comparisons using hypothesis tests for centrality (e.g. T-test, Wilcoxon Test, Permutation Test).

- Winsor Brook is west of the Property and flows south to the Ponaganset River, which flows to Hopkins Mill Pond and Barden Reservoir, which flows into the Scituate Reservoir. Discussed surface and pore water samples. Porewater samples are needed to assess risk in the ecologically bio-active zone as well as to assess whether there is COPC transport from groundwater to surface water. Because of the low concentrations observed in Site data as well as dilution potential for surface water samples alone, the pore water samples serve multiple purposes. The Porewater data will also allow of evaluations of hydraulic gradient to aid in assessing groundwater to surface water interaction/variations at the Site. Evaluating porewater samples in the absence of substantial sediment or areas of shallow bedrock will be challenging. Team discussed whether or not to include sediment samples. CENAE observed that Winsor Brook is fast moving and consists of cobbles/gravel without robust sedimentation to sample. Prior investigations had successfully installed Henry Samplers. CENAE noted a small wetland complex and surface water pond south of the Property at 33 Winsor Road that likely receives flow from Oak Hill. CENAE has identified numerous potential locations where sediment is also available for sampling in addition to porewater. Specific surface water and sediment sampling locations will be discussed in a follow-up meeting.

- Team discussed indoor soil vapor intrusion. A 2013 soil vapor study was performed to screen shallow soils for VOCs to refine the placement of proposed sampling points. That soil vapor

[2] Historical aerial photographs do not appear to show the easternmost portion of the Site having been used as an orchard (USAGC, 2015).

study was not conducted to assess indoor soil vapor intrusion. CENAE explained that soil vapor intrusion at the former Mess Hall building and nearby residential buildings will need to be considered in development of the QAPP. USACE-EMCX said that due to the age of potential releases from former DoD activities, VOCs likely degraded to some degree and migrated downwards to at least the cobble/boulder and/or weathered bedrock zones. USACE-EMCX added that direct exposure is typically 10-15 ft bgs, which is approximately the bottom depth of residential basements as well as close to the top of bedrock where residual CVOC's may exist.

- Soil sampling should be done with a systematic random sampling design constructed over an area that represents the exposure point (e.g., one acre for a residential lot)[3].

Action Items

- AECOM preparing the draft RI Work Plan in an Optimized UFP for Quality Assurance Project Plans (UFP-QAPP) format. The team agreed that preliminary working draft worksheets (Worksheets 10, 11, and 17) that describe the CSM, study goals, sampling design and rationale will be shared with the PDT in advance for team discussion. There will be no "formal" comments or response-to-comments (RTCs) prior to CENAE and USACE-EMCX review of the full draft QAPP. The team expects the full draft QAPP to be provided in September 2019 with formal comments, formal responses, and a subsequent "final" QAPP occurring in the winter 2020 to position the team for presentation of the QAPP to stakeholders and field mobilization in spring 2020.

- Follow-up focused technical planning meetings will be scheduled as needed to discuss technical topics. For instance, Risk assessors will meet to discuss exposure points and develop sampling design for the RAWP. Chemists and geologists will meet to determine the subset of potential impacts associated with historical activities.

[3] A systematic random sampling design was discussed in June 2019; however, after further consideration during the RI planning, the team decided in September 2019 to proceed with a biased sampling approach as summarized in Worksheet 9-3. Discrete sampling was chosen to achieve data gaps regarding the conceptual site model. Discrete sampling results will be evaluated on a Site-wide basis in comparison to human health and ecological risk-based screening levels and Site-specific background, as further described in Section 11.5. If biased sample results exceed risk-based screening levels and associated background levels, then a second RI phase will be designed with an unbiased and exposure-based sampling approach and decision units and exposure units to be further evaluated in a risk assessment.

QAPP Worksheet #9-2: Project Scoping Session Participants Sheet

Project Name: Remedial Investigation
Site Name: Former NIKE PR-79 Control Area
Projected Date(s) of Sampling: Spring 2020
Site Location: Foster, Rhode Island
Project Manager (Contractor): Gregory Hencir
Date of Session: 8 July 2019
Location of Session: Former NIKE PR-79 Control Area, Foster, Rhode Island
Scoping Session Purpose: Site Walkthrough

Name	Title	Affiliation	Phone #	E–Mail Address	Project Role
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Patrick Haskell	Technical Leader	AECOM	401-854-2808	Patrick.Haskell@aecom.com	Technical Lead

The following bullets summarize the significant topics that were discussed and decisions made during the Site walkthrough that contribute to the RI QAPP.

Site Overview

- Team discussed "beneficial use" of infrastructure transferred to the Town of Foster in 1965, including the Barracks, Administrative, and Mess Hall buildings and associated USTs. The three heating oil USTs currently remain in-place. The 2,000 gallon No. 2 fuel oil UST adjacent to the Mess Hall building was recently in use and is reportedly leaking. Team acknowledged that possible hazardous building materials related to beneficially used buildings and the associated USTs are ineligible for investigation and cleanup under DERP-FUDS and are excluded from this RI.[1]

[1] The UST adjacent to the Mess Hall building was removed by the Town of Foster in September 2019.

- Team acknowledged that AOC-5 was used by the property owner for residential and agricultural dumping and is not attributable to former DoD activities. CENAE said that neighboring landowners repeatedly told CENAE that AOC-5 was used for agricultural dumping and not by DoD. Because AOC-5 is not attributable to former DoD activities, AOC-5 is ineligible for investigation and cleanup under DERP-FUDS.

- The team observed former NIKE PR-79 control area features including AOC-1 ("Radar Area"), AOC-2 ("Operations and Maintenance Area"), AOC-3 ("Southern Leach Field"), AOC-4 ("Western Sewage Disposal Area"), and AOC-5 ("Western Disposal Area"). Team observed significant overgrowth in AOC-1. Team discussed possible laydown area for equipment located north of the former Administration building.

- The team observed potential areas of former motor pool vehicle maintenance located northwest of the former Guard Post along Theodore Foster Drive and northwest of the former water storage tank and pump house.

- The numerical standards of the RIDEM Rules and Regulations for the Investigation and Remediation of Hazardous Materials Releases 250-RICR-140-30-1 codified on January 8, 2019 will be considered in the development of the project action levels (PALs).

- Limited sediment for sediment sampling was observed along Winsor Brook due to the high water velocity and linear steam segment which is likely oriented along shallow bedrock fracture. Sediment sample locations do exist however they require flexibility in location and experienced field judgment to determine reasonable locations.

- Talked about properly decommissioning the abandoned drinking water well outside the fence along the dirt road.

- Leaks in the water line may have likely caused a diffuse source in the overburden. Impacts from this "secondary release remain to be seen, however trend data in existing analytical data make it appear a temporary concern.

- Original leach field was beneficially re-used until the Town of Foster built a new one.

Drinking Water Sampling

- CENAE collects three residential water samples (pre-filtration, between the two carbon filters, and post-filtration) from each of the four supply wells (NIKE-1, ROU-1, ROU-2, and ROU-3). A

full list of VOCs is tested annually at each of the four supply wells, including TCE and its breakdown products.

- In 2010, the U.S. Army Public Health Command (USAPHC) collected pre-filtration water samples from 13 residential wells within a half mile of the former NIKE PR-79 control area, including the four supply wells (NIKE-1, ROU-1, ROU-2, and ROU-3) (USAPHC, 2010). USAPHC reported TCE in supply well ROU-2 (5.2 µg/L) and an estimated value[2] (i.e., J-qualified) in a residential well at 32 Winsor Road (0.1 J µg/L); MTBE in a residential well at 17 Winsor Road (0.2 J µg/L); cis-1,2-dicholorethene in supply well ROU-2 (0.4 J µg/L); and, chloroform in a residential well at 54 ½ Maple Road (0.2 J µg/L) and 63 Maple Rock (0.3 J µg/L). TCE has not been detected in post-treatment samples.

- CENAE occasionally expands residential water sampling to residential supply wells located along Theodore Foster, Maple Rock, Winsor, Hartford Pike, and Old Danielson roads. No TCE or other Property-related constituents have been detected in these wells.

Community Involvement

- CENAE explained that CENAE regularly communicates Site information to homeowners during sampling. CENAE added that homeowners encountered near the Property (e.g. homeowners along Maple Road, Maple Rock Road, and Winsor Road, etc.) have not expressed interest to CENAE about forming a Restoration Advisory Board (RAB). The last public meeting was held in 2012 with participation from the Town of Foster, CENAE, RIDEM, congressional representatives, and local residents who asked to be informed about future investigation and cleanup activity. Site information is available and maintained at the Town of Foster public library. RIDEM suggested a newspaper notice to determine whether there was interest in a public informational session and mailing a factsheet to homeowners.

Surface Water and Sediment

- A portion of the team conducted a drive along Winsor Road to look for access to Winsor Brook from the Winsor Road overpass for possible surface water sampling locations and to see a small pond and wetland complex near 33 Winsor Road. Team noted that access up Winsor Brook from the Winsor Road overpass may be necessary to identify possible sediment sampling locations. Seeps were not observed during the Site walkthrough.

[2] J qualifier indicates an estimated value (result is between reporting limit and DL)

Existing Piezometers

- A portion of the team located the 14 piezometers (PZ-001 through PZ-022) that were installed as pre-pack wells using a direct push drill rig during a Phase I site investigation (AMEC, 2014a). The Phase I site investigation placed piezometers where refusal was encountered between 8 and 24.5 ft bgs. The team observed the 14 piezometers to be intact and collected water level and bottom depth measurements for use in determining whether the piezometers can be repurposed in the RI. The average water depth was approximately 10 ft bgs. Two of the piezometers (i.e., PZ-005 and PZ-015) were dry as evident by the water depth being measured below the bottom of the screen, which is consistent with historically dry measurements from this piezometer in July 2013 and June 2014. Bentonite was observed on the water level probe at one well (PZ-019), which is consistent with historical observations when sampled in the past (Johnson Company, 2016). The team observed dense overgrowth while locating the piezometers and most notably in the vicinity of PZ-011. The team also observed supply well NIKE-1.

QAPP Worksheet #9-3: Project Scoping Session Participants Sheet

Project Name: Remedial Investigation
Projected Date(s) of Sampling: Spring 2020
Project Manager (Contractor): Gregory Hencir
Date of Session: 12 September 2019
Location of Session: CENAE, Concord, MA
Scoping Session Purpose: QAPP Discussion

Site Name: Former NIKE PR-79 Control Area
Site Location: Foster, Rhode Island

Name	Title	Affiliation	Phone #	E–Mail Address	Project Role
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Andrew Borden	Geologist	AECOM	978-905-2405	Andrew.Borden@aecom.com	Technical Support

The following bullets summarize the significant topics that were discussed and decisions made during the meeting that contribute to the RI QAPP.

Study Goals

- Four study goals were presented to the team and were accepted, except, for clarity, the team agreed to split one of the study goals into two: identify possible source area(s) and assess nature and extent.

- Team discussed adding soil vapor intrusion as an exposure point. The RI includes an overburden monitoring well screened across the groundwater table located upgradient of the former Mess Hall to assess soil vapor intrusion of that building. The team agreed that soil vapor intrusion does not need to be explicitly stated in the study goals and that soil vapor will be assessed if it

is identified as a concern in overburden groundwater and as it applies to the impacted bedrock groundwater if untreated or additional impacted residential supply wells are found[1].

- Regional anthropogenic background COPC concentrations representative of areas beyond the limits of the Areas of Concern (AOCs) will be collected in the undisturbed ground immediately outside of the historic cemetery located north of AOC-1.[2]

General Field Sampling Approach

- The team agreed that the overall field sampling approach should be flexible to account for field observations. AECOM proposed a decision tree diagram to confirm the installation of monitoring wells at each location with CENAE during overburden drilling based on field observations (e.g., PID readings).

- Two soil samples are planned to be collected from each soil boring (i.e. a surface soil sample at 0-2 ft bgs and a subsurface soil sample collected in a two foot depth internal between 2 to 25 ft bgs). The surface soil sample will be collected from a depth interval of 0-2 ft bgs to be consistent and allow for comparison to the existing dataset. The subsurface soil sampling depth will be selected based on the observed groundwater table, lithology, and or where potential soil impacts were identified by field observation (i.e. elevated PID readings, etc.). Rotosonic drilling allows for overburden couplet wells of 1 to 1.5-inches in diameter to be installed in the same borehole. CENAE and AECOM geologists recommended that wells be 2-inches in diameter. With relatively shallow depths to bedrock, the team decided that two side-by-side overburden soil borings would be advanced to complete wells as 2-inches in diameter where cluster wells were proposed.

- Samples collected during the RI will be used to supplement an existing dataset collected by AMEC in 2013 during the initial phase of the RI (AMEC, 2014b) and residential drinking water collected by USACE since 2015 and used in a statistical evaluation to inform the risk assessment. A result of collecting data from within potential source areas is that the dataset would be biased

[1] A passive soil vapor study concluded that COPCs were not present in the vadose zone or off gassing from the water table beneath the areas tested within each AOCs (provided in Section 2.1, Soil Gas Survey, AMEC, 2013a). Based on those previous findings and conclusions, further investigation of soil vapor is not included as a principle study goal in Section 11.5 "Analytical Approach". However, if the RI determines, by screening overburden groundwater data for potential vapor intrusion results, that potential DoD-related releases in the vadose zone area present that may be a vapor intrusion concern to nearby residential structure, then a second mobilization will be designed and executed to investigate that concern.

[2] Historical aerial photographs appear to show the cemetery as having been used as an orchard prior to the 1950s (USAGC, 2015). The easternmost portion of the Site will be used as the background study area, as discussed in Worksheet 9-1.

towards higher COPC concentrations that may not be representative of Site-wide conditions. The Phase I investigation found low concentrations or no COPCs Site-wide and did not determine the nature and extent of the source area; therefore, there is not enough information at this time to define the decision unit (DU). The team agreed to bias sampling locations in the RI within potential source areas and based on a review of those results, additional samples may be required to provide a robust dataset.

Sampling Design and Rationale

- CENAE recommended the proposed location of SB-101 in AOC-1 be completed as an overburden monitoring well to assess groundwater in till/weathered bedrock in that area.

- CENAE recommended collecting a surface soil sample in the stressed/unvegetated area observed within AOC-1 as this feature has not adequately been investigated.

- CENAE recommended the proposed location of AOC-1-MW03 be moved from the floor drain location to where drums were formerly stored in-between PZ-019 and the location of the former 6,000 gallon diesel fuel UST. The purpose of AOC-1-MW03 is to assess overburden groundwater in the area surrounding PZ-019 where COPCs were identified (AMEC 2014b). Team agreed to abandon PZ-019 as this piezometer was installed as a temporary pre-pack well that contains bentonite in the water column. AOC-1-MW03 will serve as a permanent replacement well.

- Team reviewed top of bedrock and top of till maps showing the depth of bedrock and agreed to move BR-05 from the former Mess Hall to around the road interception located west of the Barracks to obtain bedrock information from within that zone of bedrock depression.

- Team acknowledged that the sediment and pore water samples along Winsor Brook are subject to change based on the locations of depositional area identified in the field and right-of-entry agreements being coordinated between CENAE and property owners to allow for safe access to sampling locations.

- CENAE recommended a sediment and pore water sample be collected in the unnamed stream where it intersects a large wetland complex to the south of the Site.

QAPP Worksheet #9-4: Project Scoping Session Participants Sheet

Project Name: Remedial Investigation **Site Name:** Former NIKE PR-79 Control Area
Projected Date(s) of Sampling: Spring 2020 **Site Location:** Foster, Rhode Island
Project Manager (Contractor): Gregory Hencir
Date of Session: 21 November 2019
Location of Session: Former NIKE PR-79 Control Area, Foster, RI
Scoping Session Purpose: Review Proposed Background Study Area and Sediment Sampling Locations

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John Shannon	Geologist	AECOM	401-854-2835	John.Shannon@aecom.com	Field Team Leader

The following bullets summarize the significant topics that were discussed and decisions made during the meeting that contribute to the RI QAPP.

Background Study Area

- The proposed background study area is in the easternmost portion of the Site near the perimeter fence. Early aerial photographs from 1951 show that this area was forested and cleared sometime between 1962 and 1963 (USAGC, 2015). Field observations during the Site visit indicate some evidence of surficial disturbance, such as young trees that likely reestablished after clearing in the early 1960s and boulders that were likely moved by heavy equipment during clearing. The team did not observe indications of development except for the perimeter fence and three half-drums that were believed to be placed for baiting game. The background study area is located beyond an area where concrete blocks were dumped as labeled "ground scars" on historical aerial photographs (USAGC, 2015). CENAE visually inspected soil collected from a hand auger that appeared native, except for some surficial disturbance likely due to previous clearing activities. A visual characterization of soils in the background study areas located beyond the limits of the AOCs were found to be of the same soil type as native soils observed in the AOCs. The team agreed that the proposed background study area would be suitable for background testing in the RI.

- Team discussed the possibility of multiple background study areas; however, it was concluded that a dataset collected from one background study area is preferred for the RI.

Sediment Sampling

- Team reviewed AECOM's draft map showing proposed co-located sediment/pore water/surface water sample locations overlain by bedrock lineaments and a preliminary map showing Groundwater Screening Level Modelling that was prepared by CENAE in 2011. CENAE noted that the highest elevation is located on Steere property; therefore, surface water from the Site presumably flows towards the west and south. CENAE explained that the Groundwater Screening Level Model shows groundwater flowing from the Site towards the west and south. Based on the inferred surface and groundwater flows from the Site, the team decided to reduce the number of sediment/pore water/surface water samples from a small stream located northeast of the Property from four samples to one sample. The rationale for that one remaining sample is for groundwater delineation in the northwestern direction.

- Team walked from AOC-4 to Winsor Brook. The wastewater outfall from AOC-4 was identified near the northeastern corner of former utility shed located within the perimeter fence. A shallow dug trench was observed from that wastewater outfall to Winsor Brook.

- Team walked a short stretch of Winsor Brook where proposed co-located sediment/pore water/surface water samples will be collected. The stream is a straight chute, rocky, fast-moving, and starved of sediment in the steep-gradient stretch near AOC-4; however, some pockets of coarse sand exists between boulders/cobbles. Team concurred that coarse sediment in Windsor Brook came from a more recent upstream source and would not be suitable for sampling in the RI. Adequate depositional areas for sampling were not observed in Windsor Brook. Small tributaries observed along the eastern bank of Winsor Brook that drain the abutting forested swamp were also inadequate for sampling. Team agreed to collect sediment/soil and pore water samples from the edge of the eastern bank of Winsor Brook as a substitute for collecting sediment samples from the stream bottom. Surface water samples would be collected from within Winsor Brook. CENAE noted the seasonal nature of seep hydrology and recommended sampling between March through June.

- Team discussed possible background study areas for sediment/pore water/surface water samples. Team concurred that the upstream portion of Winsor Brook is suitable as a background study area. It was noted that background samples will need to be collected under similar conditions as the normal RI samples (i.e. surface water in-steam and sediment and pore water from areas along the bank) to maintain similar conditions (i.e. TOC and grain size). Team agreed to collect 10 sediment/pore water/surface water background samples to allow for a statistical comparison to normal RI samples.

- Team toured a portion of 33 Winsor Road with the residential property owner Ms. Karen Squillacci. Two streams (i.e. an underground stream to the west, and "Fairy Glenn," as referred to by Ms. Squillacci, to the east) were observed flowing into the pond from paralleling valleys. Based on a topographic map, both streams appear to originate from the wetland complex located at a higher elevation south of the Site. Both streams may interact with potential seeps as the tributaries descend the valleys towards the pond. Team agreed to collect sediment/pore water/surface water samples at each stream culvert located upstream of the pond. A sediment/pore water/surface water sample was proposed from the pond. Ms. Squillacci said the basin was sandy throughout. However, upon inspection of the pond shoreline, the team observed organic material and fines ("muck") along the northern shore of the pond that would be adequate for sampling.

- Team walked north along a portion of Winsor Brook from the Windsor Road bridge. This stretch of Winsor Brook was observed as having a lower gradient with riffle and pool habitat. There were pockets of depositional areas with sand that were visible in the channel. The team discussed sampling upstream and away from a confluence coming in from the west. The team also agreed to collect a sample from the dug well located nearby at 41 Windsor Road.

- Team toured a portion of 29 Winsor Road, which is at a low elevation and the confluence from the pond at 33 Winsor Road, Winsor Brook, and the Ponaganset River. Discussed this location for monitoring groundwater elevations and possibly for evaluating fate and transport from the Site since deep groundwater discharge would be expected here. Team agreed to collect groundwater elevations from two gauging stations at this low elevation area. Based on a review of the gauging data, the team will decide whether an additional sample will be collected near the Ponaganset River.

QAPP Worksheet #10: Conceptual Site Model

10.1 Overview

This worksheet presents general background information and the preliminary CSM for the Site. This discussion of the existing CSM is intended to identify potential data gaps and provide the basis for developing DQOs, study goals, and the appropriate technical approach for the sampling and analysis program to complete the RI phase of the CERCLA process. This preliminary CSM is subject to change based on the results of this investigation.

10.2 Conceptual Site Model Summary

The following subsections briefly summarize the preliminary CSM and identify data gaps to provide the basis and framework for developing the QAPP to complete the RI. It is divided into the specific AOCs identified for investigation, shown in **Figure 10-1**.

AOC-1 Radar Area

Study Area: The study area includes a former helicopter pad and former target acquisition and tracking radar equipment located at Radar Pad A, Radar Pad B, Radar Pad C, Radar Control Van, Battery Control Van, Frequency Changer/Generator building (including a floor drain), associated cesspools and a dry well. The study area includes an area north of the former Radar Control Van and Battery Control Vans where stunted vegetation growth was observed, and a former ground scar area to the east of the former Frequency Changer/Generator building identified in the HPA that may have been caused by dumping (USAGC, 2015).

The floor drain was previously investigated in the initial phase of the RI (AMEC, 2014b). Upon uncovering the floor drain and associated drainage trench, AMEC advanced soil boring SB-019 through the opening below the steel grate and installed groundwater well PZ/GW-019. Another soil boring ("SB-020") was advanced through a chiseled hole in the trough base to investigate the floor drain. The initial phase of the RI found VOCs (including TCE at an estimated low concentration (0.0009 J mg/L)) in a groundwater sample collected at PZ/GW-019 (Section 2.10.2 Groundwater Results, AMEC, 2014b). The RI will investigate whether VOCs are present in underlaying overburden/weather bedrock at this location with soil boring/overburden monitoring well PR79-SB-104/PR79-MW-003.

Potential Impacts: TCE, pentachlorophenol, and naphthalene and other solvents used as a cleaning and degreasing agent and used for potential de-icing the access road leading up to the Radar Area and helicopter pad.

Potentially Impacted Media and Data Needs: Surface soil, subsurface soil, and groundwater. Additional data needs are required to complete the Site delineation, including: VOCs[1], SVOCs[2], metals[3], SPLP metals, pH, TOC, grain size, and monitored natural attenuation (MNA) parameters.

AOC-2 Operations and Maintenance Area

Study Area: The study area includes the former Mess Hall, Barracks, and Administration buildings, water pump house, former transformer area, and reported motor pool vehicle maintenance areas located northwest of the former Guard Post along Theodore Foster Drive and northwest of the former water storage tank and pump house. The study area also includes an "unidentified pipe" observed west of former water pump house which is approximately 23 feet deep and is thought to be an abandoned borehole.

The transformers and associated electrical equipment were removed by the Town of Foster after being transferred to the Town of Foster in 1965 for beneficial use. The former transformer area was previously investigated twice, and the results from surficial soil samples collected at "S-5" (CDM, 1994) and "SB-015" (AMEC, 2014b) indicated PCBs were not present at concentrations above laboratory reporting limits (refer to Appendix B Laboratory Reports, CDM, 1994 and Table 2-7, AMEC, 2014b). Based on the findings of these previous investigations and that the electrical equipment was beneficially used and removed by the Town of Foster, further investigation of the former transformer area is not planned in the RI.

Potential Impacts: TCE and other degreasing solvents related to the maintenance of former buildings and potentially used for motor pool vehicle maintenance. Gasoline, diesel, and motor oil leaks and spills related to former motor pool vehicle maintenance.

Potentially Impacted Media and Data Needs: Surface soil, subsurface soil, and groundwater. Surface water, pore water, and sediment in nearby drainage swales. Additional data needs are required to complete the Site delineation for soil and groundwater, including: VOCs, SVOCs, metals, SPLP metals, pH, TOC, grain size, and monitored natural attenuation (MNA) parameters.

[1] Owning to their volatility under ambient conditions, it is very unlikely significant concentrations of VOCs will be detected in surface soils resulting from potential releases that are several decades old. Additionally, prior historical data collected including passive soil gas as well as surface soil data have not detected any chlorinated VOCs to date. Therefore, VOCs will not be analyzed in surface soil samples (i.e.: 0-2 feet bgs).

[2] SVOCs analysis will include PAHs, 1,4-dioxane, 2-methylnapthalene, and pentacholorphenol based on analytical results from the SI.

[3] Metals analysis will include the full list of TAL metals based on analytical results from the SI. Total chromium will be analyzed initially for comparison to background concentrations. If total chromium concentrations exceed background concentrations, then speciation of chromium will be performed.

Surface water, pore water, and sediment will be sampled for VOCs, SVOCs, metals (total and filtered), hardness, AVS/SEM, pH, TOC, and grain size.

AOC-3 Southern Leach Field

Study Area: The study area includes the former southern leach field, former distribution box, and drain line connected to the Western Sewage Disposal Area.

Potential Impacts: TCE and other degreasing solvents used as cleaning agents and paint waste disposed through the septic system.

Potentially Impacted Media and Data Needs: Surface and subsurface soil and groundwater. Additional data needs are required to complete the Site delineation, including: VOCs, SVOCs, metals, SPLP metals, pH, TOC, grain size, and monitored natural attenuation (MNA) parameters.

AOC-4 Western Sewage Disposal Area

Study Area: The study area includes the former sand filtration beds, former chlorine detection chamber and former utility shed located at the Western Sewage Disposal Area.

The location of the utility shed in the Western Sewage Disposal Area (AOC-4) was investigated in the initial phase of the RI with soil boring SB-023 (AMEC, 2014b). AMEC did not identify COPCs exceeding screening levels at SB-023, except arsenic that was reported in the surface soil sample at the concentration (2.69 mg/kg) above the 2014 Residential Soil RSL and RDEC. The RI will investigate the downgradient location of the utility shed with soil boring PR79-SB-119

Potential Impacts: TCE and other degreasing solvents used as cleaning agents and paint waste disposed through the septic system.

Potentially Impacted Media and Data Needs: Surface soil, subsurface soil, and groundwater. Surface water, pore water, and sediment in drainage swales, Winsor Brook or other area water features. Additional data needs are required to complete the Site delineation for soil and groundwater, including: VOCs, SVOCs, metals, SPLP metals, pH, TOC, grain size, and monitored natural attenuation (MNA) parameters. Surface water, pore water, and sediment will be sampled for VOCs, SVOCs, metals (total and filtered), hardness, AVS/SEM, pH, TOC, and grain size.

AOC-5 Western Disposal Area

AOC-5 was used for residential and agricultural dumping by the property owner and is not attributable to former DoD activities; therefore, AOC-5 is ineligible for investigation and cleanup under DERP-FUDS.

Additionally, three heating oil USTs that supplied the Barracks, Administrative, and Mess Hall buildings were transferred to the Town of Foster in 1965 and currently remain in-place, except for the UST adjacent to the Mess Hall building, which was reportedly leaking as of November 2018 and removed by the Town of Foster in September 2019. The three USTs were beneficially used by the Town of Foster; therefore, ineligible for investigation and cleanup under DERP-FUDS.

10.3 Data Gap Analysis

The review of the historic information and working CSM has identified data gaps in the technical understanding of the hydrogeology, migration pathways, and nature and extent of impacts. The following briefly outlines some of the data gaps which will be developed into data objectives and goals for the RI in subsequent Worksheets.

Hydrogeology and Migration Pathways

Groundwater flow: The primary understanding of Site hydrogeology is based on the bedrock water supply wells, which are open borehole wells completed for hundreds of feet through bedrock and a limited on-Site overburden monitoring well network. The current Site monitoring well network is limited to overburden piezometers which insufficiently characterize groundwater flow from the overburden to the weathered bedrock and bedrock unit. The existing overburden monitoring well network consists of 14 piezometers which are screened from approximately 3 to 24.5 ft bgs. Outside of the six water supply wells, there are no weathered bedrock or bedrock monitoring wells on-Site. New overburden, weathered bedrock, and bedrock monitoring wells are required to assess the groundwater interaction among the overburden, weathered bedrock, and bedrock units. New monitoring wells will be in specific areas where limited information exists regarding groundwater flow and potential transport to nearby receptors.

Geologic contacts: Much of the geologic data used in development of the working CSM is based on information obtained from installation of the overburden piezometers, surface geophysics, and a visual evaluation of bedrock outcrops. None of the existing piezometers are screened through the complete overburden through the cobble/boulders bedrock or below weathered bedrock. Additional monitoring wells are needed to confirm the saturation, chemistry, depth, and thickness

of permeable unconsolidated deposits and weathered bedrock. New monitoring wells will be screened in each of the lithologic units (i.e., overburden, weathered bedrock, and bedrock).

Estimates of hydraulic conductivity: The existing hydraulic conductivity estimates from the overburden piezometers may not be fully representative of aquifer conditions, since the piezometers are screened in overburden above the cobble/boulders as well as weathered bedrock. Additional hydraulic conductivity data of the overburden, weathered bedrock, and bedrock is needed to further assess the interaction among the water bearing units at the Site.

Surface bedrock fracture characterization and mapping: A fracture trace analysis (USAGC, 2016) and three surface geophysics studies (CENAE, 2003; AMEC, 2017a; HGI, 2017) were completed and reviewed (Hager-Richter, 2017). These data have been used to help identify well locations and potential surface water sample points in identified nearby surface water bodies that might intercept fracture traces (Woodard & Curran, 2019). These data need to be supplemented by bedrock drilling and borehole geophysical surveys.

Downhole bedrock fracture characterization: Additional bedrock wells are needed to intercept and further characterize potential migration pathways via transmissive fractures and measure the vertical component of the hydraulic gradient within bedrock.

Observed hydraulic interconnectivity between wells: Additional data are needed from pumping isolated fractures to assess interconnected fractures in nearby bedrock wells and between the bedrock and overburden.

Nature and Extent of Impacts in Soil, Overburden, and Bedrock

Plume delineation (horizontal and vertical): The existing dataset for soil and groundwater is not sufficient to adequately assess the horizontal and vertical extent of impacts, particularly in deep overburden, weathered bedrock and bedrock. The current data is limited with respect to the media assessed and the likely pathways which COPCs migrated over time. The Phase I site investigation concluded that TCE, pentachlorophenol, and naphthalene are the only Site-related COPCs identified in overburden groundwater and are isolated to the area surrounding PZ-019 located immediately adjacent to the former Frequency Changer/Generator Building (AMEC, 2014b). Additional data is needed to assess this potential source area in overburden, identify whether a discrete or diffuse continuing source exists, and assess transport mechanisms in weathered bedrock and bedrock. This will be achieved by installing new monitoring wells screened to target permeable overburden and transmissive bedrock fractures and conducting sampling for

COPCs and geochemical parameters to understand the chemical and physical processes impacting the plume.

10.4 Description and Current Use

The Former NIKE PR-79 Control Area (Property) is located in Providence County, in Foster, Rhode Island, as shown on **Figure 10-1**. The coordinates for the Site obtained from the US Geological Survey (USGS) 7.5 minute Quadrangle for Clayville, Rhode Island are approximately:

- Latitude: N41° 50' 32"
- Longitude: W71° 42’57"

The Property is located at the end of Theodore Foster Drive. According to the Town of Foster Tax Assessor Online database, the address is 23 Theodore Foster Road and recorded on Lot 10, Map 18 (Town of Foster, 2019). The parcel is 6.62 acres in size[4]. The land is zoned for municipal use.

- The area surrounding the Former NIKE PR-79 Control Area is comprised of northern hardwood forest and rural development. The Property is located on top of Oak Hill and residences, farms, and businesses are located south of the Property. Three residences are located within 300 to 400 ft of the Property with approximately 68 residences located within a one-mile radius with private water supply wells in Foster and North Scituate, Rhode Island (USAPHC, 2010). The majority of these homes are located along Maple Rock Road, Winsor Road, and Old Hartford Pike. The nearest residential private water supply well (ROU-1) is located approximately 200 ft east of the Property. Businesses located near the Property include solar panel arrays located to the northeast and southeast of the Property. The location of on-Property supply wells (NIKE-1, NIKE-2), nearby residential water wells (ROU-1, ROU-2 and ROU-3) and solar panel arrays are shown on **Figure 10-1**.

The Foster-Glocester Regional School District, Northwest Special Education Region currently occupies the former Mess Hall building for administrative purposes. The former mess hall building, which is the only building on the Former NIKE PR-79 Control Area property, was temporarily declared "unsafe" in July 2020 by the Town of Foster Building Inspector due to potential asbestos containing building

4 Town of Foster Tax Assessor Online database indicates the parcel is 6.62 acres in size; however, there is conflicting information from other sources regarding the exact acreage. The DERP-FUDS Inventory Project Report indicates the Site is 19.59 acres in size (13.36 acres and a 6.23-acre easement). Historic reports from Camp Dresser & McKee, Inc. (CDM) and CENAE reference 8 acres (CDM, 1994; CENAE, 2003). On September 22, 1995, 1.38 acres was transferred to Lot 11 on Plat 18 (AMEC, 2013).

materials. The Town of Foster is considering demolishing that building and future use of the property as a solar array

10.5 Topography and Geology

The Former NIKE PR-79 Control Area is located on top of Oak Hill at approximately 620 ft above mean sea level (msl). The surrounding terrain is characterized by low hills and shallow valleys. The elevation of Winsor Brook is approximately 470 ft above msl.

The surficial geology in the vicinity of the Site is made up of glacial-fluvial deposits in the valleys and lodgment and ablation till along the hills. The glacial outwash is composed of sand and gravel interbedded with silt and clay (CENAE, 2003). The units form unconsolidated and generally well sorted and stratified sequences that reach up to 50 ft thick in the area surrounding the Site. Site-specific geophysical and boring log information indicates a much thinner layer of overburden of between 5 to 25 ft thick (AMEC, 2014a). Boulders, sand, silt, and clay are found within the poorly sorted and unstratified glacial materials. Glacial till forms a thin, discontinuous mantle over the bedrock surface averaging 20 ft in thickness (CENAE, 2003).

Below the overburden material is a layer of weathered bedrock. Surface geophysics performed at the Site indicate the thickness of this layer varies between one and 15 ft beneath the Site. However, the weathered bedrock is sometimes thicker and the bedrock surface elevation lower in areas of highly fractured bedrock. The bedrock beneath the Site is composed of the South Foster Migmatite and Ponaganset Gneiss of the Esmond Igneous Suite. The South Foster Migmatite consists of a heterogeneous composite of quartz-biotite schist and quartzite members. The Ponaganset Gneiss is a coarse grained, porphyritic, pink to gray diorite gneiss. The gneiss is the most predominant rock type across the Site (Johnson Company, 2018).

Based on borehole geophysics and outcrop field observations, the competent bedrock is characterized by steeply dipping fractures and occasional fracture zones spaced from approximately 4 to greater than 10 ft apart. The orientations of field-measured bedrock fractures are similar to the orientations of topographic lows feeding into Barden Reservoir located south of the Site, suggesting that ground surface topography is sometimes controlled by preferential erosion along zones of closely spaced bedrock fractures (Johnson Company, 2018).

The steeply dipping fracture orientations in bedrock predominantly strike north-northwest (NNW) to south-southeast (SSE) and north-northeast (NNE) to south-southwest (SSW) on Site, although north to south and east-northeast (ENE) to west-southwest (WSW)-striking fractures are also present. The

primary water-bearing fracture sets encountered in Site wells NIKE-1, NIKE-2, ROU-1, ROU-2, and ROU-3 strike NNE to SSW and NW to SE and dip moderately (The Johnson Company, 2018).

South of the Site, NNW to SSE and northwest (NW) to southeast (SE)-striking fractures are more prevalent (USAGC, 2016), reflecting the primary orientation of the Ponaganset River drainage to Barden Reservoir. These fracture strike orientations are reflected in the topographic drainage patterns, suggesting that the bedrock joints and associated fracture sets represent preferential erosional zones.

10.6 Hydrology and Hydrogeology

There are no surface water bodies in the Former NIKE PR-79 Control Area (Site). Surface water runoff from the Site is directed into drainage ditches which flow along the slopes of Oak Hill. Surface water not captured by these ditches infiltrates or flows radially in all directions, since the Former NIKE PR-79 Control Area is located on top of a hill. The nearest surface water bodies include three streams and a 16 acre wetland complex located approximately 0.25 mile to the south; a 0.15 acre wetland followed by Winsor Brook located approximately 0.25 miles to the west; and a 0.07 acre open water body to the north of the Site as shown on Figure 17-3 (Woodard & Curran, 2019). The Site is located within the Scituate Reservoir Watershed and is within the Scituate Reservoir Protection Area. The northwestern most portion of the Scituate Reservoir (known as the Barden Reservoir) is located approximately 3 miles southeast of the Site. Winsor Brook is a tributary to the Ponaganset River, which flows into the Barden Reservoir. Local potable water is supplied with private bedrock drinking water supply wells, not municipal water. There are no wellhead protection areas within one-mile of the Site. The groundwater beneath the Site is classified as Group GA which is presumed to be suitable for drinking water.

The main source of overburden groundwater at the Site comes from infiltration. Precipitation and melt water infiltrate and likely form a saturated zone in the coarse-grained sand and gravel above the less permeable till and bedrock. Over time, the overburden groundwater slowly drains into the weathered bedrock and competent bedrock fractures below or migrates radially from the top of the hill (Johnson Company, 2018). Groundwater within bedrock likely receives water from a series of fractures which are recharged from the overlying glacial overburden.

Surface geophysics, fracture trace analysis, and borehole geophysics investigations have been completed at the Site and are discussed in Section 10.7. The integration of these data indicates that fracture strikes trend primarily north-south and dip steeply to the southwest and southeast. Large low-velocity anomalies indicating fracture zones at the Site were identified along seismic and GPR geophysical lines (Hager Geoscience, Inc. [HGI], 2017). The orientation of fractures observed at

outcrops and identified in fracture domain analysis provide evidence that a fracture zone could connect supply wells NIKE-1 and ROU-1 (United States Army Geospatial Center [USAGC], 2016). Borehole geophysical logging at supply wells NIKE-1, NIKE-2, ROU-1, ROU-2 and ROU-3 identified eight water producing fracture sets dipping to the southwest that intersect two or more supply wells (Johnson Company, 2018). The deepest three fracture sets were identified as primary water producing fractures. During 2013, AMEC performed four-hour pump test in NIKE-2 to assess hydraulic connectivity between NIKE-1, ROU-1, ROU-2 and ROU-3. AMEC concluded that NIKE-1 and ROU-1 are hydraulically connected and there is evidence of weak interference between NIKE-1 and NIKE-2 (AMEC, 2014a).

Groundwater flow from the Site has a radial character, reflecting the elevated topography of the Site with components of flow ranging from the west to southeast. There is an ENE drainage from Oak Hill, as well, but that is located over 1,000 ft northeast of the previously active portions of the Site and therefore likely has less potential for influencing contaminant transport. The predominant topographic drainage patterns in near-Site area are generally SSW to the west of the Site and SSE to the south and east of the Site. The westerly and south-southeasterly drainage features converge south of the Site and converge with the Ponaganset River in close proximity to one another approximately 0.7 miles SSW of the Site.

10.7 Operational History and Environmental Areas of Concern

The Property was originally developed for agricultural use, namely as an apple and peach orchard. The US Government acquired the subject property between 1955 and 1957 and developed it for radar missile tracking as part of the NIKE Missile Defense System. NIKE sites were constructed throughout the continental US in the mid-1950s during the Cold War era to defend major industrial and urban areas. The location of NIKE PR-79 was selected for defense of Providence, Rhode Island. NIKE sites generally consisted of a missile launcher area and a separate integrated fire control and radar missile tracking area (NIKE control area) which typically operated less than two miles apart. The launcher area is where missiles were stored, maintained, and if necessary, launched. The NIKE control area is where radar and communication equipment needed to detect potential targets and guide launched missiles were maintained and stored. The former launcher area for NIKE PR-79 is a separate property located on Winsor Road in Foster, Rhode Island designated FUDS Property/Site Number D01RI0063/01 and is not the subject of this document.

The Former NIKE PR-79 Control Area was reported as excess property by the General Services Administration (GSA) in 1964. In July 1965, the Site was closed and the Property was transferred to the Town of Foster. The Town of Foster used the former Mess Hall, Barracks, and Administrative

buildings as the Fogarty Elementary School until 1989 (RIDEM, 1992). The Foster-Glocester Regional School District, Northwest Special Education Region currently occupies the former Mess Hall building for administrative purposes.

The following structures were transferred to the Town of Foster in good condition for beneficial reuse:

- Mess Hall, Barracks, and Administrative buildings
- Heating Oil USTs supplying the Mess Hall, Barracks, and Administrative buildings
- Utility Lines
- Southern Leach Field

The locations of the above referenced structures are shown on **Figure 10-1**.

In 1988, the Foster Board of Education requested that CENAE investigate groundwater at the Former NIKE PR-79 Control Area to determine whether TCE detected by RIDEM in water supply wells was related to former DoD activities. CENAE conducted a field survey and Inventory Project Report (INPR) that same year, which concluded that former DoD activities may have resulted in the release of TCE to the environment. Based on the findings of the INPR, the Former NIKE PR-79 Control Area entered DERP and was designated FUDS Property/Site Number D01RI0063/02 (CENAE, 1988).

The INPR field survey identified a 6,000-gallon diesel fuel UST (originally assumed to be a 1,000-gallon UST) in the northeast corner of the Former NIKE PR-79 Control Area, next to the former Frequency Changer/Generator Building shown in **Figure 10-1**. The UST was removed in June 1994. There is limited documentation beyond that the UST was closed under RIDEM UST regulations and no additional UST investigation was recommended (ESS, 1994).

In March 1992, USEPA designated the Former NIKE PR-79 Control Area as Site Number RID987492485 in Comprehensive Environmental Response, Compensation and Liability Information System (CERCLIS). CERCLIS is a management system used by the USEPA to track activities at hazardous waste sites considered for cleanup under CERCLA. The Site is not included on the National Priorities List (NPL).

On August 24, 2000, RIDEM issued a Letter of Responsibility (LOR) to the US Army 94th Regional Support Command at Fort Devens, Massachusetts, indicating that a potential release of hazardous materials occurred at the Site and identified DoD as the potentially responsible party. The LOR requested the US Army conduct a site investigation of the source area in accordance with Rhode

Island Remediation Regulations 250-RICR-140-30-1. On September 5, 2000, the US Army 94th Regional Support Command sent a response letter to RIDEM refuting ownership of the Site (The Johnson Company, 2018).

Beginning in 2001, CENAE conducted a series of residential water supply well sampling events, which targeted two on-Property water supply wells (NIKE-1, NIKE-2) and three off-Property residential water supply wells located at 23A Theodore Foster Drive (ROU-1, ROU-2 and ROU-3) that are in close proximity to the Former NIKE PR-79 Control Area. Based on the analytical results for the residential sampling, in 2002, a Time Critical Removal Action (TCRA) was initiated as a temporary remedy for groundwater impacts in drinking water. The TCRA included installation of point of use duel carbon filtration systems at one on-Property water supply well (NIKE-1) and three off-Property residential water supply wells (ROU-1, ROU-2, and ROU-3). CENAE continues to monitor these four carbon filtration systems.

Several previous site investigations have been performed, as discussed in Section 10.7. In 2013, AMEC conducted a passive soil vapor study that identified five AOCs (AMEC, 2013a):

- AOC-1: Radar Area
- AOC-2: O&M Area
- AOC-3: Southern Leach Field
- AOC-4: Western Sewage Disposal Area
- AOC-5: Western Disposal Area (ineligible for investigation and cleanup under DERP-FUDS)

Based on a review of historic aerial photographic analysis (USAGC, 2015), historical maps, and other documents regarding historical operations, there is no evidence to suggest fire training activities or other activities using per- and polyfluoroalkyl substances (PFAS) occurred at the Former NIKE PR-79 Control Area. Additionally, DoD activities at the Former NIKE PR-79 Control Area precedes dates of common PFAS use as aqueous film forming foam (AFFF). Therefore, PFAS is not a site-specific COPC.

10.8 Previous Site Investigations and Available Dataset

Previous investigations at the Former NIKE PR-79 Control Area have included a Preliminary Assessment (PA) and Site Inspection (SI), off-Property residential and on-Property water supply well sampling, soil, groundwater, and soil vapor studies, and surface and geophysical investigations. Key reports that provide historical data for the Site are summarized below.

RIDEM, 1992. Preliminary Assessment (PA), Foster NIKE Control Area, Theodore Foster Drive, Foster, Rhode Island.

In 1987, the Rhode Island Department of Health (RIDOH) conducted routine sampling at NIKE-1 and NIKE-2 and detected TCE at concentrations ranging from 14 to 99 micrograms per liter (µg/L), which are in excess of the 5 µg/L Safe Drinking Water Act (SDWA) maximum contaminant level (MCL) for TCE. In response, RIDOH expanded sampling to include 22 residential water supply wells in the vicinity of the Former NIKE PR-79 Control Area. RIDOH reported TCE in one water sample collected from residential water supply well ROU-1 at a concentration of 37 µg/L.

In October 1987, RIDEM screened soil gas with a PID behind the Administration Building and south of the Frequency Changer/Generator Building. RIDEM reported concentrations of VOCs in surficial soil gas at or slightly greater than the instrument detection limit (DL) south of the Frequency Changer/Generator Building.

This PA provides a summary of Site history and investigations. It contains a preliminary "Pathway and Environmental Hazard Assessment" for the groundwater, surface water, soil and soil gas pathways. For the groundwater and surface water hazard assessment, this PA inventories groundwater wells and surface water sources and provides estimates of the populations served by each and their distance from the Site. This PA concluded that additional investigation was necessary to further assess potential hazards resulting from the presence of TCE in water supply wells NIKE-1, NIKE-2, and ROU-1.

CENAE, 1988. Inventory Project Report, John E. Fogarty School (NIKE Site 79 Control Area).

In 1988, the Foster Board of Education requested that CENAE investigate groundwater at the Former NIKE PR-79 Control Area to determine whether TCE detected by RIDEM in water supply wells was related to former DoD activities. CENAE conducted a field survey and INPR that same year, which identified a 6,000-gallon diesel fuel UST (originally estimated to be around a 1,500-gallon sized UST) next to the former Frequency Changer/Generator Building. This INPR concluded that former DoD activities may have resulted in a release of TCE to the environment and recommended removal of the UST and a confirmation study of two suspected source areas of TCE impacts: the Southern Leach Field and Radar Control Area. The Former NIKE PR-79 Control Area entered DERP and was designated FUDS Property/Site Number D01RI0063/02.

Environmental Science Services, Inc. (ESS), 1994. UST Closure Assessment, NIKE Launch Site PR-79. Foster, Rhode Island.

A 6,000-gallon diesel fuel UST was removed from the Former NIKE PR-79 Control Area in June 1994. There is limited documentation in this report beyond that the UST was closed under RIDEM UST regulations and no additional UST investigation was recommended.

Camp Dresser & McKee, Inc. (CDM), 1994. Site Inspection Report, Former Foster NIKE Control Area Site.

In 1994, a SI was conducted by CDM on behalf of RIDEM. Field investigation activities included collecting an unfiltered groundwater sample from a faucet located in the water pump house that is supplied by NIKE-1 and NIKE-2 and collecting surficial soil samples from areas associated with potential sources of impacts based on field observations (e.g., areas of staining, drainage swales, etc.) The locations where surficial soil samples (identified as S-1 through S-8) were collected are approximated based on a description in this SI report. The SI report concluded that TCE was present in the overburden groundwater sample collected in the water pump house at a concentration of 7 µg/L, which is above the SDWA MCL of 5 µg/L. The report also concluded that semivolatile organic compounds (SVOCs) and lead were present in a surficial soil sample collected at S-1 located behind the former Administration Building where stained soil was observed during the PA at concentrations above background concentrations, which were estimated in a background soil sample (S-7) collected in a cleared area north of the Radar Control Area. Target analyte list (TAL) metals and SVOCs were detected at concentrations above background concentrations in soil samples that were collected in a drainage swale near the Barracks (S-4) and Radar Control Area (S-6D). The PCB Aroclor-1260 was detected at a concentration of 2.88 milligrams per kilogram (mg/kg) in a surficial soil sample collected at S-3 located approximately 50 ft south of the end of the access road extension. However, Aroclor-1260 was not detected above laboratory reporting limits in a surficial soil sample collocated where S-3 was believed to be been placed (AMEC, 2014a). The SI concluded that environmental media had been impacted by former DoD activities, but that the conditions did not pose an imminent danger to health or welfare.

CENAE, 2003. Limited Investigation within a Study Area at PR-79 Former NIKE Control Area, Foster, Rhode Island.

The study objectives of this limited investigation were to confirm TCE impacts at NIKE-1, NIKE-2, ROU-1, ROU-2, and ROU-3; characterize hydrogeology; develop a simple conceptual model; and, assess the risks that are posed by TCE impacts in groundwater. CENAE performed surface geophysics, bedrock geophysics, a pump test, and collected water supply samples for laboratory analysis.

In August 2001, USACE, Baltimore District (CENAB) and the Center for Environmental Restoration Systems of Argonne National Laboratory (ANL) of the US Department of Energy (DoE) completed surface geophysics and bedrock geophysics at the Former NIKE PR-79 Control Area and at the three

adjacent residential properties. ANL found that seismic refraction surveying along three profiles showed that the Site is covered with a variable thickness of till and weathered bedrock ranging from 4 to 16 ft thick, and while there are some subtle depressions in the bedrock surface, the bedrock surface does not indicate the presence of major topographic depressions, mounds or topographic highs, or fracture zones that could act as near-surface geologic control over COPC transport towards ROU-1, ROU-2, and ROU-3. ANL concluded from bedrock geophysics that groundwater recharge likely occurs near the Site and that the investigated wells draw groundwater primarily from one major fracture zone located approximately 180 to 530 ft bgs that intercepts all wells surveyed in this report except ROU-3, which has a low-producing fracture that may intersect another large fracture given that all the wells were identified as containing TCE. Geophysical findings from this investigation were later reinterpreted and revised (HGI, 2017; Hager-Richter, 2017).

In the spring of 2002, CENAE conducted a pump tests at ROU-2 and ROU-3, during which the water elevation of one well was monitored during pumping of the second well to check for hydraulic connection between the two wells. CENAE concluded that hydraulically connectivity between ROU-2 and ROU-3 is plausible, but the findings are inconclusive.

Between December 15 and December 18, 2000, RIDEM collected unfiltered water samples from 14 off-Site water supply wells and analyzed those samples for TAL metals and VOCs. The laboratory reports provided as **Appendix D** of this CENAE report indicate VOCs were not detected above laboratory reporting limits in any of the water samples tested, except MTBE, a gasoline additive developed in the 1980s, that was detected in one water sample collected from the residential water supply well located at 53 Maple Rock Road at a concentration of 7.2 and 7.53 µg/L and chloroform detected in one water sample collected from a residential water supply well located at 40 Winsor Road at a concentration of 1.67 µg/L. Since MTBE was developed in the 1980s after former DoD activities at the Former NIKE PR-79 Control Area ended, it was later concluded that MTBE is not attributable to former DoD activities and that MTBE might have been the result of activity/operations in the area unrelated to the Site (USAPHC, 2010).

In 2001, CENAE began a series of sampling events at NIKE-1, NIKE-2, ROU-1, ROU-2 and ROU-3. Unfiltered water samples were collected at the point of exposure (i.e. from the faucet or spigot) in May 2001, December 2001, March 2002, and May 2002. Water samples were also collected as grab samples from two off-Property dug wells less than 10 ft in depth at 23A Theodore Foster Drive. CENAE reported that that all five water supply wells contained TCE at concentrations greater than the SDWA MCL of 5 µg/L and four constituents (chloroform, cis-1,2-dichloroethene [cis-1,2-DCE], TCE, and isopropyl benzene) were detected in one or more of those water samples. VOCs were not detected above laboratory reporting limits in the grab water samples collected from the two dug

wells. The detection of cis-1,2-DCE at ROU-2 suggests that some biodegradation of TCE occurred; thus, this report recommended analyzing for natural attenuation parameters to better understand the extent to which biodegradation is occurring.

In April 2002, CENAE initiated a TCRA as a temporary remedy for groundwater impacts in drinking water. The TCRA included installation of duel carbon filtration systems at NIKE-1, ROU-1, ROU-2, and ROU-3. NIKE-2 is listed inactive as of 2003. This report recommended:

- On an annual basis, collect water samples before and after the first carbon filter to determine the TCE concentrations of untreated water and to detect if there has been breakthrough of the first filter. The annual sample should be collected during low groundwater conditions and thereby, possibly maximize COPC levels within the samples.

- Provide an annual report to RIDEM that documents the monitoring results and discusses the data collected during this annual sampling event.

- Continue annual sampling and analysis of wells and replacement of filters until the concentration of TCE in each water well is below SDWA MCL of 5 µg/L over three consecutive annual testing periods. At such time, and after concurrence with RIDEM, monitoring and replacement of filters will be discontinued. The filter systems will remain on the wells unless the residents request that CENAE remove them.

CENAE, 2007. Draft NIKE PR-79 Chemical Data Summary Report (2001 – 2006), Water Supply Wells, Foster, Rhode Island.

This report summarizes water supply sampling performed by CENAE from 2001 through 2006 at NIKE-1, ROU-1, ROU-2, and ROU-3. Before the carbon filtration systems were installed in April 2002, water samples were collected at the point of exposure (i.e. from the faucet or spigot). After April 2002, three water samples were collected at each location to assess conditions pre-filtration, between the two carbon filters, and post-filtration. Water samples were collected in May 2001, December 2001, March 2002, May 2002, May 2003, September 2004, October 2004, November 2004, August 2005, April 2004, and September 2006. Each water sample was tested for VOCs, including TCE and its degradation byproducts. All carbon filters were exchanged in April 2006 after a TCE detection of 2.9 µg/L was reported in ROU-1, which indicated breakthrough between the first and second carbon filters. CENAE collected confirmatory water samples on April 11, 2006 shortly after replacement of the carbon filters to establish baseline conditions. CENAE concluded that TCE was not detected in post-treatment samples at concentrations above laboratory reporting limit after duel carbon filtrations systems were installed.

CENAE, 2008. Draft Technical Memorandum, Evaluation of Vapor Intrusion Pathway at PR-79.

CENAE conducted a vapor intrusion evaluation around buildings on or near the Property. The objectives of this evaluation were to determine if there was a complete vapor intrusion exposure pathway for VOCs, primarily TCE, to receptors (e.g., residents) and if the estimated indoor air concentrations posed a potential health threat. The evaluation included risk characterization based on historical groundwater sampling data from 2001 to 2007 and vapor intrusion modeling using the Johnson-Ettinger Model. The calculated cancer risks for various scenarios considered in the study were less than the USEPA lower limit for lifetime increased cancer risk of 10^{-6}. Similarly, the calculated hazard quotients (HQs) associated with non-cancer health effects were below the USEPA HQ threshold of 1. Based on CENAE's understanding of Site conditions and the groundwater TCE concentrations used in the calculations, CENAE concluded that TCE impacts did not present an unacceptable human health risk due to migration of vapors into associated buildings. CENAE recommended that new information on subsurface conditions, COPC distribution and concentrations, source areas and preferential pathways should be included in future exposure pathway and risk assessments. USEPA's current vapor intrusion guidance (USEPA, 2015) recommends use of modeling be done in conjunction with other lines of evidence evaluated in a vapor intrusion assessment. Therefore, new information obtained as a result of the RI will be evaluated in accordance with current USEPA vapor intrusion guidance (USEPA, 2015) to determine whether further evaluation of a potentially complete vapor intrusion pathway is necessary.

Col. David T. Hottel (Hottel), 2009. Teleconference/Interview Record. Prepared by AMEC. Interview dated August 14, 2009.

On August 14, 2009, CENAE conducted an interview with retired US Army Colonel David T. Hottel who served as 1st Lt. Executive Officer at the PR-79 NIKE Battery from August 1957 through May 1958. According to Col. Hottel, solvents were used as a cleaning and degreasing agent at the Site, particularly for cleaning radar and electrical equipment in the Radar Control Area. Col. Hottel added that radar equipment was considered maintenance intensive, meaning that radar components were removed, cleaned with solvents, and replaced on a very frequent basis. This is consistent with the *Final Report NIKE Missile Battery, Environmental Conditions Assessment Guide* (USACE-EMCX, July 2003), which states that radar equipment required frequent cleaning, which included the removal and cleaning of air filters by dipping them into solvents and then recharging them with oil, and routine cleaning of equipment slides, cables and plugs, terminals, high voltage cables, and the areas surrounding the radar power supplies (USACE-EMCX, July 2003). Col. Hottel said that, in general, solvents were taken to the work location and excess solvent after cleaning was directly disposed on the ground at that location. According to Col. Hottel, solvents were for other purposes where it was found useful, such as a deicer for the access road

leading up to the Radar Control Area and helipad, and potentially for motor pool vehicle maintenance. Col. Hottel said an unofficial motor pool existed between the Barracks and the water storage tank at the bottom of the Radar Control Area access road. The runoff and drainage from the motor pool ran down the slope, behind the Barracks, and towards the leaching field.

USAPHC, 2010. Water Supply Management Project Number 31-EC-0D1R-10, Volatile Organic Chemicals Sampling at NIKE PR-79 Integrated Fire Control Area, Foster, Rhode Island.

In 2010, USAPHC collected samples from 13 off-Property water supply wells located within a half mile of the Former NIKE PR-79 Control Area, including NIKE-1, ROU-1, ROU-2, and ROU-3. USAPHC reported TCE in ROU-2 (5.2 µg/L) and an estimated value (i.e., J-qualified) in a residential well at 32 Winsor Road (0.1 J µg/L); MTBE in a residential well at 17 Winsor Road (0.2 J µg/L); cis-1,2-DCE in supply well ROU-2 (0.4 J µg/L); and, chloroform in a residential well at 54 ½ Maple Road (0.2 J µg/L) and 63 Maple Rock (0.3 J µg/L). TCE was not detected in post-treatment water samples.

Mr. Bob Steere (Steere), 2012. Final Meeting Minutes, Bob Steere Interview and Site Walk, Former PR-79 NIKE Control Area Foster, Rhode Island. Prepared by AMEC. Interview dated December 13, 2012.

On December 13, 2012, CENAE interviewed Mr. Bob Steere, who is an abutting property owner to the Property at 15 Theodore Foster Drive and is knowledgeable of former DoD activities at the Former NIKE PR-79 Control Area. According to Mr. Steere, an area approximately 850 ft west/northwest of the Site containing four large (approximately 50 by 50 ft) sand filters, which was discovered by CENAE during a site walk in 2009, is part of the Former NIKE PR-79 Control Area septic system (i.e., the Western Sewage Disposal Area). Mr. Steere stated that several partially buried drums, exposed drums and miscellaneous metal scrap located on an adjacent parcel approximately 200 ft north of the Western Sewage Disposal Area (i.e., Western Disposal Area) was used for residential and agricultural dumping by the property owner and is not attributable to former DoD activities.

According to Mr. Steere, the orchard at 15 Theodore Foster Drive used pesticides, fungicides, and herbicides from approximately 1920 through the 1990s. Spraying of chemicals and dusting (sulfur) was conducted in and around the orchard. Light oil was usually mixed with a fungicide (Captan) to spray the orchard. Mr. Steere indicated that the empty drums in the Western Disposal Area were the light oil drums.

AMEC, 2013a. Passive Soil Vapor Survey Trip Report, Former NIKE PR-79, Foster, Rhode Island.

AMEC conducted a passive soil vapor study in selected AOCs on the Site to identify if VOCs, including chlorinated constituents and petroleum constituents, were present in soil or shallow groundwater.

The results of the survey were to be used to assess the need for, and optimal placement of, previously proposed Phase I site investigation soil, groundwater, and surface water exploration locations. This passive soil vapor survey included screening soil vapor using a PID for VOCs and collecting passive soil vapor samples from within the radius of influence of the vapor probes for analysis of VOCs, SVOCs, and petroleum constituents. Sixty-two passive soil vapor samples were collected and analyzed. VOCs (including TCE) were not reported above laboratory reporting limits in any of the soil vapor samples, except toluene that was detected in nine passive soil vapor samples at concentrations between 25 to 83 nanograms. Because the detected concentrations were low, AMEC suggested that the toluene detections may reflect ambient, atmospheric conditions or possibly residuals from commercial products containing toluene. AMEC also noted that toluene was used in a variety of commercial/industrial processes, while the use of TCE at the Site was more limited. AMEC concluded that the absence of VOCs (specifically TCE) in the passive soil gas samples indicated that these COPC were not likely present in the vadose zone or off-gassing from the water table beneath the areas tested because the chemical and physical properties of TCE would make it more likely than toluene to be detected in the samples.

AMEC identified the following five AOCs:

- AOC-1: Radar Area
- AOC-2: O&M Area
- AOC-3: Southern Leach Field
- AOC-4: Western Sewage Disposal Area
- AOC-5: Western Disposal Area

This Remedial Investigation / Feasibility Study (RI/FS) Work Plan summarized historical inspections, studies, and interviews to identify data gaps and develop a phased RI/FS field program. In developing this RI/FS Work Plan, AMEC discovered the following information:

- A hand-dug, stone-lined feature in the basement of the house at 23A Theodore Foster Drive was discovered during a CENAE groundwater sampling event. This feature is sometimes dry, and at other times has contained water that is presumed to be an expression of groundwater during periods when the water table is elevated in the overburden. Groundwater from this feature was sampled for VOCs in September 2011 and no VOCs were detected above the laboratory reporting limit.

- On December 13, 2012, AMEC personnel examined an "Unidentified Pipe" located in AOC-2. AMEC's evaluation of the "Unidentified Pipe" found water at approximately 13 ft below the top of the 6-inch steel pipe casing and a hard bottom was encountered at approximately 23 ft. VOC vapors were not detected in the "Unidentified Pipe" by PID. AMEC concluded that the pipe was an abandoned borehole.

AMEC, 2014a. Phase I Field Site Investigation Trip Report, NIKE 2, Former NIKE PR-79, Rhode Island.

This trip report summarized Phase I site investigation activities conducted in 2013 that included a pump test; bedrock geophysics; soil boring and monitoring well installation; surface water, pore water, sediment and groundwater sampling; a supplemental drum investigation; and, three-dimensional groundwater visualization. A summary of these investigation activities is provided below.

Pump Test

A pump test was conducted to assess hydraulic connectivity between NIKE-1, NIKE-2, ROU-1, ROU-2 and ROU-3 and was performed by installing transducers in each water supply well and pumping one well while monitoring water level changes in the other wells. AMEC found that pumping ROU-1 produced a visible response in NIKE-1. No response was observed in the other three wells while pumping ROU-1. The Memo concludes that NIKE-1 and ROU-1 are hydraulically connected and there is evidence of weak interference between NIKE-1 and NIKE-2. Data from the 4.5-hour pumping test on NIKE-2 was used to estimate a transmissivity of 29.5 ft^2/day.

Bedrock Geophysics

Bedrock geophysics was completed by Geophysical Applications, Inc. (GAI) on NIKE-1, NIKE-2, ROU-1, ROU-2 and ROU-3 in June and July 2013 that included measuring borehole temperature, fluid conductivity, and overall well condition to determine fracture characteristics such as depth, strike and dip, and aperture. Geophysical findings from this investigation were later reinterpreted and revised (HGI, 2017; Hager-Richter, 2017).

Additionally, six groundwater samples were collected from each well at inferred fracture zones ranging in depth from 26 ft bgs in ROU-1 to 628 ft bgs in ROU-3. Groundwater samples were analyzed for VOCs. AMEC reported TCE in 11 of the 25 samples. NIKE-1 and ROU-1 each had a detection of TCE at the deepest sample. NIKE-2 had no detections of TCE above laboratory reporting limits. Both ROU-2 and ROU-3 had detections of TCE in all sample depths (with the exception of ROU-3 at 82 ft bgs) ranging from 0.8 to 4.5 µg/L. Three additional VOCs (bromodichloromethane,

chlorodibromomethane and chloroform) were detected in water samples that AMEC concluded were likely related to the disinfecting of bedrock wells following the geophysical investigation.

Soil Boring and Monitoring Well Installation

In July 2013, AMEC advanced 22 soil borings to refusal using a direct push track mounted drill rig to depths ranging from 4.5 to 24.5 ft bgs. Two soil borings were placed to characterize the area around a former dry well in the Radar Control Area, the area north of the Main Gate where vehicle maintenance reportedly occurred, and the area around two steel grates found in the concrete slab of the former Frequency Change Generator Building. One surficial soil and one subsurface soil sample was collected from each boring for analysis of VOCs, SVOCs, TAL metals, total organic carbon (TOC), and grain size. Pre-packed wells with 2-inch PVC risers and 10 ft screens were installed in overburden to bottom depths ranging from 8 to 16 ft bgs at 14 selected soil boring locations (identified at PZ-001 through PZ-014).

VOCs were not detected in surficial soil samples, except 1,1-DCE that was estimated in surface soil samples collected from SB-003 (Radar Pad C) and SB-005 (Radar Pad B) at concentrations of 20.1 J and 7.8 J µg/kg, respectively, which is below the 2014 Residential Soil Regional Screening Levels (RSLs) and Rhode Island Residential Direct Exposure Criteria (RDEC) used for reference. SVOCs were detected above laboratory reporting limits in surficial soil samples collected from SB-019, SB-008, and SB-020 at concentrations above the 2014 Residential Soil RSL and RDEC; and arsenic was detected in all samples above the 2014 Residential Soil RSL, but below the RDEC. VOCs were not detected in subsurface soil samples, except 1,1-DCE which was detected or estimated in SB-005 and SB-022 at concentrations of 6.1 and 1.6 J µg/kg, respectively, which is below the 2014 Residential Soil RSL. SVOCs were detected above laboratory reporting limits in surficial soil samples collected from SB-019, SB-008, and SB-020, but at concentrations below the 2014 Residential Soil RSL. TAL metals were not detected above the 2014 Residential Soil RSLs, except arsenic and thallium which were detected in six subsurface soil samples above the 2014 Residential Soil RSLs.

Surface Water, Pore Water, Sediment and Groundwater Sampling

A combination of surface water, pore water and sediment samples were collected at ten locations between June 25 and July 8, 2013. A sediment sample was also collected from the grease trap located at the former Mess Hall building. All samples were analyzed for VOCs, SVOCs and TAL metals. Sediment was also analyzed for TOC and grain size and the surface water metals data was used to calculate hardness.

No VOCs were detected in the surface water samples. Two SVOCs (Benzyl alcohol and Di-n-butylphthalate) were detected in only one of the eight surface water samples at concentrations below the 2014 Tap Water RSLs used for reference. Metals were detected in the surface water samples at concentrations below the Ambient Water Quality Criteria (AWQCs) and 2014 Tap Water RSL, except arsenic which was detected in surface water sample SW-008 and manganese which was detected in surface water sample SW-006. AMEC concluded that none of the constituents detected in surface water samples are Site-related or derived from former DoD activities and proposed no further testing of surface water in a Phase II site investigation.

No VOCs were detected in the pore water samples, except toluene which was estimated in pore water sample PW-008 at a concentration of 0.3 J µg/L, which is below the 2014 Tap Water RSLs used for reference. One SVOC (di-n-butylphthalate) was detected in one of the eight pore water samples at a concentration below the 2014 Tap Water RSL. Metals were also detected in pore water samples; however, only arsenic was detected in pore water sample (PW-006) above the 2014 Tap Water RSL. AMEC concluded that none of the constituents detected in pore water are Site-related or derived from former DoD activities and proposed no further testing of pore water in a Phase II site investigation.

Several VOCs were detected in the sediment samples (acetone, 2-butanone, etc.); however, none exceeded the 2014 Residential Soil RSL or RDEC for Residential Soil. One SVOC (butylbenzylphthalate) was detected in two sediment samples at concentrations below the 2014 Residential Soil RSL and RDEC. Arsenic was detected in multiple locations at concentrations above the Residential Soil RSL, but below the RDEC, and manganese was detected in sediment sample SD-008 at a concentration above the RDEC. AMEC concluded that the low-level VOCs, SVOCs and naturally occurring metals in sediment were not derived from former DoD activities and proposed no further testing of sediment in a Phase II site investigation.

In addition to collecting groundwater samples from inferred fracture zones in NIKE-1, NIKE-2, ROU-1, ROU-2, and ROU-3, AMEC collected groundwater samples from 13 temporary piezometers; two residential water supply wells located at 15 Theodore Foster Drive (identified as bedrock wells ST-1 and ST-2); and a shallow hand-dug well located at 23A Theodore Foster Drive (identified as RU-4). Aqueous samples were also collected from two cesspools in AOC-1 and the grease trap at the former Mess Hall building.

AMEC found that overburden groundwater collected from the temporary piezometers contained several VOCs, SVOCs and metals (total and dissolved). None of the VOCs exceeded the federal SDWA MCLs or RIDEM GA groundwater classification criteria used for reference, but some VOCs did exceed the 2014 Tap Water RSL. TCE was detected in all groundwater samples ranging from 0.8 to 4.5 µg/L,

including one overburden well located near the former Frequency Changer/Generator Building and within the four water supply wells as shown below:

- PZ-019 (0.9 J µg/L)
- NIKE-1 (2 µg/L)
- ROU-1 (1.4 µg/L)
- ROU-2 (3.5 to 4.5 µg/L)
- ROU-3 (0.8 J to 3.8 µg/L)

The one other VOC found in overburden groundwater samples was naphthalene estimated in a groundwater sampled collected from PZ-019 at a concentration of 0.3 J µg/L. AMEC concluded that naphthalene is likely associated with the former UST near the Frequency Changer/Generator Building. The three other detected VOCs (bromodichloromethane, chlorodibromomethane and chloroform) were only found in bedrock groundwater collected from water supply wells. AMEC concluded that all three VOCs appear to be related to the disinfecting of bedrock wells following the geophysical investigation. AMEC concluded that two SVOCs (bis-2-ethylhexyl-phthalate and pentachlorophenol) detected in groundwater samples are likely the result of laboratory contamination.

Unfiltered groundwater samples were found to contain inorganics. Antimony and lead exceeded the SDWA MCL in a groundwater sample collected from PZ-019, while arsenic, cobalt, manganese and thallium exceeded the Tap Water RSL or Rhode Island GA criteria. AMEC concluded that the antimony and lead may be related to former DoD activities, but the other four inorganics do not appear to be related to former DoD activities.

VOCs were not detected above laboratory reporting limits in groundwater samples collected from ST-1, ST-2, or ROU-4.

The cesspools and grease trap were previously investigated in the initial phase of the RI (AMEC, 2014b). The initial phase of the RI included surface water sampling from both cesspools ("Cesspool 1" and "Cesspool 2") and from the grease trap ("SW/SD-012") for testing of VOCs. Sediment samples were also collected from both cesspools and tested for VOCs, SVOCs, TAL metals, TOC, and grain size. Additionally, a groundwater well ("PZ/GW-010") was installed to investigate the cesspools and a groundwater sample collected from that well was tested for VOCs, SVOCs, and TAL metals. The results are presented in Tables 2-5, 2-7, 2-8, 2-11, and 3-1, AMEC, 2014b. AMEC did not identify COPCs exceeding screening levels, except arsenic that was reported in the sediment sample collected

from Cesspool 1 at the concentration (1.84 mg/kg) above the 2014 Residential Soil RSL and RDEC and manganese that was reported in a groundwater sample collected from PZ/GW-010 at a concentration (1.3 mg/L) above the 2014 Tap Water RSL. AMEC concluded that arsenic and manganese are unrelated to former DoD activities.

Supplemental Drum Investigation

Soils under three drums located east of AOC-1 were investigated. The three drums were found cut in half and thought to be used for baiting deer. AMEC collected and jarred soil samples from 0 to 1 foot at each location. Head space readings in each jar using a PID returned 0.0 parts per million (ppm) by volume. AMEC proposed no further investigation of these drums.

Three-Dimensional Groundwater Visualization

To assist in the conceptual model development, historic and current investigation results were incorporated into a three-dimensional data visualization model using TecPlot. Spatial data in the three-dimensional model included overburden, bedrock surface, fracture network, and COPC distribution in all media.

AMEC, 2014b. RI/FS Phase I Summary/Updated Conceptual Site Model/Data Gap Analysis & Phase II Work Plan Addendum.

This report updated the CSM based on data collected during the Phase I site investigation, identifies data gaps and presents a Phase II site investigation work plan. Several findings of the report pertinent to the CSM are summarized here.

- AOC-5 was used for residential and agricultural dumping by the property owner and is not attributable to former DoD activities; therefore, AOC-5 is ineligible for investigation and cleanup under DERP-FUDS. This conclusion is supported by two findings. First, an interview with Mr. Bob Steere on December 13, 2012 documented that the disposal was done by the property owner. Second, the soil vapor survey in October 2012 found no chlorinated VOCs in six soil vapor samples locations in and around AOC-5.

- The very low toluene detections in the Passive Soil Vapor Study may reflect ambient conditions from commercial/industrial products containing toluene.

- Phase I sampling data indicate that former DoD activities did not adversely affect pore water, surface water or sediment in local wetlands. Arsenic and manganese were detected above screening levels in these media but thought to be from natural weathering of minerals.

- A limited number of VOCs and SVOCs were detected in soil and groundwater. These analytes were detected at low concentrations, are limited to the Site or areas adjacent to the Site and are likely associated with former DoD activities.

- Two troughs were located in the concrete floor of the Frequency Changer/Generator Building, one with a grate and one with a chiseled drain to the subsurface. Borings SB-019 and SB-020 and piezometer PZ-019 were installed in the floor adjacent to these features.

- SVOCs were detected above laboratory reporting limits in surficial soils collected near the former 6,000-gallon diesel field UST (SB-008) and under the Former Frequency Changer/Generator Building (SB-019 and SB-020), but not in subsurface soils at these locations.

- AMEC concluded that TCE, pentachlorophenol, and naphthalene are the only Site-related COPCs identified in overburden groundwater and are isolated to the area surrounding PZ-019 located immediately adjacent to the former Frequency Changer/Generator Building.

- Transducer data in late May and early June of 2013 from NIKE-1 and ROU-1 show a hydraulic connection between the wells. This is supported by the fractures identified in the borehole geophysical logging. Transducer data and borehole geophysical data also indicate that water bearing fractures intersecting NIKE-2 are different from those that intercept NIKE-1 and ROU-1.

- Analytical data from bedrock wells under non-pumping conditions found that TCE was not detected in five depth intervals at NIKE-2 but was detected in 16 and 25 ft depth intervals sampled at NIKE-1, ROU-1, ROU-2, and ROU-3. This is evidence that NIKE-2 does not intercept the fractures that connect to the source of TCE at the Site or NIKE-2 is upgradient of the source.

- TCE distribution in NIKE-1, ROU-1, ROU-2, and ROU-3 is consistent with observations about the interconnectivity between wells based on hydraulic interference and geophysical logging. TCE was only detected in the deepest samples at ROU1 and NIKE-1. The detection of TCE in all discrete samples in ROU-2 and ROU-3 is consistent with the finding of a near vertical fracture in these wells during the geophysical logging.

- 1,1-DCE was detected in four soil samples near the former Radar Pads A and C and dry well, suggesting reductive dechlorination of TCE, however TCE was not detected in the same soil samples.

- Cis-1,2-DCE is consistently detected at ROU-2 which indicates degradation of a "mature" TCE plume at this location.

USAGC, 2015. Former Nike Control Area PR-79, Rhode Island, Historic Photographic Analysis

At the request of the CENAE, the USAGC completed a historic photographic analysis (HPA) of the Site and surrounding area. The purpose of the HPA was to identify features both spatially and temporally associated with previous activity, particularly former DoD activities. Specific tasks included: conducting archival research for historical documents regarding the use and purpose of the Site; and performing imagery acquisition, inspection, and rectification using historical photography, satellite imagery, and aerial photography covering the Site from 1951 to 2014. The HPA identified various AOCs where chemical usage was suspected or where disposal, burial, or land use change (e.g., ground scarring or the removal of vegetation) was identified.

USACE, May 2015 Well Development, Hydraulic Conductivity Assessment, and Sampling

In May 2015, the USACE self-performed well re-development at the existing 14 piezometers. Each piezometer was developed using a surge block and purged while collecting water quality parameters. The flow rates measured during development were recorded and used to estimate hydraulic conductivity and transmissivity values for the screened aquifer materials. Hydraulic conductivity values ranged from 3.5 x 10-6 to 1.7 x 10-4 centimeters per second, which is consistent with the silty sand material that comprises much of the overburden material.

USAPHC, June 2016. Groundwater Consultation, Former Nike PR79, US Army Public Health Center

In June 2016, the USAPHC collected groundwater samples from 14 temporary piezometers and six water supply wells located at and near the Site, including one off-Site water supply well in bedrock (ST-2) located about 1,000 ft north of the Property at 15 Theodore Foster Drive to estimate background groundwater conditions. High turbidity was noted at PZ-004, PZ-011, PZ-019, and PZ-022 that USAPHC contributed to possible silting within the wells. Samples were collected for analysis of VOCs. TCE was detected in an unfiltered water sample from ROU-2 at a concentration of 5.1 µg/L which is slightly above the SDWA MCL of 5 µg/L. TCE was estimated or detected at concentrations at or above laboratory reporting limits, but at concentrations below the SDWA MCL, in pre-filtration water samples collected from NIKE-1 (1.6 µg/L), NIKE-2 (0.7 µg/L), and ROU-3 (0.4 J µg/L). Cis-1,2-DCE was estimated at the laboratory reporting limit at ROU-2 (0.4 J µg/L). 1,1, -DCE was estimated below the laboratory reporting limit at PZ-005 (0.2 J µg/L). MTBE was detected in the background groundwater sample collected from ST-2 at a concentration of 1 µg/L. The source of MTBE in this sample was unknown; however, USAPHC concluded that it might have been the result of activity/operations in the area unrelated to the Site. TCE was not detected in overburden groundwater samples collected from the 14 piezometers, however there were detections of acetone, 2-butonone, and carbon disulfide at PZ-019. USAPHC acknowledged that the extent of the TCE plume is unknown.

USAGC, 2016. Integrated Fracture Trace Analysis, Former Nike PR-79 Control Area, Foster, Rhode Island

USAGC completed a fracture trace analysis of the Site and surrounding area. The objective of this investigation was to identify bedrock fracture orientation and density in the Site vicinity using a combination of remote sensing, field mapping, and data collection techniques.

The overburden was described based on borehole geophysical data as 0 to 20 ft thickness of glacial till with increasing thickness to the south (within one mile) to a maximum of 60 to 100 ft. The bedrock geology was described as the South Foster migmatite (metamorphic, quartz biotite schist) intruded by the Ponaganset quartz diorite gneiss. Glacial float was found throughout the study area requiring careful identification of in situ bedrock outcrops for examination.

Suspected fracture traces identified using remote sensing were reviewed within a 3-mile radius of the Site to identify a total of 1,057 fracture traces and then were verified in the field by collecting fracture measurements at 39 bedrock outcrop locations. Mapping at 39 bedrock outcrops included strike and dip measurements and observations such as fracture length, openness, roughness, density and spacing. Over 1,000 fracture measurements were collected at 39 outcrops. In addition, optical and ATV data from water supply wells NIKE-1, NIKE-2, ROU-1, ROU-2, and ROU-3 were reinterpreted to identify non-transmissive and transmissive fracture data sets. The remote-sensing data, outcrop-scale data, and borehole geophysical data were analyzed and compared using industry standard techniques.

USAGC concluded that the orientation of fractures observed at outcrops and identified in fracture domain analysis provide evidence that a fracture zone could connect supply wells NIKE-1 and ROU-1. A total of 135 transmissive fractures were identified among the five water supply wells, and the study further concluded that bedrock fractures are likely conduits for TCE migration from source to downgradient receptors. Additionally, it was found the joint orientation shifted from north to south depending on the bedrock type.

Hager Geoscience Inc. (HGI), 2017. RE: Seismic Refraction Investigation and Data Re-Interpretation, PR-79 NIKE Control Area, Foster, Rhode Island.

HGI employed several approaches to improve the resolution of existing overburden and bedrock geologic models, and to locate low velocity fracture zones. Previous seismic data collected by ANL in 2002 was re-assessed using new software processing techniques to improve resolution of the interpreted sections. HGI performed one vertical seismic profiling survey, five ground penetrating radar (GPR) profiles and an outcrop velocity test of exposed rock were completed to locate new boreholes in interpreted fracture zones.

HGI confirmed the predominant north-south trend of fracture traces observed on-Site. Large low-velocity anomalies indicating fracture zones were identified along existing and new geophysical lines ANL Line 1, ANL Line 2 and HGI Line 100. Multiple cross sections were presented showing the interpreted overburden type (dry sandy soil, saturated gravel soils, and fractured rock/compact till), water table elevation and bedrock fracture density (i.e., intact, fractured, heavily fractured). Twelve borehole locations were recommended for future investigations to confirm low velocity zones identified along seismic and GPR lines. Three of these locations were proposed to see if a north-south low velocity zone (approximately 100 ft west of ROU-1) seen on ANL Line 1, ANL Line 2 and HGI Line 100 represents a continuous fracture zone.

HGI corrected fracture strike and elevation data in ANL's 2002 seismic refraction study and fracture strike and dip orientations in USAGC's 2016 integrated fracture trace analysis.

Hager-Richter Geoscience, Inc. (Hager-Richter), 2017. RE: Geophysical Evaluation, Former NIKE PR-79 Control Area, Foster, Rhode Island.

Hager-Richter reviewed the geophysical methods and techniques previously used in acquiring geophysical data and determined if those methods and techniques are technically sufficient for the development of an RI/FS Work Plan.

Hager-Richer reviewed USAGC's 2016 integrated fracture trace analysis and recommended that additional work focus on local/borehole transmissive fracture interaction for development of a conceptual fracture model that can be used to effectively guide the selection of receptor wells for downgradient well-monitoring.

Hager-Richer reviewed HGI's seismic refraction investigation and data reinterpretation and agreed with HGI's recommendation that the low-velocity zones must be ground-truthed by other methods to confirm the applicability of the methodology for future use at the Site.

Hager-Richer reviewed GAI's borehole geophysical logs and made corrections to fracture strike and dip orientations.

Hager-Richter concluded the following:

- Bedrock fracture orientations are generally consistent from a regional scale to borehole scale in the study area with dominant populations of northwest-north by northwest and north by northeast striking fractures and minor populations of north and east by northeast striking fractures.

- Possible bedrock fracture zones identified based on surface geophysical data generally conform to the orientations identified in the fracture trace analysis.

- The geophysical logs are dominated by discrete bedrock fractures and small bedrock fracture zones that range in aperture from a few inches to as much as two ft in the open bedrock portions of the five logged boreholes and such features are not likely to be detected by surface geophysical methods.

- Confirmation of bedrock fracture zones identified on the basis of a surface geophysical method is required prior to additional use of the method as a tool in identifying such zones.

- Hydraulically transmissive bedrock fractures primarily dip moderately to the northeast and southeast and secondarily to the northwest and southwest.

- Additional borehole geophysical logging data, namely cross-borehole flow testing, should be acquired in the available boreholes at the Site to attempt to determine direct and indirect fracture connectivity between boreholes.

The Johnson Company, 2018. Client Draft Remedial Investigation/Feasibility Study Work Plan, Former NIKE PR-79 Control Area, Foster, Rhode Island.

This Draft RI/FS Work Plan provides a comprehensive review of Site background history and investigations; identifies data gaps; and proposes an RI/FS field investigation approach.

Alliance Environmental Group (AEG), 2019. UST Closure Assessment Report, Town of Foster School Department Property, 23 Theodore Foster Road, Foster, Rhode Island.

A 2,000 gallon No. 2 fuel oil UST removal was performed under direction of RIDEM at the former Mess Hall Building on September 3, 2019.

- Oversight of the UST closure activities was conducted on September 3, 2019. F.W. Webb/AEG documented excavation conditions following UST removal and provided oversight for impacted soils removal activities. Representative composite soil samples from the sidewalls of the excavation, beneath the UST, and beneath the UST piping were screened in the field for total VOCs using a PID.

- Suspected fuel oil-impacted soil was encountered in the southern portion of the excavation and was attributed to a corroded hole observed in the base of the UST.

- Eight confirmatory soil samples from the sidewalls, endwalls, and base of the UST excavation were analyzed at a Rhode Island-certified laboratory for total petroleum hydrocarbons (TPH) by USEPA Method 8260. Samples of the stockpiled soil were also analyzed for applicable disposal criteria. Two of the eight confirmatory soil samples analyzed had TPH concentrations detected above the laboratory reporting limits. The highest concentration was 358 mg/kg from a sample on the sidewall located below a chimney that could not be further excavated due to structural concerns. Both TPH detections were below the RIDEM TPH standard of 500 mg/kg for soil in GA groundwater classification areas.

- On October 9, 2019, 18.46 tons of stockpiled No. 2 fuel oil-impacted soil was transported by Western Oil, Inc. to Rhode Island Resource Recovery Corporation in Johnston, RI for recycling and reuse.

- The report concluded that the UST was evaluated in accordance with the RI UST Regulations and the Rules of Regulations for the Investigation and Remediation of Hazardous Materials Releases. The report concluded that TPH detections were below the RIDEM TPH soil and leachability standards for GA groundwater classification areas; and therefore, no further action is required.

The former mess hall building, which is the only building on the Former NIKE PR-79 Control Area property, was temporarily declared "unsafe" in July 2020 by the Town of Foster Building Inspector due to potential asbestos containing building materials. The Town of Foster is considering demolishing that building and future use of the property as a solar array

10.9 Receptors and Exposure Pathways

The human health and ecological receptors and potentially complete exposure pathways under current and reasonably anticipated future land use scenarios to be considered for the Site are summarized below. The Former NIKE PR-79 Control Area FUDS property is currently zoned as "municipal" and the current land use is non-residential (i.e., used for municipal administration). The area to the south of the FUDS property is currently residential use. As discussed in Section 10.7, the DoD is implementing an interim remedy to treat residential drinking water wells, in which Site-related VOCs have been identified, in this off-Property residential area. Future use of the FUDS property and the surrounding area is anticipated to remain consistent with current use. However, due to the residential areas located in proximity to the FUDS property, residential and recreational use of the FUDS property are considered reasonable future use scenarios. Therefore, an unlimited use and unrestricted exposure (UU/UE) scenario will be evaluated to provide information for making risk-management decisions.

Human Health

Current/future human receptors at the Site are as follows:

- Current/Future On-Property Trespasser (Adolescent);
- Current/Future On-Property Commercial/Industrial Worker;
- Current/Future On-Property Construction/Utility Worker;
- Current/Future Off-Property Resident (Adult/Child);
- Future Recreational User (Adult/Child); and
- Hypothetical Future On-Property Resident (Adult/Child).

The potentially complete exposure scenarios for the above receptors and Site media are as follows:

- Exposure to surface soil may occur by current/future trespassers and on-Property commercial/industrial workers, future recreational users, and hypothetical future on-Property residents. Surface soil exposure pathways include incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles.
- Exposure to the combined surface and subsurface soil interval (to a maximum depth of 10 ft) may occur by current/future on-Property construction/utility workers and by future on-Property trespassers, future on-Property commercial/industrial workers, future recreational users, and hypothetical future on-Property residents, assuming soils become mixed during potential future redevelopment activities. Soil exposure pathways include incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles.
- Exposure to shallow groundwater may occur by:
    - current/future on-Property construction/utility workers via incidental ingestion, dermal contact, and inhalation of volatiles in an excavation trench.
    - current/future on-Property commercial/industrial workers via inhalation of indoor air within the current on-Property building and hypothetical on-Property buildings that may be constructed in the future (i.e., potential vapor intrusion pathway).

- current off-Property residents and hypothetical future on-Property residents via inhalation of volatiles in indoor air (i.e., potential vapor intrusion pathway).

- Exposure to groundwater (from on-Property monitoring wells [overburden and bedrock], piezometers, and water supply wells [using groundwater collected prior to carbon filtration]) may occur by:

  - future on-Property commercial/industrial workers via ingestion of drinking water. (This exposure pathway is not complete under a current scenario due to the presence of a carbon filtration system on existing on-Property water supply well NIKE-1 and NIKE-2 is inactive. Evaluation of the future scenario will also provide information on a hypothetical scenario in which the carbon filtration system on NIKE-1 fails)

  - current/future off-Property residents and hypothetical future on-Property residents via ingestion of drinking water, dermal contact and inhalation while bathing/showering. (This exposure pathway is not complete under a current scenario due to the presence of a carbon filtration system on existing water supply wells in closest proximity to the Property. Off-Property residential water supply wells located further away from the Property ("DW-wells") do not all have carbon filtration systems installed. These wells will not be evaluated within the scope of the risk assessment. However, recommendations for further assessment of these wells during a later RI phase may be made following assessment of groundwater results from the on-Property area and adjacent off-Property area.)

- Exposure to sediment and surface water in water bodies potentially impacted by Site groundwater may occur by future recreational users via incidental ingestion (sediment only) and dermal contact (sediment and surface water) while wading.

Ecological

The undeveloped, wooded portions of the Site are expected to provide habitat for ecological receptors such as plants, soil invertebrates, small birds and mammals, and reptiles and amphibians. These receptors may be directly exposed to constituents released to the surface soil (e.g., terrestrial plants, earthworms) or via ingestion of impacted food items (e.g., birds or mammals consuming impacted earthworms).

Ecological receptors are typically not directly exposed to groundwater. However, exposure to constituents present in groundwater may occur when groundwater discharges into a water body such as Winsor Brook, or contributes to seeps and the forested swamp located to the south of the Property.

Therefore, aquatic receptors such as invertebrates, fish, or amphibians may be directly exposed to constituents in the water column and benthic (sediment-dwelling) invertebrates may be directly exposed to constituents in the sediment or porewater (as groundwater discharges through the sediment into the water body). Birds and mammals may be exposed to constituents in water bodies through the incidental ingestion of sediment, ingestion of drinking water, or ingestion of impacted food items.

Therefore, the ecological exposure scenarios most likely to be complete at the Site include the following:

- Direct contact with surface soil by terrestrial plants and invertebrates;
- Incidental ingestion of surface soil and ingestion of impacted food items by terrestrial birds and mammals in terrestrial areas which provide habitat for wildlife;
- Direct contact with sediment by benthic/wetland invertebrates in water bodies potentially impacted by Site groundwater;
- Direct contact with pore water (i.e., Site groundwater discharging into water bodies) by benthic invertebrates;
- Direct contact of surface water by amphibians, aquatic invertebrates and/or fish in water bodies potentially impacted by Site groundwater; and,
- Ingestion of sediment, surface water, and impacted food items by semi-aquatic birds and mammals foraging in water bodies potentially impacted by Site groundwater.

QAPP Worksheet #11: Project/Data Quality Objectives

The planned field investigation is designed to amend the existing Site dataset and determine the presence and/or absence of COPCs associated with former DoD activities in Site media using a biased sampling approach to identify potential source areas. The biased sampling results will be evaluated in comparison to risk-based human health and ecological screening levels and Site-specific background levels to determine whether COPCs are present in Site media, for which exposure-based, unbiased sampling should be performed in a second RI phase for the purposes of evaluation in a human health and ecological risk assessment. In general, the field investigation will refine the existing working CSM and resolve data gaps for the four identified AOCs. DQOs are developed in this worksheet based on the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G4, EPA/240/B-6/001) (USEPA, 2006) and are presented below.

11.1 Problem Statement

The project team reviewed details from historical investigations and the existing dataset to identify data gaps at each AOC. The evaluation determined the existing CSM inadequately characterizes the hydraulic connection between the overburden and bedrock aquifers, nature and extent of potential impacts, and the fate and transport of Site-related constituents to potential on-Property and off-Property human and ecological receptors. Additional sampling and investigation are required to refine the preliminary CSM, determine the presence and/or absence of COPCs associated with former DoD activities in Site media, and establish a representative local background dataset.

11.2 Goals of the Study

The project team will collect surface soil, subsurface soil, groundwater, surface water, pore water, and sediment samples to establish datasets to refine the preliminary CSM and evaluate whether Site-related constituents are present at concentrations greater than risk-based human health and ecological screening levels and Site-specific background levels. Soil sampling will be performed at discrete locations to supplement the existing dataset from within the existing AOC boundaries. Sampling locations for groundwater, pore water, surface water, and sediment will be established to investigate sources and end points to determine groundwater flow paths, migration pathways and the interaction of groundwater, pore water, and surface water.

Parameters selected for analysis are based on:

a) parameters that exceed the selected screening criteria during the preliminary screening assessment (The Johnson Company, 2018);

b) COPCs identified in the *Final Report NIKE Missile Battery, Environmental Conditions Assessment Guide* (USACE-EMCX, 2003);

c) data needs for performing a comparison to risk-based human health and ecological screening levels protective of current and reasonably anticipated future land use scenarios;

d) data needs for establishing a Site-specific background dataset; and,

e) data needs to support the FS (e.g., geochemical and MNA parameters).

To achieve these overall project objectives, the following specific DQOs have been established:

- **Goal 1** – Collect a dataset of potentially impacted surface and subsurface soil associated with each individual AOC. Using this data, refine the preliminary CSM and determine whether constituents associated with former DoD activities are present in soil at concentrations greater than risk-based human health and ecological soil screening levels protective of current and reasonably anticipated future land use scenarios and Site-specific background levels.

- **Goal 2** – Establish a dataset of surface water, pore water, and sediment samples from drainage features, seeps, streams, and/or other surface water features in potentially impacted downgradient surface water as identified in a *Wetland and Waters of the US Delineation Report* (Woodard & Curran, 2019). Using this data, refine the preliminary CSM and determine whether constituents associated with former DoD activities are present in surface water, pore water, and/or sediment at concentrations greater than risk-based human health and ecological screening levels protective of current and reasonably anticipated future land use scenarios and Site-specific background levels.

- **Goal 3** – Establish a groundwater monitoring well network within the overburden, weathered bedrock, and bedrock units to assess potential continuing sources of COPCs and collect groundwater samples on a Site-wide scale to determine:

    - The distribution of COPCs (nature and extent),

    - Define source area(s) (discrete or diffuse sources),

    - the fate and transport of COPCs (migration to potential exposure points); and,

    - determine whether COPCs are present in groundwater at concentrations greater than risk-based human health screening levels protective of current and reasonably anticipated future land use scenarios and concentrations in upgradient groundwater wells.

- **Goal 4** – Establish a representative local background dataset based on Site conditions beyond the limits of the AOCs and calculate background threshold values (BTVs), as described in Section 11.5 (Goal 4).

11.3 Information Inputs to Resolve the Problem

Information inputs used to develop this QAPP consist of prior study reports, available historical data, consideration of potential human health and ecological exposure pathways and receptors, and consideration of potential data needs to support the FS. Refer to **Worksheet #10** for a summary of prior investigations and reports. Additional information inputs that will be generated from the RI field investigation described in this QAPP will include the following items:

- Field observations and measurements, including geologic logs, ambient air (e.g., PID) measurements, water quality parameters, photographs, GPS coordinates, and survey data.
- Surface soil samples (0 to 2 ft bgs).
- Subsurface soil samples (collected no deeper than 10 ft bgs, the maximum depth for potential contact by human receptors; however, potentially deeper soil samples will be collected based on field observations (i.e. PID readings) for nature and extent purposes).
- Background surface (0-2 ft bgs) and subsurface (2-10 ft bgs) soil samples for derivation of soil BTVs. An unbiased sampling approach will be used for collection of subsurface soil samples by randomly selecting soil depths between 2-10 ft bgs using Pacific Northwest National Laboratory Visual Sample Plan software.
- Groundwater samples from existing piezometers and newly installed permanent monitoring wells.
- Surface water, pore water, and sediment samples from drainage features, seeps, and streams.
- Chemical and physical data (e.g., Flexible Liner Underground Technologies [FLUTe] transmissivity profile, etc.)

11.4 Define the Study Boundaries

The Property is located at the southern end of Theodore Foster Drive in Foster, Rhode Island as shown in **Figure 10-1**. The Site is the area where contamination associated with the Property has come to be located. General spatial and temporal boundaries are described below.

Spatial Boundaries

A total of four AOCs (AOC-1, AOC-2, AOC-3, and AOC-4) previously identified at the Site comprise the RI field investigation spatial boundaries. Three AOCs (AOC-1, AOC-2, and AOC-3) are within close proximity to each other on top of Oak Hill. One AOC (AOC-4) is located further west. Currently, parts of the Site are overgrown and brush clearing will be necessary. Portions of the Site are forested with natural obstructions (e.g., mature trees, boulders, etc.) that may require sample locations to be shifted due to access restrictions. The spatial boundaries also include Winsor Brook and the other streams and wetlands surrounding the Property where surface water, pore water, and sediment samples will be collected, as well as the Roukus property where a bedrock will be installed near water supply well ROU-1. Two background study areas are also within the spatial bounds of the Property. One is located on the easternmost portion of the Property and the second is location along Theodore Foster Drive. Each background study area is 0.5 acres to represent the typical size of a residential lot.

Temporal Boundaries

The temporal boundaries for this study will be the RI field investigation, anticipated to begin after March 2020 and end during September 2020.

11.5 Analytical Approach

The analytical approach for the planned RI field investigation includes specific "if… then…" statements to expand upon the study goals established earlier in this worksheet. These decision statements will guide the sampling design and will be continually referenced by the PDT in evaluating the results of the investigation and proceeding with risk-based decisions.

Goal 1 – Collect a dataset of potentially impacted surface and subsurface soil associated with each individual AOC. Using this data, refine the preliminary CSM and evaluate whether COPCs are present in surface and/or subsurface soil at concentrations greater than risk-based human health and ecological screening levels protective of current and reasonably anticipated future land use scenarios or Site-specific background levels.

- For each AOC, select soil boring locations based on the presence of historic borings and identified data gaps to determine the distribution of COPCs (nature and extent), define source area(s) (discrete or diffuse sources), and evaluate fate and transport of COPCs.

- Collect a soil dataset for performing a comparison to risk-based human health and ecological screening levels protective of current and reasonably anticipated future use scenarios and Site-

specific background levels. The approach for the screening-level comparison that will be performed is further described in **Appendix G**.

- Collect surface soil samples from a depth of 0 to 2 ft bgs and collect subsurface soil samples no deeper than 10 ft bgs to represent the interval to which human receptors have the potential to be exposed.

- Collect additional soil samples or deeper samples based on real-time field observations (e.g., PID detection, staining, odor, etc.) as specified in **Worksheet #14** and **Worksheet #17** to assess the nature and extent of constituents in soil. Specific analytes include VOCs, SVOCs, TAL metals[1], potential of hydrogen (pH), TOC, and grain size. Select samples will be analyzed for SPLP metals.

- If unanticipated areas of limited accessibility are encountered during the RI field investigation (either horizontally or vertically), then boring locations and sampling intervals will be adjusted as necessary to ensure collection of the minimum number of samples needed for statistical analysis at sampling locations representative for each AOC.

Soil results from the first phase of the RI will be used to determine the presence and/or absence of analytes associated with former DoD activities using a biased sampling approach to identify potential source areas. It is understood that this biased sampling design does not provide representative exposure-based samples for statistical analysis as necessary to perform a Site-specific risk assessment. However, in understanding the limitations of that dataset, the biased sampling results will be evaluated in comparison to risk-based human health and ecological screening levels and soil BTVs to determine which chemicals, if any, exceed the risk-based soil screening levels and soil BTVs, and in which areas. If biased soil sample results exceed risk-based screening levels and BTVs, then a second RI phase will be designed with an unbiased and exposure-based sampling approach and decision units and exposure units to be evaluated in a Site-specific risk assessment. If biased sample results do not exceed risk-based screening levels or BTVs, then no further risk assessment will be performed. **Appendix G** provides a risk assessment work plan, which describes the approach for comparison to risk-based human health and ecological screening levels and BTVs in the Phase I RI and for conducting further risk assessment in the second phase of the RI, if necessary. **Goal 2** – Establish a dataset of surface water, pore water, and sediment samples from drainage features, seeps, streams, and/or other surface water features in potentially impacted downgradient surface

[1] Since there is no historical evidence that chromium was specifically related to a particular process or material in past DoD operations at this Site, total chromium analysis is proposed at this phase of the investigation. If Site total chromium results exceed background total chromium results, hexavalent chromium analysis may be performed in the second phase of RI, if applicable.

water as identified in a *Wetland and Waters of the US Delineation Report* (Woodard & Curran, 2019). Using this data, refine the preliminary CSM and evaluate whether COPCs are present in sediment, surface water, and/ or pore water at concentrations greater than human health or ecological screening levels protective of current and reasonably anticipated future land use scenarios or Site-specific background levels.

- Select surface water, pore water, and sediment samples based on historic and recent identification of nearby, downgradient wetlands, seeps, and surface water features for the purposes of performing a comparison to risk-based human health and ecological screening levels protective of current and reasonably anticipated future use scenarios and Site-specific background levels. The approach for the screening-level comparison that will be performed is further described in **Appendix G**.

- Locations will consist of adjacent and downstream drainage channels, streams, seeps, or wetlands that are in the vicinity of the Property as identified in a *Wetland and Waters of the US Delineation Report* (Woodard & Curran, 2019). If insufficient water is in the drainage features, seeps, streams, and/or other surface water features, then a substitute location will be selected as close as possible to the originally proposed sample. If no representative location can be identified, then no sample will be collected.

- Collect sediment samples from a depth of 0-1 ft bgs as specified in **Worksheet #17**. Specific analyses include VOCs, SVOCs, TAL metals, AVS-SEM, pH, TOC, and grain size.

- Collect pore water samples from a depth of 0-1 ft bgs as specified in **Worksheet #17**. Specific analyses include VOCs, SVOCs, TAL metals, and hardness.

- Collect surface water samples from the midpoint of the water column at the specific sampling point as specified in **Worksheet #17**. Specified analyses include VOCs, SVOCs, TAL metals, and hardness.

Goal 3 – Establish a groundwater monitoring well network within the overburden, weathered bedrock, and bedrock units to assess potential continuing sources of COPCs and collect groundwater samples on a Property-wide scale to determine:

- the distribution of COPCs (nature and extent),

- define the source area(s) (discrete or diffuse sources),

- the fate and transport of COPCs (migration to exposure points); and,

- whether COPCs are detected in Site groundwater at concentrations above risk-based human health screening levels protective of current and reasonably anticipated future land use scenarios and concentrations in the upgradient groundwater well. The sources of the groundwater screening levels that will be used in this comparison are described in **Appendix G**.

- Complete nine soil boring locations as overburden or weathered bedrock monitoring wells to:

  - identify sources of groundwater impacts which are bound to overburden material,

  - determine the interaction between the overburden and weathered bedrock groundwater; and,

  - establish a monitoring well network to assess the downgradient extent of overburden groundwater impacts.

- Complete five soil boring locations as bedrock monitoring wells to

  - determine the magnitude of bedrock groundwater impacts immediately beneath and/or adjacent to the historic DoD activities that resulted in release to the environment,

  - determine the interaction between the weathered bedrock and bedrock groundwater; and,

  - create a Property-wide bedrock monitoring well network to assess horizontal distribution and vertical transport of COPCs in bedrock groundwater.

- Construct all new overburden monitoring wells with the appropriate total well depth and screen interval to enable well development and low-flow groundwater sampling per USEPA guidelines (USEPA, 2017). Bedrock monitoring wells will be assessed and constructed with FLUTe sampling manifolds.

- Collect groundwater samples from the midpoint of the screen or designated sample port for VOCs, SVOCs, TAL metals (filtered and unfiltered), and a suite of MNA parameters (TOC, ferrous iron, chloride, sulfate/sulfide, nitrates/nitrites, alkalinity, and methane/ethane/ethene).

- Abandon an unidentified pipe and existing temporary piezometers which have been previously identified to have construction and long-term usability issues (i.e., PZ-002, PZ-003, PZ-004, PZ-006, PZ-009, PZ-011, PZ-015, PZ-019, PZ-022). The remaining five piezometers (PZ-001, PZ-005, PZ-007, PZ-010, PZ-014) will be completed as permanent monitoring wells, re-

developed, and sampled as part of the monitoring well network. The rationale for piezometer abandonment is provided in **Worksheet #14**.

Goal 4 – Determine the regional anthropogenic background COPC concentrations representative of areas beyond the limits of the AOCs.

- Collect surface soil, subsurface soil, groundwater, surface water, pore water, and sediment samples from pre-determined background areas representative of the immediate area, but not impacted by historic DoD activities at the Property.

- Medium-specific BTVs will be derived for constituents detected in Site surface soil, subsurface soil, surface water, pore water, and sediment at concentrations greater than risk-based human health and ecological screening levels. Media-specific constituents with a maximum detected concentration less than or equal to the associated BTV will not be identified as Site-related COPCs and further risk assessment will not be performed for these constituents. BTVs will be calculated in USEPA's ProUCL software (USEPA, 2016) as the 95 percent (%) upper tolerance limit (95UTL). The 95UTL statistic will be selected based on the distribution of the raw dataset (e.g., if the detected concentrations follow a normal, lognormal, or gamma distribution, then the normal, lognormal, or gamma 95UTL will be selected, respectively). In cases of no discernible distribution, the nonparametric 95UTL statistic will be selected. If the dataset includes non-detects, the Kaplan-Meier BTV statistics will be selected on the basis of the distribution of the detected concentrations. Prior to calculation of BTVs, the media-specific background datasets will be evaluated for the presence of outliers using the default outlier test in USEPA's ProUCL software (USEPA, 2016). Outliers identified will be eliminated from the background dataset, as appropriate.
- Groundwater results from the upgradient (background) groundwater well will be utilized in a direct comparison with Site groundwater concentrations to determine if Site groundwater concentrations are consistent or elevated compared with upgradient groundwater. Constituents with a maximum detected concentration less than or equal to the associated BTV will not be identified as Site-related COPCs and further risk assessment will not be performed for these constituents.

11.6 Performance Criteria

The selection of performance criteria is based on potential sources of study error (i.e., field error, analytical error), methods that will be applied to reduce the potential sources of error, and an approach on how team decisions will be managed relative to potential occurrences of error.

Sources of Error

For this field program, sources of error consist of two main categories: sampling errors and measurement errors. A sampling error occurs when the sampling design, planning, and implementation do not provide for a representative range of heterogeneity at the Site. For this RI, the use of FLUTe liners has the potential to introduce additional sample error through leaching. Researchers from the Pacific Northwest National Laboratory conducted eight week leaching tests on behalf of the DoE and documented that the use of FLUTe liners has the potential to leach trace levels of toluene and benzene from the FLUTe liner systems (T.J. Gilmore et al., 2004). Trace levels of toluene have been documented to leach from the urethane coating applied to the FLUTe liner fabric and trace levels of benzene from the inner nylon-11 tubing. As a result, it is not uncommon to see low concentrations of toluene and benzene in samples collected from FLUTe liner MLS ports. Typical concentrations of toluene observed in ambient groundwater range from 10 to 60 µg/L but may be as high as 280 µg/L. Benzene from the nylon-11 tubing peaked at 1.37 µg/L and was typically at 1 µg/L or less. These concentrations are below the toluene SDWA MCL of 1,000 µg/L and benzene SDWA MCL of 5 µg/L. The DoE study showed that the concentrations peaked after two weeks and then started to decrease over time. A measurement error occurs when there is a performance variance from laboratory instrumentation, analytical methods, and/or operator error. EPA identifies the combination of these errors as a "total study error" (EPA, 2006). For this RI field investigation, the team has prepared this QAPP to reduce the potential for total study error by documenting the DQOs, decision strategy, sampling design, analytical requirements, and other details, all of which provide team alignment with the study objectives and goals. Despite the relatively minor possibility that a measurable influence will be observed, the team will monitor and document the toluene and benzene concentrations in the samples collected from the FLUTe liner MLS ports.

Managing Decision Error

This RI field investigation will use decision-error minimization techniques in sampling design, sampling methodologies, and laboratory measurement of COPCs. Possible decision errors will be minimized during the RI field investigation by using the following methods:

- Use standard field sampling methodologies (as discussed in **Worksheets #14, #18, and #21**).
- Use applicable analytical methods and standard operating procedures (SOPs) for sample analysis by a competent analytical laboratory having state-appropriate National Environmental Laboratory Accreditation Program (NELAP) accreditation and be accredited through the DoD Environmental Laboratory Accreditation Program (ELAP).

- Validate analytical data to identify and control potential laboratory error and sampling error by using matrix spikes (MS), blanks, and duplicate samples.

Decision Error

Decision errors associated with judgmental sampling are based on sample design and measurement errors. Assuming that the best possible professional judgment was used to develop the judgmental sampling plan (e.g., selection of sampling locations, depths, and analytical parameters), remaining decisions and opportunities to mitigate potential errors will be associated with field decisions on refined sampling locations and depths, managing insufficient groundwater yields or quality, managing and packaging analytical samples, managing analytical results through the data validation process, and derivation of BTVs. To allow for derivation of BTVs using USEPA's ProUCL software, as discussed in Section 11.5, 10 background soil borings will be placed within a 0.5 acre area in each of the two background study areas. Each background soil boring will consist of one surface (0-2 ft bgs) and one subsurface soil sample collected between 2-10 ft bgs that is selected at subsurface depths randomly using Pacific Northwest National Laboratory Visual Sample Plan software. Locations and vertical intervals are randomly chosen using the VSP random number generator. Statistical analysis associated with the derivation of exposure point concentrations (EPCs) and hypothesis test background evaluation are not within the scope of the data evaluation based on the biased sampling dataset being collected in this phase of the RI. If biased sample results exceed risk-based screening levels and BTVs, then a second RI phase will be designed with an unbiased and exposure-based sample approach and decision units to be evaluated in a Site-specific risk assessment. If a second RI sampling event is determined to be necessary, the Phase I dataset (i.e., variability) will inform the sampling design to be used in the second RI phase such that these data are appropriate for use in deriving estimates of the mean concentration (i.e., 95% upper confidence limit (UCL)) within an exposure area and in hypothesis tests that compare Site and background data with specified Type I and Type II error rates.

Analytical data will be considered acceptable if they meet the appropriate data validation criteria presented in **Worksheet #34, 35, and 36**.

11.7 Sampling Design

This RI field investigation was designed to assess the distribution of impacts, identify whether a discrete or diffuse continuing source exists, and provide additional information to characterize presence and/or absence of COPCs associated with potential releases from historic DoD activities detailed in Section 10.6. The objective of the first phase of the RI is to determine the presence and/or absence of analytes associated with former DoD activities using a biased sampling approach to

identify potential source areas. It is understood that this biased sampling design does not provide representative exposure-based samples for statistical analysis as necessary for evaluation in a risk assessment. However, in understanding the limitations of that dataset, the biased dataset will be evaluated in comparison to risk-based human health and ecological screening levels and BTVs to determine which chemicals, if any, exceed the risk-based screening levels and BTVs, and in which areas. If biased sample results exceed risk-based screening levels and BTVs, then a second RI phase will be designed with an unbiased and exposure-based sampling approach and decision units and exposure units to be further evaluated in a risk assessment (in accordance with the work plan presented in **Appendix G**. If biased sample results do not exceed risk-based screening levels or BTVs, then no further risk assessment will be performed.

The critical objective is to obtain a dataset of known quality and sufficient sensitivity to meet DQOs. The laboratory selected for the RI field investigation (Katahdin Analytical Services, LLC) is expected to achieve limits of quantitation (LOQs) low enough to measure constituents at concentrations less than the PALs except where identified in **Worksheet #15.** Additional details on the sampling design and the exact media specific sampling details and procedures are discussed further in **Worksheet #17**.

QAPP Worksheet #12: Measurement Performance Criteria

Matrix: Groundwater, Surface Water, Pore Water, Surface Soil, Subsurface Soil, Sediment
Analytical Group or Method: VOCs, SVOCs
Concentration Level: Full Scan and SIM

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Overall Precision	Matrix Spike / Matrix Spike Duplicates	See WS 28
Overall accuracy/bias (contamination)	Trip Blanks (VOCs Only) / Equipment Blanks	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. No common contaminants detected > LOQ.
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LOD and LOQ Verification	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Groundwater, Surface Water, Pore Water, Surface Soil, Subsurface Soil, Sediment
Analytical Group or Method: Metals (total), Metals (dissolved), SPLP Metals
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Overall Precision	Matrix Spike / Matrix Spike Duplicates	See WS 28
Overall accuracy/bias (contamination)	Equipment Blanks	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. No common contaminants detected > LOQ.
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LOD and LOQ Verification	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Groundwater
Analytical Group or Method: Dissolved Gases
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Overall Precision	Matrix Spike / Matrix Spike Duplicates	See WS 28
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LO D and LOQ Verification	Per DoD QSM **and WS #24**
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Surface Soil, Sediment
Analytical Group or Method: pH
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	pH ± 0.5 S.U.
Laboratory Precision	Laboratory Duplicate	See WS 28
Completeness	As shown under Verification on WS 34	See WS 37

Matrix: Groundwater, Surface Water, Surface Soil, Sediment
Analytical Group or Method: Total Organic Carbon
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Laboratory Precision	Laboratory Quadruplicate (Solid), Laboratory Duplicate (Water)	See WS 28
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	MDL Study	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Groundwater
Analytical Group or Method: Anions
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Laboratory Precision	Laboratory Duplicate	See WS 28
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LOD and LOQ Verification	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Groundwater
Analytical Group or Method: Sulfide
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Laboratory Precision	Laboratory Duplicate	See WS 28
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LOD and LOQ Verification	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Surface Water
Analytical Group or Method: Alkalinity
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Laboratory Precision	Laboratory Duplicate	See WS 28
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LOD and LOQ Verification	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Sediment
Analytical Group or Method: AVS/SEM
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for aqueous and RPD ≤ 50% for solid when detects are at least 5x LOQ or within ±2x LOQ for results <5x LOQ
Overall accuracy/bias (matrix)	Matrix Spikes	See WS 28
Analytical Precision	Laboratory Duplicate	See WS 28
Analytical Accuracy/Bias	Laboratory Control Samples	See WS 28
Sensitivity	LOD and LOQ Verification	Per DoD QSM and WS #24
Completeness	As shown under Verification on WS 34	See WS 37

QSM – Quality Systems Manual

Matrix: Surface Soil, Sediment
Analytical Group or Method: Grain Size
Concentration Level: Not Applicable

Data Quality Indicator (DQI)	**QC sample or measurement performance activity**	**Measurement Performance Criteria**
Completeness	As shown under Verification on WS 34	See WS 37

QAPP Worksheets #14 /16: Project Tasks & Schedule

The following project tasks will be performed as part of the RI:

- Field Investigation Activities
- Sample Analysis and Reporting
- Data Management, Review, and Validation
- Report Preparation

The RI field activities will be executed over two field mobilizations. The project tasks and subtasks are described in the schedule and text below. The rationale for the specific sampling design and approach is presented in **Worksheet #17**.

14.1 Field Investigation Tasks

The following subsections present the specific field tasks to be completed as part of the field investigation. These activities include preparation of planning documents, obtaining permits and notifications, and Site preparation.

Health and Safety Requirements

Health and safety requirements for field activities will be specified in the Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). Personnel mobilized to the Site will be required to meet training requirements identified in Federal Regulation 29 CFR 1910.120 and applicable Occupational Health and Safety Administration (OSHA) training, including Hazardous Waste Operations and Emergency Response (HAZWOPER) and medical surveillance requirements. At least two personnel trained in first aid and cardiopulmonary resuscitation (CPR) will be on-Site during field activities. Training certificates for personnel (HAZWOPER 40-hour training; current HAZWOPER 8-hour refresher training; and first aid/CPR) will be maintained on-Site.

The Site Safety and Health Officer (SSHO) will have completed the 30-hour OSHA General Industry or Construction Industry Safety Class, as specified in EM-385 (USACE, 2014). The SSHO will be responsible for managing, implementing, and enforcing the health and safety program in accordance with the accepted APP/SSHP. The SSHO will be a competent person that can identify existing and predictable hazards in the working environment or working conditions that are dangerous to personnel, and who has authorization to take prompt corrective measures to eliminate them.

The FTL will have completed the 8-hour OSHA Supervisor training, as specified in EM-385. The FTL will lead field operations, coordinate field activities, and act as the liaison between Site and subcontractor personnel, among other responsibilities.

Field personnel will wear Level D personal protective equipment (PPE). Detailed Task Hazard Analysis (THA) forms identifying the physical, chemical, and biological hazards that may be encountered at the Site and the associated mitigation methods are presented in the APP/SSHP. If a change in conditions or scope occurs, the team will stop work and perform a THA to determine if the unanticipated change has resulted in a change sufficient enough to increase the level of PPE.

Personnel and visitors who enter the Site will be required to review the APP/SSHP and sign the acknowledgement form. Site workers will be required to sign the daily tailgate safety meeting form and fill out daily THA forms. Safety issues that arise during implementation of field activities will be addressed during tailgate safety meetings held daily before the workday and will be documented in the daily tailgate safety meeting.

Permits and Notifications

Site activities will be performed in accordance with permits, licenses, approvals, and/or certificates necessary to accomplish work specified in this QAPP. AECOM and subcontractors will remain in compliance with applicable zoning and ecological regulatory requirements. No intrusive work with mechanized equipment is being performed in wetlands and no wetland permitting is required. The Site has been identified as a suitable habitat for the Northern Long-Eared Bat and precautions have been detailed in this QAPP as a protective measure (see section regarding brush clearing). No further habitat assessment will be performed prior to the start of field activities.

In accordance with project kick-off meetings, anticipated work hours will be 0700 to 1900. Field activities conducted as part of this RI will be coordinated with the CENAE, stakeholders, and interested parties (e.g., Town of Foster, private homeowners, etc.).

Site Visit

Prior to the initiation of intrusive field work, the team will conduct a Site visit to mark out the locations of the soil boring locations. Selected locations will be marked in the field and coordinates will be recorded using a Trimble (or similar) global positioning system (GPS) receiver capable of sub-meter accuracy. Obstructions that limit access will be noted and contingent locations identified. The mark out of the locations will be utilized for utility clearance (see section regarding utility survey).

Brush Clearing

Portions of the Site are significantly overgrown with vegetation which will have to be removed to access sample locations for the safe installation of soil borings and permanent monitoring wells. To facilitate access, hand tools and mechanized equipment will be used to trim the overgrowth along the Site access road and clear paths to the sampling locations. Only small shrubs, brush, and saplings will be cleared. No hardwood trees will be removed. To protect the habitat of Northern Long-Eared Bats, no trees or sapling with a diameter 3-inces or larger will be removed. Cut brush will be left on-Site in the general vicinity of its generation. The vegetation will be cleared prior to the initiation of field activities.

Utility Survey

Per CENAE policies, utility clearance is required for intrusive work, regardless of planned intrusive depth. Prior to intrusive activities, the FTL is responsible for marking-out planned intrusive locations and opening a ticket with the DigSafe one-call utility clearance contractor. Coordination with the Town of Foster and representatives familiar with the Site will performed to obtain information on replacement water lines and other infrastructure. Precautionary measures (e.g., geophysical survey, air knifing methods, hand-digging to 5 ft, etc.) are required if utility clearance is not confirmed. Lack of confirmation can include urban locations, areas adjacent to roadways, areas not previously assessed, areas with insufficient utility information, or areas with multiple lines. The location of utilities will be noted and recorded during the Site visits and referenced when selecting investigation locations. Utility Clearance will be conducted in accordance with AECOM *SOP 3-01: Utility Clearance* (**Appendix C**).

Field Instrument Calibration and Quality Control

Equipment will be checked to ensure its completeness and operational readiness. Equipment found to be damaged or defective will be returned to the point of origin, and a replacement will be secured. Instruments and equipment that require route maintenance and/or calibration will be checked initially upon arrival and then prior to use each day, if needed to support the field activities scheduled for that day. Equipment calibration will be conducted in accordance with AECOM *SOP 3-20: Operational and Calibration of a PID* and AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling* (**Appendix C**).

This system of checks ensures that the equipment is functioning properly. If an equipment check indicates that a piece of equipment is not operating correctly and field repair cannot be made, the equipment will be tagged and removed from service, and a request for replacement equipment will

be placed immediately. Replacement equipment will meet the same specifications for accuracy and precision as the equipment removed from service.

Soil Boring Advancement and Surface and Subsurface Soil Sampling

Approximately 19 exploratory soil borings will be advanced across the Site to a maximum depth of 25 ft bgs. An additional four soil borings will be advanced solely for installation of monitoring wells (i.e. MW-006S, BR-002, BR-003, and BR-005). Drilling activities will be performed by a driller licensed by Rhode Island and have experience using roto-sonic drilling methods. Prior to the start of intrusive work, an exclusion zone will be established with cones, safety flagging, and notice surrounding the drilling operation. The drill team will place a sheet of plastic under the drill rig and pull it up around the tracks to act as a containment barrier in the event of a spill. The sonic drill will be advanced through a tub or similar wash bin to allow water to circulate through the borehole as necessary. A 4-inch core barrel and a 6-inch override casing will be utilized to core and case through to the target depth. Soil within each five-foot long core barrel will be extruded into a plastic sleeve. The plastic sleeve will be placed horizontally on clean plastic for logging and sampling purposes. The geologist will measure to the bottom of the boring within the 6-inch override casing to ensure the inside casing is open and clear to the bottom of the borehole. IDW generated (soil and water) will be properly handled and containerized within the Site laydown area.

Soil samples will be collected from each of the advanced borings. The target depth is specified for each location in **Worksheet #17** of this QAPP. The recovered soil bore will be screened for VOCs with a PID immediately upon opening the sleeve. The soil core will be logged for descriptions by an experienced and qualified field geologist. Observations and measurements will be recorded on a soil boring log. At a minimum, depth interval, recovery, PID concentrations, moisture, and texture using the Unified Soil Classification System (USCS) will be recorded. Additional observations to be recorded may include detectable odors, groundwater depth, organic materials, cultural debris, or color changes indicative of oxidation changes or staining. See AECOM *SOP 3-16: Soil and Rock Classification* and AECOM *SOP 3-21: Surface and Subsurface Soil Sampling Procedures* (**Appendix C**).

Two soil samples will be collected from each boring location unless otherwise specified in field modification documentation. Surface soil samples are considered to be 0 to 2 ft bgs and subsurface soil samples are considered 8-10 ft bgs. VOC samples will be collected directly from the soil core using a sampling corer (e.g., Terra Core, plastic syringe with tip removed, or similar) and in according with AECOM SOP: 3-21. For other analyses, soil will be removed and transferred to a disposable, re-sealable plastic bag. The sample will then be homogenized, which will consist of mixing the soil until the sample is a uniform color, texture, and particle size. Non-homogeneous particles, organic matter,

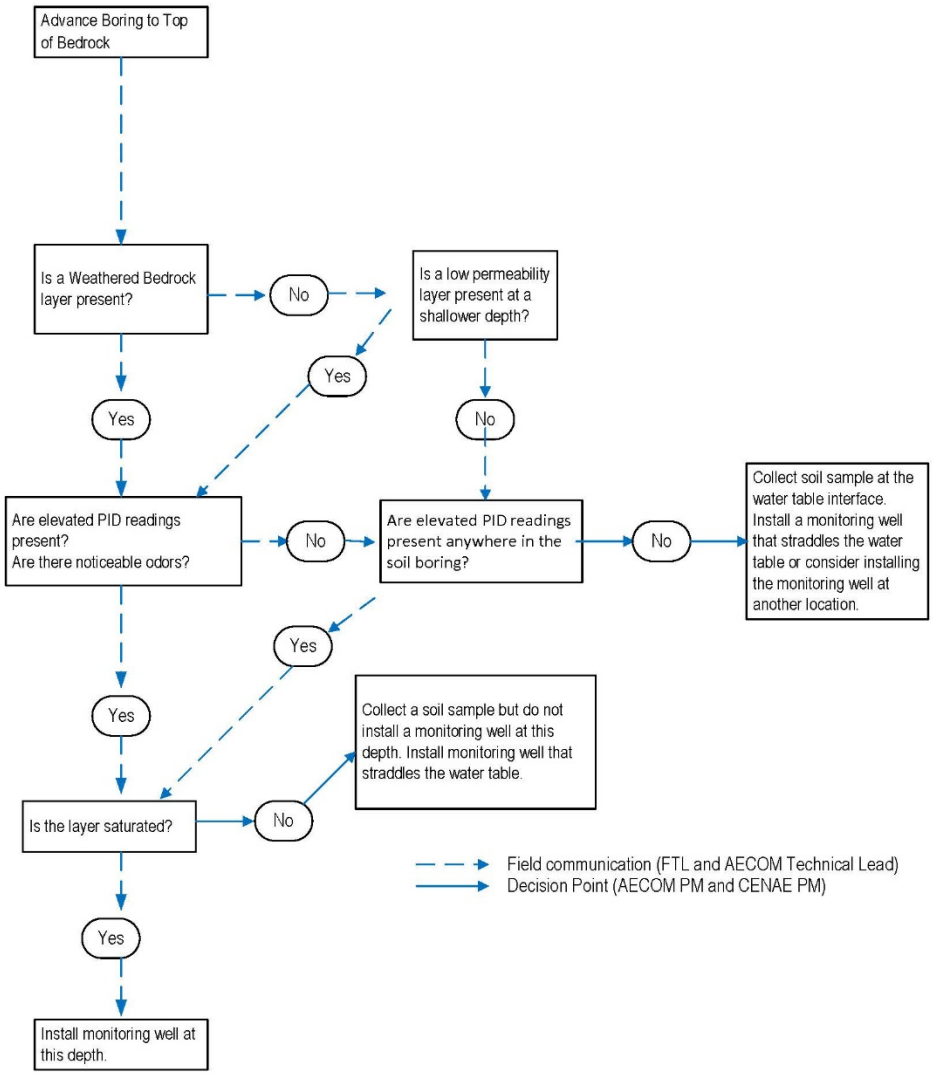
or other non-soil debris will be removed. After homogenization, the sample will be transferred to the appropriate sample containers for laboratory analysis. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain a preservation, as appropriate. The required samples containers, preservatives, and holding times are specified in **Worksheet #19 & 30**. Sample locations will be marked with a pin flag labeled with the sample identification number. The locations will also be photo-documented and recorded with a hand-held GPS.

To optimize the use of the monitoring well network, contingencies have been developed to relocate the proposed overburden, weathered bedrock, and bedrock monitoring wells based on observations and screening data obtained in real-time from the advancement of soil borings and soil sampling. With the exception of a few locations, the proposed monitoring well locations will be relocated based on these observations. To direct these decisions, a flow chart has been developed to direction field staff on protocols and procedures for engaging the team and receiving a decision point on a specific location. The flow chart has been included below.

Background Soil Sampling

In addition to the soil sampling performed at each AOC, surface and subsurface soil samples will be collected from within a 0.5 acre area within two background study areas. The two background study areas were identified as areas surrounding the Property representative of background soil conditions and sized to represent a typical residential lot. Twenty soil borings (ten in each study area) will be advanced to a depth of 10 ft bgs. All drilling activities will follow the same procedures as described above. Soil samples will be collected from each of the advanced borings. Surface soil samples are considered to be 0 to 2 ft bgs and subsurface soil samples are considered 2-10 ft bgs. An unbiased sampling approach will be used for evaluating subsurface soil by randomly selecting soil depths between 2-10 ft bgs using Pacific Northwest National Laboratory Visual Sample Plan software. This approach was taken rather than compositing the entire 2-10 foot interval due to site conditions (presence of cobbles/boulders) requiring large bore sonic cores. This technical drilling approach would generate too much soil to adequately composite for sampling. The target depth for subsurface sampling is specified for each location in **Worksheet #17** of this QAPP.

Figure 14-1: Monitoring Well Decision Tree



Overburden/Weathered Bedrock Monitoring Installation and Development

Nine permanent overburden and/or weathered bedrock monitoring wells will be installed during the RI field investigation. Overburden drilling procedures are detailed above in the *Soil Boring Advancement and Surface and Subsurface Soil Sampling* section. The rationale for the selected overburden monitoring well locations is provided in **Worksheet #17**. The exact depth and monitoring well construction details will depend on field observations of the geology and potential impacts.

An unused 2-inch diameter milled slot PVC screen and casing will be installed to the bottom of the borehole. The sand pack material will be selected based on the grain size distribution of the surrounding aquifer and placed in lifts as the drilling crew pulls the casing back to expose the well

screen. The screen slot size and length will be determined in the field based on the field observations and screening. The borehole above the sand pack will be sealed with 2 ft of hydrated bentonite chips. Water will be added if the seal is not in saturated material. Bentonite chips will be allowed to hydrate for one-hour prior to grouting. The remaining annular space between the well casing and the 6-inch override casing from the top of the bentonite seal to the ground surface will be pressure grouted with a bentonite/cement grout using a tremie pipe. The remaining 6-inch diameter sonic casing will be pulled from the ground. Vibration will be applied to the casing as it is pulled to densify and degas the grout, as well as knit the grout into the borehole wall, creating a superior seal.

Permanent overburden monitoring wells will be developed no sooner than a minimum of 24 hours after completion of well installation. Development will be completed by a combination of surging with a surge block and over-pumping with a submersible monsoon pump or Wattera pump and associated HDPE tubing, in accordance with AECOM *SOP 3-13: Monitoring Well Development* (**Appendix C**).

Low-flow sampling will be performed following the USEPA Guidance on low stress purging and sampling (USEPA, 2017). Water clarity will be visually monitored and water quality parameters, including dissolved oxygen (DO), specific conductance (SC), oxidation-reduction potential (ORP), pH, temperature, and turbidity will be measured using a flow-through cell every 5 minutes during purging to determine progress of development per the AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling* (**Appendix C**). The multi-parameter water quality meter will be calibrated at the beginning of each day. A calibration check[1] will be performed at the end of each day and anytime anomalous readings are observed. Each well will be developed until the well produces clear (silt-free) water with a minimum of 3 stable water quality readings as outlined below:

- pH – within ± 0.2 units.
- DO – within ± 10%
- SC – within ± 3 percent (%).
- ORP – within ± 10 millivolts (mV).
- Temperature – within ±1 degree Celsius.

[1] An anomalous reading is an unexpected measurement that is often accompanied with an instrument error message. For example a creeping reading, elevated readings in ambient air, etc. that would necessitate a calibration check

- Turbidity – at or below 10 nephelometric turbidity unit (NTU) or within ± 10% if above 10 NTU.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry a minimum of three times in succession and the turbidity has decreased. If water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development.

Reusable sampling equipment will be properly decontaminated after each use in accordance with AECOM SOP 3-06 (**Appendix C**). Excess soil or groundwater generated will be containerized, managed, and disposed of as IDW.

Bedrock Monitoring Well Installation and Development

Five permanent bedrock monitoring wells will be installed during the RI field investigation. The rationale for the selected bedrock monitoring well locations and target depths is provided in **Worksheet #17**. Drilling activities will be performed by a driller licensed by the Rhode Island and have experience using air rotary drilling methods. Prior to starting, the overburden and weathered bedrock units should be sealed off (this should be done by the roto-sonic drill rig team). To seal off the overburden and weathered bedrock, 6-inch permanent steel casing will be advanced 5 ft into competent rock and tremie-grouted to the surface as 8-inch temporary casing is withdrawn. The top of the 6-inch casing will be threaded to allow future attachment of a temporary casing extension at the surface, if needed during FLUTe installation. Air rotary drilling may proceed after a minimum 24-hour curing period.

Drilling will advance to the target depths listed in **Worksheet #17**. The recovered rock chips/fragments will be logged (as best as possible) for descriptions by an experienced and qualified field geologist. Unlike the USCS for soils, there is no single standard rock classification system; however, the field geologist will describe the essential items. At a minimum, depth interval, rock classification name, color, mineralogical composition and percentage, and texture/grain size should be recorded. See AECOM *SOP 3-16: Soil and Rock Classification* and EM-1110-1-1804.

Permanent bedrock boreholes will be developed at least 24 hours after completion of well installation. Development will be completed by a combination of surging with a surge block and a venturi air lift pump. The construction of the surge block will be appropriate for the 5-inch diameter borehole and be mounted on rods for downhole advancement. The boreholes will be surged in 10-foot intervals followed by purging. Bedrock borehole development will be performed by the drilling subcontractor with oversight from the field geologist.

Similar to the overburden well development, water clarity will be visually monitored and water quality parameters measured using a flow-through cell every 5 minutes during purging to determine progress of development per the AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling*. Groundwater generated will be containerized, managed, and disposed of as IDW (see section regarding IDW management).

Bedrock Borehole Geophysics and FLUTe

After completion of the bedrock well development, borehole geophysical logging will be performed on all five bedrock boreholes. If a significant period of time is scheduled between the borehole development and geophysical logging, a blank FLUTe liner will be installed within the borehole to act as a seal and prevent intra-borehole flow. Once geophysical logging is scheduled, testing will be performed include using the following tools:

- Acoustic caliper
- Fluid temperature
- Fluid conductivity/resistivity
- Optical televiewer (OTV)
- Acoustic televiewer (ATV)
- Natural gamma ray
- Spontaneous potential/resistivity
- Heat pulse flow meter (HPFM) under ambient conditions
- HPFM under pumping conditions

The results of the geophysical logging will be one line of support evidence in the development of the final FLUTe Multi-Level Sampler (MLS) liners.

Following completion of the geophysical logging, a blank FLUTe liner will be installed in each of the five bedrock boreholes. The blank FLUTe liner will be equipped with FLUTe Activated Carbon Technique (FACT) covered with felt strips. The FACT strips will remain in the borehole for a minimum period of two weeks. After this wait period, the FACT strips will be removed and visually inspected and screened with PID. The FACT strip will be cut into three-foot segments, bottled, and shipped to

a laboratory subcontracted by FLUTe for analysis. The FACT strip will be labeled with the borehole identification and placed in a clean trash bag for on-Site storage.

As a final test, the FLUTe blank liner will be reused to conduct a transmissivity and reverse head profile in each of the five bedrock boreholes. Upon completion of the transmissivity and reverse profiling, the blank FLUTe liners will remain in the borehole to act as a seal and prevent intra-borehole flow until the blank FLUTe liners can be replaced. Borehole information obtained during the FLUTe testing will be compiled and processed by FLUTe. The specific procedure for the FLUTe transmissivity testing is provided in the FLUTe SOPs in **Appendix D**.

Results of the borehole geophysical logging will be combined with FLUTe transmissivity and reverse head profiles, and FLUTe FACT strip analytical results. The objectives of the geophysical logging will be to identify potential water-bearing fracture zones in the bedrock along the length of each open hole, to define fracture depths, strikes, and dips, and to rate the actual and potential inflow/outflow of a subset of those fractures. A secondary goal is identifying changes in bedrock lithology. The FLUTe testing will provide information regarding the transmissivity of the borehole every foot and the relative distribution of dissolved phase VOCs in the borehole.

The team will review the integrated geophysical and FLUTe borehole data and determine the FLUTe MLS liner construction details for the five bedrock monitoring wells. FLUTe will then construct the MLS liners in accordance to the specifications provided. Each of the FLUTe MLS liners will include three sampling ports for a total of 15 sampling ports. If warranted and based on the review of the integrated well logs, additional ports at one or more of these locations may be included in the design and construction of the MLS liners. All FLUTe work will be performed by qualified personnel trained by FLUTe in the installation and operation of their equipment.

Overburden Groundwater Sampling

Overburden and weathered bedrock monitoring wells will be sampled at least 48-hours after completion of well development, which is a deviation from the 24-hour wait period referenced in AECOM *SOP 3-14: Groundwater Sampling* (**Appendix C**).

Groundwater levels will be measured in each well prior to sampling using a water level meter (Solonist or equivalent). The monitoring wells will be purged using low-flow sampling techniques using a bladder or peristaltic pump and disposable tubing in accordance with AECOM SOP 3-14. Water clarity will be visually monitored and water quality parameters, including DO, SC, ORP, pH, temperature, and turbidity will be measured using a flow-through cell per the AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling*. Readings will be collected every 5 minutes until the

well produces clear (silt-free) water for a minimum of 3 stable water quality readings, as outlined above in the Well Development procedures. The multi-parameter water quality meter and turbidity meter will be calibrated at the beginning of each day. A calibration check will be performed at the end of each day and anytime anomalous readings are encountered.

Once the water quality parameters reach stabilization, samples will be collected in laboratory-supplied bottleware for the parameters established in **Worksheet #18**. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain a preservation, as appropriate. Non-disposable sampling equipment will be decontaminated between each well per AECOM *SOP 3-14: Monitoring Well Sampling*.

Bedrock Groundwater Sampling

Bedrock groundwater sampling will be performed after the MLS liners are installed in each of the five bedrock boreholes. Groundwater sampling procedures will be developed by FLUTe based on the transmissivity results. The specific sampling gas pressure (purge and sample) will be calculated and used during the purge and sampling process. Per FLUTe guidance, the pump and sample tube should be purged at the specific purge pressure four times prior to sampling. After this, sampling at the designated sample pressure can begin on the fifth cycle. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain a preservation, as appropriate. The specific sampling procedures for all five of the FLUTe bedrock wells is included in the FLUTe SOP in **Appendix D**.

Background Bedrock Groundwater Sampling

In addition to the groundwater sampling performed within the AOCs, one bedrock groundwater sample will be collected from a nearby non-impacted upgradient well as a background sample. One sample will be collected following the same procedures outlined in the *Overburden Groundwater Sampling* subsection. Once the water quality parameters reach stabilization, samples will be collected in laboratory-supplied bottleware for the parameters established in **Worksheet #18**. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain a preservation, as appropriate. Non-disposable sampling equipment will be decontaminated between each well per AECOM *SOP 3-14: Monitoring Well Sampling*.

Surface Water, Pore Water, and Sediment Sample Collection

Surface water and co-located pore water and sediment/soil samples will be collected from surface water exposure areas surrounding the Property. Surface water samples will be collected first at each location, prior to sediment sampling according to the procedures in AECOM *SOP 3-10: Surface Water and Liquid Sampling*. Surface water samples should not be collected directly following a rain event. Every effort will be made to collect surface water samples after several days without precipitation so that samples will represent baseflow conditions (i.e. surface water sampling to occur following three consecutive days of zero precipitation). Sampling will occur from downstream to upstream in locations where surface flow direction can be clearly identified; agitation of the sediment and water at shallow locations will be minimized. Physical characteristics of the sampling locations (e.g., water depth, stream width, etc.) will be documented. The full list of surface water analyses is included in **Worksheet #18**.

For unfiltered surface water samples, water will be dipped or pumped from the source using a peristaltic pump with disposable tubing and placed into the appropriate laboratory-supplied bottleware. At the completion of sampling at each location, field parameters including ORP, pH, SC, temperature, DO, and turbidity will be measured with a water quality meter and recorded in the field logbook or sampling form.

Once surface water sampling is complete, sediment samples will be collected from 0 to 0.5 ft bgs using a hand-driven coring barrel or a dedicated disposable scoop in accordance with AECOM *SOP 3-22: Sediment Sampling*. Sediment sample locations will target fine grained material from depositional areas. The full list of sediment analyses is included in **Worksheet #18**. Material such as twigs, leaves, and stones will be removed from the samples prior to homogenization and documented in the field log or field forms. Sediment samples will be homogenized in disposable bags prior to filling laboratory-supplied sample containers. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain a preservation, as appropriate.

After completion of surface water and sediment sampling, pore water samples will be collected from the bioactive zone as defined by the USEPA (0 to 1 ft bgs) using a push-point sampler (i.e.: Henry sampler) in accordance with AECOM *SOP 3-45: Pore Water Sampling* (USEPA, 2013). Prior to sampling, the sample locations should be assessed (during the Site walk) to determine the best method for collection. The pore water samples should specifically target the water within the interstitial pore space of sediment below the surface of the overlaying sediment and within the bioactive zone.(define and cite USEPA Guidance). To achieve this and prevent intrusion of overlaying

surface water intrusion, a flange should be placed at the desired sampling location with the push-point sampler removed to allow water to escape. The size of the flange should correlate to the volume of water required for the analytical sampling suite. With the flange in place, the push-point sampler can be inserted through the compression adapter on the flange and pushed to the desired depth for sample collection. Manometers will be used to gauge water level data following sample collection from within the interval sampled by the Henry sampler.

Surface water, sediment, and pore water sampling locations will be flagged in place and depicted in the field logbook for use during land surveying. Additionally, at each location the depth of water and width of channel will be recorded. Refer to AECOM *SOP 3-10: Surface Water and Liquid Sampling* and AECOM *SOP: 3-22 Sediment Sampling* for additional details.

In addition to the proposed surface water, pore water, and sediment sampling, two gauging stations will be installed along the Ponaganset River south of the Property to measure flow at the lowest elevation in the area. The gauging station locations are shown in **Figure 17-3**.

Ferrous Iron Analysis

The concentration of ferrous iron in groundwater samples will be analyzed in the field using the HACH DR890 Colorimeter and HACH Method 8146 according to the procedures in AECOM *SOP 3-18: Field Analysis of Ferrous Iron Using the HACH DR890 Colorimeter and HACH Method 8146*. Samples will be analyzed immediately after collection because ferrous iron readily oxidizes to ferric iron upon exposure to air. Further details are provided in AECOM *SOP 3-18: Field Analysis of Ferrous Iron Using the HACH DR890 Colorimeter and HACH Method 8146*.

Hydraulic Conductivity Analysis (Slug Testing)

Hydraulic conductivity testing will be performed in the new overburden and weathered bedrock monitoring wells. The slug testing will commence at least a week after development and 48-hours after groundwater sampling to allow the well to return to equilibrium. Hydraulic conductivity will be conducted in accordance with AECOM *SOP 3-35: In-Situ Hydraulic Conductivity Testing via Rising or Falling Head Slug Testing*.

Field Quality Control Samples

Field QC samples will be collected as part of this investigation, including field duplicates (FDs), MS/MSDs, equipment blanks (EBs), trip blanks (TBs), and temperature blanks. FD samples will be collected at a rate of 10% and analyzed for the same parameters as the accompanying samples. MS

and MSD samples will be collected at the rate of 5% and analyzed for the same parameters as the accompanying samples.

TBs will accompany each cooler containing samples for VOC analysis and will be analyzed for select VOCs. If non-dedicated sampling equipment is used, an EB will be collected and analyzed for whatever parameters were collected using the non-dedicated sampling equipment. A temperature blank shall be placed in each cooler to ensure that samples are preserved at or below 4 degrees Celsius (°C) during shipment.

Sampling Handling, Storage, and Transport

Samples will be stored on ice, packaged, and submitted to the analytical laboratory for analysis. **Worksheet #17** provides the soil sampling design and rationale. The combined **Worksheet #19 & 30** provide sample identifications, necessary sample volume and preservative requirements, and hold time limitations. Samples will be quality-control checked by the FTL (label correctness, completeness, etc.) and recorded on Chain-of-Custody forms. Samples will be packaged on ice and transported via overnight commercial carrier or a laboratory courier under standard chain-of-custody procedures to the laboratory. Sample handling activities will be performed in accordance with AECOM *SOP 3-04: Sample Handling, Storage, and Shipping*.

Field Documentation

Field documentation will be performed during this investigation in accordance with AECOM SOP 3-02. Sample collection information will be recorded in bound field notebooks, tablet computers, or specific field forms. A summary of field activities will be properly recorded in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and stored in a secured area when not in use. Entries will be written in indelible ink, and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; and the person making the correction will initial and date the change. Sampling forms and other field forms will also be used to document field activities.

Equipment Decontamination

To the maximum extent possible, the team will utilize dedicated and disposable sampling equipment to avoid the potential for cross contamination of samples due to inadequate decontamination processes. The dedicated/disposable sampling equipment will include disposable polyethylene tubing, disposable gloves, and laboratory-supplied sample bottles.

Non-disposable or non-dedicated sampling equipment (e.g., core barrel, bladder pumps, water level meters, etc.) will be decontaminated prior to sampling and between samples following AECOM SOPs. Cleaning of equipment is performed to prevent cross-contamination between samples and to maintain a clean working environment for field personnel. Decontamination will generally consist of a water rinse station to remove gross contamination (if needed), followed by a non-phosphate detergent (e.g., Liquinox) water rinse, and a rinse with de-ionized water (provided by the laboratory). Paper towels containing recycled paper content are prohibited. If decontaminated equipment is to be stored or transported, it will be wrapped in aluminum foil after air-drying. Decontamination activities will be performed in accordance with AECOM *SOP 3-06: Decontamination*.

Land Surveying

The horizontal and vertical position of permanent monitoring wells will be surveyed by a state-registered surveyor to a horizontal accuracy of 0.1 ft and a vertical accuracy of 0.01 ft. These positions will be tied to a permanent benchmark located near the Site and referenced to the NAD83 (horizontal) and North American Vertical Datum (NAVD) 88 (vertical) datums. Land survey of subsurface sample locations will be conducted in accordance with applicable specifications. Land survey activities will be conducted in accordance with AECOM *SOP 3-07: Land Surveying*.

Investigation Derived Waste Management

IDW generated during Site field activities will be managed pursuant to applicable Federal, State, and local regulations and guidance, including USACE guidance (2013) and RIDEM Policy Memo 95-01 Guidelines for the Management of Investigative Derived Waste (RIDEM, 1995). Refer to AECOM *SOP 3-05: Investigation-Derived Waste Management* for procedures related to IDW management. Department of Transportation (DOT) compliant shipping containers will be used to stage IDW prior to off-Site transport. Solid IDW (e.g., drill cuttings from boring/monitoring well installation that cannot be returned to the borehole of origin) will be stored in 55-gallon metal drums and/or a 20 cubic yard closed-top roll-off bin; liquid IDW (e.g., monitoring well development water, purge water, decontamination water) will be stored in frac tanks and/or 55-gallon metal drums.

The IDW containers will be properly labeled, sampled for waste characterization, and temporarily staged on-Site at a designated secure location until waste characterization is completed. The IDW containers will subsequently be transported to the approved off-Site disposal facility; the intended facility will confirm their acceptance of the waste prior to transport. IDW removal from the Site will be documented by manifest or bill of lading prepared by the waste disposal subcontractor.

Piezometer and Borehole Abandonment

An unidentifeid pipe and existing piezometers that are deemed comprimised or inadequent for long-term use will be abandoned in accordance with the procedures in the RIDEM Groundwater Quality Rules Appendix 1 (RIDEM, 2010) and AECOM *SOP 3-15: Monitoring Well and Borehole Abandonment*. The rationale for the piezometers selected for abandonment is provided below. Soil boring locations not converted into permanent monitoring wells will be abandoned by backfilling with bentonite chips. Borings in asphalt or concrete shall be abandoned by backfilling with bentonite chips to approximately 6 inches bgs, and the remainder of the borehole will be patched with asphalt cold patch or hydraulic concrete. The surface at each location will be restored to match the surrounding area. Piezometer and borehole abandonment will be performed by a state licensed driller.

Table 14-1: Rationale for Piezometer Abandonment

Piezometer ID	Abandon (Y/N)	Rationale
PZ-001	N	Keeping due to previous VOC detections and location as future gauging well.
PZ-002	Y	Very slow recharge rate.
PZ-003	Y	Very slow recharge rate.
PZ-004	Y	Highly turbid well, very slow recharge rate.
PZ-005	N	Keeping due to previous 1,1-DCE detection, good pairing with proposed WB well MW-04.
PZ-006	Y	No detections during previous sampling event. Dry during gauging events in July 2013, June 2014, and July 2019.
PZ-007	N	Keeping due to previous VOC detections and location between sources and southern Site boundary.
PZ-009	Y	No VOC detections during previous sampling event.
PZ-010	N	Keeping due to location as gauging well.
PZ-011	Y	Highly turbid well.
PZ-014	N	Keeping due to location as gauging well, good pairing with proposed BR well BR-01.
PZ-015	Y	No detections during previous sampling event. Dry during gauging events in July 2013, June 2014, and July 2019.
PZ-019	Y	Highly turbid well, likely due to bentonite in piezometer.
PZ-022	Y	Highly turbid well.
Unidentified Pipe	Y	Unknown history of use or purpose.

14.2 Data Management, Review, and Validation

The principal data generated for this project will be from laboratory analytical data which will be managed through the FUDSChem database. Copies of the field forms, Chains of Custody (CoCs), air bills, and logbooks will be placed in the project files after completion of the field program. CoCs are also uploaded to FUDSChem. The field logbooks for this project will be used only for this Site and will also be categorized and maintained in the project files after the completion of the field program. Project records will be maintained in a secure location.

Data Tracking

Data are tracked from the sampling event planning phase through the completion of validation through the FUDSChem database. Data are uploaded by the laboratory directly to the database. Reports are uploaded in pdf format. With the exception of grain size, Electronic Data Deliverables (EDDs) are also submitted to the database. These are in Staged Electronic Data Deliverable (SEDD) 2a format. Once data are successfully submitted, the Project Chemist will oversee the data validation effort.

Data Review and Validation

Upon successful upload of SEDD files to FUDSChem by the analytical laboratory, data will undergo verification and partial validation through the Automated Data Review (ADR) software utilizing the project eQAPP. The Chemist will then verify the validation conducted by ADR and augment with manual validation as needed for the Stage of validation selected. To assess whether the analytical results meet the project quality objectives, the laboratory data will undergo verification and validation as cited in **Worksheets #34 through #36** and described below. The usability assessment processes are described in **Worksheet #37**.

Prior to data validation, electronic laboratory data will be verified for accuracy against the hardcopy laboratory report, and the electronic QAPP (eQAPP) will be established using the project-specific criteria defined in **Worksheets #12, #19,** and **#28**. The laboratory will be requested to resubmit electronic data found to be inaccurate. Laboratory calibration will be assessed against the criteria presented in **Worksheet #24**.

Data Storage, Archiving, and Retrieval

Fixed laboratory data packages are stored in the FUDSChem database along with validated analytical results and completed validated reports. Field data such test kits results and water quality parameters measured during low-flow sampling are entered into the FUDSChem database as EMI (Environmental

Measurement Information) files. CoCs are loaded to FUDSCHem. Field records including field logbooks, sample logs, CoC records, and field calibration logs will be submitted by the AECOM field team lead to be entered into the file system before archiving in secure project files. Project files will be kept in a secured, limited access area.

14.3 Report Preparation

Following the completion of data collection during the field investigation, laboratory analysis, and data validation, a comprehensive RI Report will be prepared per CERCLA guidance. The RI report will present the methods used for the RI, the refined CSM resulting from the investigation, the results of the Site characterization and comparison to risk-based human health and ecological screening levels and BTVs, and a recommendation of whether further investigation, consisting of un-biased, exposure-based sampling is needed.

Additionally, the RI Report will include the following elements:

- Restatement of the study goals;
- Facility background, environmental setting, previous investigations, and current and future land use;
- Summary of field investigation conducted (e.g., sampling dates, soil samples collected, monitoring wells sampled, parameters analyzed, and field procedures);
- Physical characteristics of the study area, including soils, geology, hydrogeology, hydrology, and ecological setting;
- Deviations from the QAPP and/or QAPP modifications;
- Tables summarizing the samples collected and sample analytical data;
- Figures showing the layout of each sampling area, updated Site features, results of geophysical surveys, soil boring locations, and summaries of pertinent analytical results;
- Discussion of data validation and precision, accuracy, representativeness, comparability, completeness, and sensitivity;
- Data evaluation and identification of critical data gaps.

- Fate and transport discussion, including potential routes of migration, COPC persistence, and COPC migration;
- Summary of the areas with COPCs identified as being detected above human health and ecological screening levels and BTVs; and
- Conclusions, summary of the RI findings, and recommendations.

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

The following Worksheet #15 tables identify the project action limits (PALs) and project quantitation limit goals (PQLGs) and provide a comparison of the PALs and PQLGs to analytical laboratory reference limits (i.e., LODs, and LOQs) for surface soil, subsurface soil, leachate, groundwater, sediment, surface water, and porewater, 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. Details on PAL selection are provided by matrix in notes pages following the Worksheet #15 tables. For analytes for which published screening levels are not available, surrogate analytes have been identified, as available, as identified in the Worksheet #15 tables.

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.

Analytes without published screening levels/regulatory criteria for which appropriate surrogate analytes are not available do not have PALs established in the Worksheet #15 tables. However, these analytes are being tested to provide data necessary for use in the RI or later stage in the CERCLA process, including adjustment of ecological screening criteria which are dependent on these parameters, to characterize bioavailability or other conditions, to provide a measure of monitored natural attenuation, etc. Major metals such as calcium, magnesium, potassium, and sodium provide useful information regarding geochemistry and will be used for qualitative comparison of overburden, bedrock, and surface water quality. Calcium and magnesium are also necessary for the laboratory to calculate hardness which is used to adjust ecological screening criteria.

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

- Detection Limit (DL) – The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

- 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.

- Project Quantitation Limit Goal (PQLG) – The desired limit of quantitation set at some fraction of the PAL to account for the potential for error around the actual result.

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

1. If no PAL is available, the PQLG = 5 times the LOQ;
2. If the LOQ ≤ ½ the PAL, the PQLG = ½ PAL;
3. If the LOQ > ½ the PAL, the PQLG = LOQ.

For certain analytes, the PAL is not analytically achievable, thus, the DL, LOQ and/or LOD > the PAL. DLs, LODs and LOQs > the PAL are highlighted in the tables. Site-related analytes for which the DL, LOD and/or LOQ > PAL are also discussed further below by analyte type.

Any J-flagged (estimated) result with estimated concentrations < the LOQ or LOD will be considered a detected result at the reported concentration, in accordance with USEPA CERCLA practice.

The data usability section and the uncertainty section of the RI/risk assessment text will summarize any laboratory issues (as reported in the data validation reports) that may have occurred to cause a result to be J-flagged, and will discuss the impact on the RI/risk assessment results in general terms (e.g., whether the use of J-flagged results for a particular analyte could bias the results high or low). For a non-detect result (reported at LOD) with the PAL < the LOD for the analyte, the LOD will be evaluated in the RI/risk assessment. For a J-flagged result between the LOQ and the DL with a PAL < LOQ, the estimated detection will be considered to be < the PAL, and the J-flagged result will be used in the RI/risk assessment. For example, if the LOQ = 5, the PAL = 4, the DL = 1, and the J-flagged estimated detected result = 3 J, it cannot be determined that the estimated detected result of 3 J is actually below the PAL of 4. Nevertheless, the J-flagged result of 3 will be the concentration used in the RI/risk assessment. The RI/risk assessment will not quantify the uncertainty associated with J-flagged results (e.g., upper bound risk/hazard estimates will not be calculated based on LODs).

VOCs/SVOCs

Analysis using Selected Ion Monitoring (SIM) will be conducted to obtain lower LOQs and LODs for a select group of VOC and SVOC analytes with PALs that are less than the full scan limits. Analytes requiring SIM were selected based on the likelihood that the analyte is Site-related and professional judgement as to the analytes that are more likely to drive potential risk in a risk assessment. Of these, the laboratory has not yet developed SIM limits for 1,2,3-trichlorobenzene.

SIM is the most sensitive option available from commercial laboratories for multi-compound analysis of VOCs and SVOCs that retains the specificity of GC/MS analysis.

- Vinyl chloride – The SIM LOQ and LOD for vinyl chloride in groundwater and surface water are greater than the associated PALs. These are discussed further below:
  - The selected groundwater PAL is equal to the USEPA tapwater RSL based on a 1×10^{-6} target risk level and target HQ of 0.1. The groundwater LOQ and LOD do not exceed the tapwater RSL adjusted for a 1×10^{-5} target risk level and target HQ of 0.1 (0.19 µg/L). They are also less than the USEPA MCL and RIDEM Method 1 GA Groundwater Objective, which both equal 2 µg/L. Therefore, the laboratory limits for vinyl chloride in groundwater are considered appropriate for meeting project DQOs.
  - The selected surface water PAL is equal to USEPA National Recommended Water Quality Criteria (NRWQC) based on human ingestion of water and organisms. The surface water LOQ and LOD are less than the USEPA NRWQC based on human ingestion of organism and the ecologically-based PALs. Therefore, there is some level of uncertainty associated with the surface water LOQ and LOD being > the associated human health PAL, which will be qualitatively acknowledged in the RI/risk assessment report (e.g., indicating whether the associated uncertainty could bias the results of the risk assessment high or low).
- PAHs – The SIM LOQ and LOD for certain PAHs in groundwater, porewater, surface water, and/or sediment are greater than the associated PALs. Additional discussion is provided below by matrix:
  - The selected groundwater PALs for benzo(a)anthracene, benzo(a)pyrene, dibenz(a,h)anthracene, and naphthalene are equal to the USEPA tapwater RSL based on a 1×10^{-6} target risk level and target HQ of 0.1. The groundwater LOQ and LOD do not exceed the tapwater RSL adjusted for a 1×10^{-5} target risk level and target HQ of 0.1. They are also equal to or less than the USEPA MCL and RIDEM Method 1 GA

Groundwater Objective for benzo(a)pyrene, which both equal 0.2 µg/L. The USEPA MCL and RIDEM Method 1 GA Groundwater Objective for benzo(a)pyrene are the most conservative of the available PAH screening levels from these sources (there are no USEPA MCL or RIDEM Method 1 GA Groundwater Objective screening levels for benzo(a)anthracene or dibenz(a,h)anthracene and the RIDEM Method 1 GA Groundwater Object for naphthalene is 100 ug/L). Therefore, the laboratory limits for PAHs in groundwater are considered appropriate for meeting project DQOs.

- The selected porewater PALs for anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(k)fluoranthene, dibenz(a,h)anthracene, and indeno(1,2,3-c,d)pyrene are based on the USEPA Region 4 freshwater surface water screening levels. There is some level of uncertainty associated with the surface water LOQ and LOD being > the associated ecological PALs.
- The selected surface water PALs are equal to USEPA NRWQC based on human ingestion of water and organisms or USEPA Region 4 ecological screening level. There is some level of uncertainty associated with the surface water LOQ and LOD being > the associated PALs; which will be qualitatively acknowledged in the RI/risk assessment report (e.g., indicating whether the associated uncertainty could bias the results of the risk assessment high or low).
- The selected sediment PALs for anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, and indeno(1,2,3-c,d)pyrene are equal to the USEPA Region 4 ecological screening levels. There is some level of uncertainty associated with the surface water LOQ and LOD being > the associated ecological PALs. However, limits are less than the human health PALs. The associated uncertainty will be qualitatively acknowledged in the RI/risk assessment report (e.g., indicating whether the associated uncertainty could bias the results of the risk assessment high or low).

Metals

Select LOQs and LODs for metals analytes were greater than the associated PALs. These are discussed further below:

- Aluminum and Copper - The ICP/AES LOQs and LODs for aluminum and copper in surface water and porewater are greater than the associated PALs. The PALs for aluminum are based on the USEPA Region 4 and RIDEM surface water screening levels for freshwater, which are protective of ecological receptors. There are no surface water screening levels for human health for

aluminum. The PAL for copper is based on the USEPA NRWQC for freshwater. The PAL is below the USEPA tapwater RSL (80 µg/L) and the USEPA MCL (1,300 µg/L). Therefore, there is some level of uncertainty associated with the surface water and porewater LOQs and LODs being greater than the associated PALs.

- Arsenic, Beryllium, Silver, and Thallium – The ICP/MS LOQs and LODs for arsenic, beryllium, silver, and thallium in surface water, groundwater, and/or porewater are greater than the associated PALs. The selected PALs for arsenic and thallium in groundwater are equal to the USEPA tapwater RSLs based on a 1×10^{-6} target risk level and target HQ of 0.1. Although the LOQ and LOD for arsenic are greater than the selected PAL, they are below the USEPA MCL for arsenic (10 ug/L). The selected PALs for arsenic, thallium, and silver in surface water and porewater are based on USEPA NRWQC and the USEPA Region 4 surface water screening levels for freshwater. The selected PALs for beryllium in surface water and porewater are based on RIDEM surface water screening levels for freshwater (chronic), which are protective of ecological receptors. There is some level of uncertainty associated with these LOQs and LODs being greater than the associated PALs. However, ICP/MS is the most sensitive option available from commercial laboratories for multi-compound analysis of metals that retains the specificity of the ICP analysis.

  - The ICP/AES LOQ and LOD for thallium in leachate also is greater than the associated PAL. The selected PAL for thallium in leachate is based on the RIDEM Method 1 GA Leachability Criteria. Therefore, there is some level of uncertainty associated with the thallium LOQ and LOD for leachate being greater than the associated PAL.

- Mercury – The Manual Cold-Vapor Technique LOQ and LOD for mercury in soil is greater than the associated PALs for surface soil and background soil. The selected PAL for mercury in soil is based on the lowest available USEPA Region 4 soil ecological screening level. Therefore, there is some level of uncertainty associated with the mercury LOQ and LOD being greater than the associated PAL. However, the LOQ and LOD for mercury are lower than the human health PAL for mercury. The associated uncertainty will be qualitatively acknowledged in the RI/risk assessment report (e.g., indicating whether the associated uncertainty could bias the results of the risk assessment high or low).

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

Matrix: **Aqueous**
Analytical Group: **Alkalinity by Titrimetric Method (A2320B D Alkalinity)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Alkalinity, Total (as CaCO3)			25000	5000	4000	228

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

Matrix: **Aqueous**
Analytical Group: **Hardness by Calculation (A2340B E Hardness)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Hardness (as CaCO3)			25000	5000	2500	1630

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

Matrix: **Aqueous**
Analytical Group: **Standard Method for Sulfide (Titrimetric, Iodine) (A4500SF D Sulfide)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Sulfide			5000	1000	800	690

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

Matrix: **Aqueous**
Analytical Group: **Standard Method for the Determination of Total Organic Carbon, Combustion IR Method (A5310B D TOC)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Total Organic Carbon			5000	1000	500	102

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

Matrix: **Aqueous**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM D SVOC SIM Groundwater)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	0.460	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.250	0.250	0.180	0.0850
2-Methylnaphthalene	3.60	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.80	0.200	0.100	0.0770
Acenaphthene	53.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	26.5	0.200	0.100	0.0640
Acenaphthylene	53.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1); Value for Acenaphthene	1.00	0.200	0.100	0.0540
Anthracene	180	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	90.0	0.200	0.100	0.0440
Benzo(a)anthracene	0.0300	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.200	0.100	0.0460
Benzo(a)pyrene	0.0250	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.200	0.100	0.0660
Benzo(b)fluoranthene	0.250	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.200	0.100	0.0890
Benzo(g,h,i)perylene	12.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1); Value for Pyrene	1.00	0.200	0.100	0.0650
Benzo(k)fluoranthene	2.50	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.25	0.200	0.100	0.0490
Chrysene	25.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	12.5	0.200	0.100	0.0360
Dibenz(a,h)anthracene	0.0250	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.200	0.100	0.0700
Fluoranthene	80.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	40.0	0.200	0.100	0.0730
Fluorene	29.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	14.5	0.200	0.100	0.0610
Indeno(1,2,3-c,d)pyrene	0.250	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.200	0.100	0.0520
Naphthalene	0.170	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.200	0.100	0.0640
Pentachlorophenol	0.0410	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.330
Phenanthrene	12.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1); Value for Pyrene	1.00	0.200	0.100	0.0510
Pyrene	12.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	6.00	0.200	0.100	0.0590

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM E SVOC SIM Porewater)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	22000	USEPA Region 4 surface water screening levels - freshwater chronic	11000	0.250	0.180	0.0850
2-Methylnaphthalene	4.70	USEPA Region 4 surface water screening levels - freshwater chronic	2.35	0.200	0.100	0.0770
Acenaphthene	1.90	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	0.950	0.200	0.100	0.0640
Acenaphthylene	13.0	USEPA Region 4 surface water screening levels - freshwater chronic	6.50	0.200	0.100	0.0540
Anthracene	0.0200	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0440
Benzo(a)anthracene	4.70	USEPA Region 4 surface water screening levels - freshwater chronic	2.35	0.200	0.100	0.0460
Benzo(a)pyrene	0.0600	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0660
Benzo(b)fluoranthene	2.60	USEPA Region 4 surface water screening levels - freshwater chronic	1.30	0.200	0.100	0.0890
Benzo(g,h,i)perylene	0.0120	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0650
Benzo(k)fluoranthene	0.0600	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0490
Chrysene	4.70	USEPA Region 4 surface water screening levels - freshwater chronic	2.35	0.200	0.100	0.0360
Dibenz(a,h)anthracene	0.0120	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0700
Fluoranthene	0.800	USEPA Region 4 surface water screening levels - freshwater chronic	0.400	0.200	0.100	0.0730
Fluorene	19.0	USEPA Region 4 surface water screening levels - freshwater chronic	9.50	0.200	0.100	0.0610
Indeno(1,2,3-c,d)pyrene	0.0120	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0520
Naphthalene	2.60	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.30	0.200	0.100	0.0640
Pentachlorophenol	0.603	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.00	1.00	0.500	0.330
Phenanthrene	2.30	USEPA Region 4 surface water screening levels - freshwater chronic	1.15	0.200	0.100	0.0510
Pyrene	4.60	USEPA Region 4 surface water screening levels - freshwater chronic	2.30	0.200	0.100	0.0590

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

Matrix: **Aqueous**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM F SVOC SIM Surface Water)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	22000	USEPA Region 4 surface water screening levels - freshwater chronic	11000	0.250	0.180	0.0850
2-Methylnaphthalene	4.70	USEPA Region 4 surface water screening levels - freshwater chronic	2.35	0.200	0.100	0.0770
Acenaphthene	1.90	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	0.950	0.200	0.100	0.0640
Acenaphthylene	13.0	USEPA Region 4 surface water screening levels - freshwater chronic	6.50	0.200	0.100	0.0540
Anthracene	0.0200	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0440
Benzo(a)anthracene	0.00120	USEPA NRWQC (Value for Water and Organism)	0.200	0.200	0.100	0.0460
Benzo(a)pyrene	0.000120	USEPA NRWQC (Value for Water and Organism)	0.200	0.200	0.100	0.0660
Benzo(b)fluoranthene	0.00120	USEPA NRWQC (Value for Water and Organism)	0.200	0.200	0.100	0.0890
Benzo(g,h,i)perylene	0.0120	USEPA Region 4 surface water screening levels - freshwater chronic	0.200	0.200	0.100	0.0650
Benzo(k)fluoranthene	0.0120	USEPA NRWQC (Value for Water and Organism)	0.200	0.200	0.100	0.0490
Chrysene	0.0380	RIDEM Ambient Water Quality Criteria (Value for Water and	0.200	0.200	0.100	0.0360
Dibenz(a,h)anthracene	0.000120	USEPA NRWQC (Value for Water and Organism)	0.200	0.200	0.100	0.0700
Fluoranthene	0.800	USEPA Region 4 surface water screening levels - freshwater chronic	0.400	0.200	0.100	0.0730
Fluorene	19.0	USEPA Region 4 surface water screening levels - freshwater chronic	9.50	0.200	0.100	0.0610
Indeno(1,2,3-c,d)pyrene	0.00120	USEPA NRWQC (Value for Water and Organism)	0.200	0.200	0.100	0.0520
Naphthalene	2.60	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.30	0.200	0.100	0.0640
Pentachlorophenol	0.0300	USEPA NRWQC (Value for Water and Organism)	1.00	1.00	0.500	0.330
Phenanthrene	2.30	USEPA Region 4 surface water screening levels - freshwater chronic	1.15	0.200	0.100	0.0510
Pyrene	4.60	USEPA Region 4 surface water screening levels - freshwater chronic	2.30	0.200	0.100	0.0590

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**

Analytical Group: **Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibrium Technique (RSK175 D Dissolved Gases Groundwater)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Ethane			50.0	10.0	5.00	1.96
Ethene			50.0	10.0	5.00	2.19
Methane			50.0	10.0	5.00	1.94

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

Matrix: **Aqueous**

Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C D Metals Groundwater)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	2000	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1000	300	100	15.0
Barium	380	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	190	5.00	3.00	0.230
Calcium			500	100	80.0	11.0
Chromium	100	USEPA MCL March 2018	50.0	10.0	4.00	0.360
Copper	80.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	40.0	25.0	10.0	0.630
Iron	1400	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	700	100	80.0	5.42
Magnesium			500	100	80.0	7.80
Manganese	43.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	21.5	5.00	4.00	1.06
Potassium			5000	1000	500	41.0
Sodium			5000	1000	500	24.0
Vanadium	8.60	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	10.0	10.0	4.00	0.230
Zinc	600	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	300	20.0	10.0	0.730

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C E Metals Porewater)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	87.0	USEPA Region 4 surface water screening levels - freshwater	300	300	100	15.0
Barium	220	USEPA Region 4 surface water screening levels - freshwater	110	5.00	3.00	0.230
Calcium	116000	USEPA Region 4 surface water screening levels - freshwater	58000	100	80.0	11.0
Chromium	42.0	USEPA Region 4 surface water screening levels - freshwater	21.0	10.0	4.00	0.360
Copper	3.10	Federal AWQC - freshwater chronic; hardness of 100 mg/L	25.0	25.0	10.0	0.630
Iron	1000	Federal AWQC - freshwater chronic; hardness of 100 mg/L	500	100	80.0	5.42
Magnesium	82000	USEPA Region 4 surface water screening levels - freshwater	41000	100	80.0	7.80
Manganese	93.0	USEPA Region 4 surface water screening levels - freshwater	46.5	5.00	4.00	1.06
Potassium	53000	USEPA Region 4 surface water screening levels - freshwater	26500	1000	500	41.0
Sodium	680000	USEPA Region 4 surface water screening levels - freshwater	340000	1000	500	24.0
Vanadium	27.0	USEPA Region 4 surface water screening levels - freshwater	13.5	10.0	4.00	0.230
Zinc	66.0	USEPA Region 4 surface water screening levels - freshwater	33.0	20.0	10.0	0.730

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C F Metals Surface Water)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	87.0	USEPA Region 4 surface water screening levels - freshwater chronic	300	300	100	15.0
Barium	220	USEPA Region 4 surface water screening levels - freshwater chronic	110	5.00	3.00	0.230
Calcium	116000	USEPA Region 4 surface water screening levels - freshwater chronic	58000	100	80.0	11.0
Chromium	42.0	USEPA Region 4 surface water screening levels - freshwater chronic	21.0	10.0	4.00	0.360
Copper	3.10	Federal AWQC - freshwater chronic; hardness of 100 mg/L	25.0	25.0	10.0	0.630
Iron	300	RIDEM Ambient Water Quality Criteria (Value for Water and	150	100	80.0	5.42
Magnesium	82000	USEPA Region 4 surface water screening levels - freshwater chronic	41000	100	80.0	7.80
Manganese	50.0	USEPA NRWQC (Value for Water and Organism)	25.0	5.00	4.00	1.06
Potassium	53000	USEPA Region 4 surface water screening levels - freshwater chronic	26500	1000	500	41.0
Sodium	680000	USEPA Region 4 surface water screening levels - freshwater chronic	340000	1000	500	24.0
Vanadium	27.0	USEPA Region 4 surface water screening levels - freshwater chronic	13.5	10.0	4.00	0.230
Zinc	66.0	USEPA Region 4 surface water screening levels - freshwater chronic	33.0	20.0	10.0	0.730

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**

Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A D ICPMS Metals Groundwater)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	0.780	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.0540
Arsenic	0.0520	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	5.00	5.00	4.00	2.20
Beryllium	2.50	USEPA Tapwater RSL Be only (TR=1E-6;THQ=0.1)	1.25	1.00	0.200	0.0340
Cadmium	0.920	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.200	0.0300
Cobalt	0.600	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.300	0.0600
Lead	15.0	USEPA MCL March 2018	7.50	1.00	0.500	0.0740
Nickel	39.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	19.5	2.00	1.20	0.150
Selenium	10.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	5.00	5.00	3.00	0.190
Silver	9.40	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	4.70	1.00	0.400	0.0500
Thallium	0.0200	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.400	0.0600

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A E ICPMS Metals Porewater)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	10.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100	5.00	1.00	0.500	0.0540
Arsenic	150	Federal AWQC - freshwater chronic; hardness of 100 mg/L	75.0	5.00	4.00	2.20
Beryllium	0.17	RIDEM SW values Be only (freshwater chronic) App B; hardness of 100 mg/L	1.00	1.00	0.200	0.0340
Cadmium	0.246	RIDEM SW values (freshwater chronic) App B ; hardness of 100	1.00	1.00	0.200	0.0300
Cobalt	19.0	USEPA Region 4 surface water screening levels - freshwater chronic	9.50	1.00	0.300	0.0600
Lead	1.25	USEPA Region 4 surface water screening levels - freshwater chronic	1.00	1.00	0.500	0.0740
Nickel	28.9	USEPA Region 4 surface water screening levels - freshwater chronic	14.5	2.00	1.20	0.150
Selenium	5.00	USEPA Region 4 surface water screening levels - freshwater chronic	5.00	5.00	3.00	0.190
Silver	0.0600	USEPA Region 4 surface water screening levels - freshwater chronic	1.00	1.00	0.400	0.0500
Thallium	1.00	RIDEM SW values (freshwater chronic) App B ; hardness of 100	1.00	1.00	0.400	0.0600

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A F ICPMS Metals Surface Water)**

Concentration Level (if applicable):

Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	5.60	RIDEM Ambient Water Quality Criteria (Value for Water and	2.80	1.00	0.500	0.0540
Arsenic	0.0180	USEPA NRWQC (Value for Water and Organism)	5.00	5.00	4.00	2.20
Beryllium	0.17	RIDEM SW values Be only (freshwater chronic) App B; hardness of 100	1.00	1.00	0.200	0.0340
Cadmium	0.246	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.00	1.00	0.200	0.0300
Cobalt	19.0	USEPA Region 4 surface water screening levels - freshwater chronic	9.50	1.00	0.300	0.0600
Lead	1.25	USEPA Region 4 surface water screening levels - freshwater chronic	1.00	1.00	0.500	0.0740
Nickel	28.9	USEPA Region 4 surface water screening levels - freshwater chronic	14.5	2.00	1.20	0.150
Selenium	5.00	USEPA Region 4 surface water screening levels - freshwater chronic	5.00	5.00	3.00	0.190
Silver	0.0600	USEPA Region 4 surface water screening levels - freshwater chronic	1.00	1.00	0.400	0.0500
Thallium	0.240	RIDEM Ambient Water Quality Criteria (Value for Water and	1.00	1.00	0.400	0.0600

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.570	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.285	0.200	0.100	0.0130

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

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.770	Federal AWQC - freshwater chronic; hardness of 100 mg/L	0.385	0.200	0.100	0.0130

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

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.140	RIDEM Ambient Water Quality Criteria (Value for Water and Organism)	0.200	0.200	0.100	0.0130

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C D VOCs Groundwater Full Scan)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	200	USEPA MCL March 2018	100	1.00	0.500	0.200
1,1-Dichloroethane	2.80	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.40	1.00	0.500	0.210
1,1-Dichloroethene	7.00	USEPA MCL March 2018	3.50	1.00	0.500	0.350
1,2,3-Trichlorobenzene	0.700	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.270
1,2,4-Trichlorobenzene	0.400	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.370
1,2,4-Trimethylbenzene	5.60	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	2.80	1.00	0.500	0.190
1,2-Dichlorobenzene	30.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	15.0	1.00	0.500	0.150
1,3,5-Trimethylbenzene	6.00	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	3.00	1.00	0.500	0.200
1,3-Dichlorobenzene	600	RIDEM Method 1 Groundwater Objective (GA)	300	1.00	0.500	0.260
2-Butanone (MEK)	560	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	280	5.00	2.50	1.30
4-Methyl-2-pentanone (MIBK)	630	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	315	5.00	2.50	1.30
Acetone	1400	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	700	5.00	2.50	2.20
Benzene	0.460	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.260
Bromodichloromethane	0.130	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.330
Bromomethane	0.750	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	2.00	2.00	1.00	0.490
Carbon disulfide	81.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	40.5	1.00	0.500	0.250
Chloroethane	2100	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1050	2.00	1.00	0.550
Chloroform	0.220	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.320
Chloromethane	19.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	9.50	2.00	1.00	0.360
cis-1,2-Dichloroethene	3.60	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.80	1.00	0.500	0.210
Dibromochloromethane	0.870	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.300
Ethylbenzene	1.50	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.210
Isopropylbenzene (Cumene)	45.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	22.5	1.00	0.500	0.230
m,p-Xylene	19.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	9.50	2.00	1.00	0.590

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Methylene chloride	5.00	USEPA MCL March 2018	5.00	5.00	2.50	1.10
n-Butylbenzene	100	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	50.0	1.00	0.500	0.230
n-Propylbenzene	66.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	33.0	1.00	0.500	0.260
o-Xylene	19.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	9.50	1.00	0.500	0.250
p-Cymene (p-Isopropyltoluene)	45.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1); Value for Isopropylbenzene (Cumene)	5.00	1.00	0.500	0.250
sec-Butylbenzene	200	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	100	1.00	0.500	0.210
tert-Butyl alcohol	200	USEPA Tapwater RSL (TR=1E-6; THQ=0.1); Value for n-Butanol	25.0	5.00	2.50	2.50
tert-Butylbenzene	69.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	34.5	1.00	0.500	0.310
Toluene	110	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	55.0	1.00	0.500	0.270
trans-1,2-Dichloroethene	36.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	18.0	1.00	0.500	0.250
Trichloroethene (TCE)	0.280	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1.00	1.00	0.500	0.280
Vinyl chloride	0.0190	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	2.00	2.00	1.00	0.250

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C E VOCs Porewater Full Scan)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	76.0	USEPA Region 4 surface water screening levels - freshwater chronic	38.0	1.00	0.500	0.200
1,1-Dichloroethane	410	USEPA Region 4 surface water screening levels - freshwater chronic	205	1.00	0.500	0.210
1,1-Dichloroethene	13.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	6.50	1.00	0.500	0.350
1,2,3-Trichlorobenzene	8.00	USEPA Region 4 surface water screening levels - freshwater chronic	4.00	1.00	0.500	0.270
1,2,4-Trichlorobenzene	1.70	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.00	1.00	0.500	0.370
1,2,4-Trimethylbenzene	15.0	USEPA Region 4 surface water screening levels - freshwater chronic	7.50	1.00	0.500	0.190
1,2-Dichlorobenzene	1.80	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.00	1.00	0.500	0.150
1,3,5-Trimethylbenzene	26.0	USEPA Region 4 surface water screening levels - freshwater chronic	13.0	1.00	0.500	0.200
1,3-Dichlorobenzene	8.70	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	4.35	1.00	0.500	0.260
2-Butanone (MEK)	22000	USEPA Region 4 surface water screening levels - freshwater chronic	11000	5.00	2.50	1.30
4-Methyl-2-pentanone (MIBK)	170	USEPA Region 4 surface water screening levels - freshwater chronic	85.0	5.00	2.50	1.30
Acetone	1700	USEPA Region 4 surface water screening levels - freshwater chronic	850	5.00	2.50	2.20
Benzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	2.95	1.00	0.500	0.260
Bromodichloromethane	340	USEPA Region 4 surface water screening levels - freshwater chronic	170	1.00	0.500	0.330
Bromomethane	16.0	USEPA Region 4 surface water screening levels - freshwater chronic	8.00	2.00	1.00	0.490
Carbon disulfide	15.0	USEPA Region 4 surface water screening levels - freshwater chronic	7.50	1.00	0.500	0.250
Chloroethane			10.0	2.00	1.00	0.550
Chloroform	32.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	16.0	1.00	0.500	0.320
Chloromethane			10.0	2.00	1.00	0.360
cis-1,2-Dichloroethene	620	USEPA Region 4 surface water screening levels - freshwater chronic	310	1.00	0.500	0.210
Dibromochloromethane	320	USEPA Region 4 surface water screening levels - freshwater chronic	160	1.00	0.500	0.300
Ethylbenzene	36.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	18.0	1.00	0.500	0.210
Isopropylbenzene (Cumene)	4.80	USEPA Region 4 surface water screening levels - freshwater chronic	2.40	1.00	0.500	0.230
m,p-Xylene	3.00	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	2.00	2.00	1.00	0.590
Methylene chloride	214	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	107	5.00	2.50	1.10
n-Butylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.230
n-Propylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.260
o-Xylene	3.00	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.50	1.00	0.500	0.250

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
p-Cymene (p-Isopropyltoluene)	16.0	USEPA Region 4 surface water screening levels - freshwater chronic	8.00	1.00	0.500	0.250
sec-Butylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.210
tert-Butyl alcohol			25.0	5.00	2.50	2.50
tert-Butylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L.Value for benzene.	5.00	1.00	0.500	0.310
Toluene	14.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	7.00	1.00	0.500	0.270
trans-1,2-Dichloroethene	558	USEPA Region 4 surface water screening levels - freshwater chronic	279	1.00	0.500	0.250
Trichloroethene (TCE)	43.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	21.5	1.00	0.500	0.280
Vinyl chloride	930	USEPA Region 4 surface water screening levels - freshwater chronic	465	2.00	1.00	0.250

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

Matrix: **Aqueous**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C F VOCs Surface Water Full Scan)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	76.0	USEPA Region 4 surface water screening levels - freshwater chronic	38.0	1.00	0.500	0.200
1,1-Dichloroethane	410	USEPA Region 4 surface water screening levels - freshwater chronic	205	1.00	0.500	0.210
1,1-Dichloroethene	13.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	6.50	1.00	0.500	0.350
1,2,3-Trichlorobenzene	8.00	USEPA Region 4 surface water screening levels - freshwater chronic	4.00	1.00	0.500	0.270
1,2,4-Trichlorobenzene	0.0710	USEPA NRWQC (Value for Water and Organism)	1.00	1.00	0.500	0.370
1,2,4-Trimethylbenzene	15.0	USEPA Region 4 surface water screening levels - freshwater chronic	7.50	1.00	0.500	0.190
1,2-Dichlorobenzene	1.80	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.00	1.00	0.500	0.150
1,3,5-Trimethylbenzene	26.0	USEPA Region 4 surface water screening levels - freshwater chronic	13.0	1.00	0.500	0.200
1,3-Dichlorobenzene	7.00	USEPA NRWQC (Value for Water and Organism)	3.50	1.00	0.500	0.260
2-Butanone (MEK)	22000	USEPA Region 4 surface water screening levels - freshwater chronic	11000	5.00	2.50	1.30
4-Methyl-2-pentanone (MIBK)	170	USEPA Region 4 surface water screening levels - freshwater chronic	85.0	5.00	2.50	1.30
Acetone	1700	USEPA Region 4 surface water screening levels - freshwater chronic	850	5.00	2.50	2.20
Benzene	0.580	USEPA NRWQC (Value for Water and Organism)	1.00	1.00	0.500	0.260
Bromodichloromethane	0.950	USEPA NRWQC (Value for Water and Organism)	1.00	1.00	0.500	0.330
Bromomethane	16.0	USEPA Region 4 surface water screening levels - freshwater chronic	8.00	2.00	1.00	0.490
Carbon disulfide	15.0	USEPA Region 4 surface water screening levels - freshwater chronic	7.50	1.00	0.500	0.250
Chloroethane	2100	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1050	2.00	1.00	0.550
Chloroform	32.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	16.0	1.00	0.500	0.320
Chloromethane	19.0	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	9.5	2.00	1.00	0.360
cis-1,2-Dichloroethene	620	USEPA Region 4 surface water screening levels - freshwater chronic	310	1.00	0.500	0.210
Dibromochloromethane	0.800	USEPA NRWQC (Value for Water and Organism)	1.00	1.00	0.500	0.300
Ethylbenzene	36.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	18.0	1.00	0.500	0.210
Isopropylbenzene (Cumene)	4.80	USEPA Region 4 surface water screening levels - freshwater chronic	2.40	1.00	0.500	0.230
m,p-Xylene	3.00	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	2.00	2.00	1.00	0.590
Methylene chloride	20.0	USEPA NRWQC (Value for Water and Organism)	10.0	5.00	2.50	1.10
n-Butylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.230
n-Propylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.260
o-Xylene	3.00	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	1.50	1.00	0.500	0.250

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
p-Cymene (p-Isopropyltoluene)	16.0	USEPA Region 4 surface water screening levels - freshwater chronic	8.00	1.00	0.500	0.250
sec-Butylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.210
tert-Butyl alcohol	200	USEPA Tapwater RSL (TR=1E-6; THQ=0.1); Value for n-Butanol	100	5.00	2.50	2.50
tert-Butylbenzene	5.9	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L. Value for benzene.	5.00	1.00	0.500	0.310
Toluene	14.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	7.00	1.00	0.500	0.270
trans-1,2-Dichloroethene	100	USEPA NRWQC (Value for Water and Organism)	50.0	1.00	0.500	0.250
Trichloroethene (TCE)	0.600	USEPA NRWQC (Value for Water and Organism)	1.00	1.00	0.500	0.280
Vinyl chloride	0.0220	USEPA NRWQC (Value for Water and Organism)	2.00	2.00	1.00	0.250

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Anion Chromatography (SW9056A D Anions)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Chloride			10000	2000	1000	99.3
Sulfate			5000	1000	500	63.7
Nitrate (as N)	3200	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	1600	50.0	25.0	17.4
Nitrite (as N)	200	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	100	50.0	25.0	9.22

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

Matrix: **Aqueous**
Analytical Group: **Method SW8260C Selected Ion Monitoring (SIM) Mode (VOCSIMC D VOCs SIM Groundwater)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,2,3-Trichlorobenzene	0.700	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	TBD	TBD	TBD	TBD
1,2,4-Trichlorobenzene	0.400	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.200	0.0500	0.0250	0.00260
Benzene	0.460	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.230	0.0500	0.0250	0.00560
Chloroform	0.220	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.110	0.0500	0.0250	0.00720
Trichloroethene (TCE)	0.280	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.140	0.0500	0.0250	0.0880
Vinyl chloride	0.0190	USEPA Tapwater RSL (TR=1E-6; THQ=0.1)	0.100	0.100	0.0500	0.00370

TBD - to be determined; pending method development.
Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Aqueous**
Analytical Group: **Method SW8260C Selected Ion Monitoring (SIM) Mode (VOCSIMC E VOCs SIM Porewater)**
Concentration Level (if applicable): Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,2,3-Trichlorobenzene	8.00	USEPA Region 4 surface water screening levels - freshwater chronic	TBD	TBD	TBD	TBD
1,2,4-Trichlorobenzene	1.70	RIDEM SW values (freshwater chronic) App B ; hardness of 100	0.850	0.0500	0.0250	0.00260
Benzene	5.90	RIDEM SW values (freshwater chronic) App B ; hardness of 100	2.95	0.0500	0.0250	0.00560
Chloroform	32.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100	16.0	0.0500	0.0250	0.00720
Trichloroethene (TCE)	43.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100	21.5	0.0500	0.0250	0.0880
Vinyl chloride	930	USEPA Region 4 surface water screening levels - freshwater chronic	465	0.100	0.0500	0.00370

TBD - to be determined; pending method development.

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

Matrix: **Aqueous**
Analytical Group: **Method SW8260C Selected Ion Monitoring (SIM) Mode (VOCSIMC F VOCs SIM Surface Water)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,2,3-Trichlorobenzene	8.00	USEPA Region 4 surface water screening levels - freshwater chronic	TBD	TBD	TBD	TBD
1,2,4-Trichlorobenzene	0.0710	USEPA NRWQC (Value for Water and Organism)	0.0500	0.0500	0.0250	0.00260
Benzene	0.580	USEPA NRWQC (Value for Water and Organism)	0.290	0.0500	0.0250	0.00560
Chloroform	32.0	RIDEM SW values (freshwater chronic) App B ; hardness of 100 mg/L	16.0	0.0500	0.0250	0.00720
Trichloroethene (TCE)	0.600	USEPA NRWQC (Value for Water and Organism)	0.300	0.0500	0.0250	0.0880
Vinyl chloride	0.0220	USEPA NRWQC (Value for Water and Organism)	0.100	0.100	0.0500	0.00370

TBD - to be determined; pending method development.
Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **EPA Draft Method for the Determination of Acid Volatile Sulfide and Simultaneously Extractable**
Concentration Level (if applicable):
Units: **µmoles/g**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Sulfide			0.500	0.100	0.0760	0.0380

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

Matrix: **Solid**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM A SVOC SIM Sediment)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	5.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2.65	0.100	0.0500	0.00112
2-Methylnaphthalene	0.0202	USEPA Region 4 sediment screening levels - freshwater	0.0200	0.0200	0.0100	0.00221
Acenaphthene	0.00670	USEPA Region 4 sediment screening levels - freshwater	0.0200	0.0200	0.0100	0.00145
Acenaphthylene	0.00590	USEPA Region 4 sediment screening levels - freshwater	0.0200	0.0200	0.0100	0.00122
Anthracene	0.0570	USEPA Region 4 sediment screening levels - freshwater	0.0285	0.0200	0.0100	0.00124
Benzo(a)anthracene	0.108	USEPA Region 4 sediment screening levels - freshwater	0.0540	0.0200	0.0100	0.00193
Benzo(a)pyrene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00329
Benzo(b)fluoranthene	0.190	USEPA Region 4 sediment screening levels - freshwater	0.0950	0.0200	0.0100	0.00244
Benzo(g,h,i)perylene	0.170	USEPA Region 4 sediment screening levels - freshwater	0.0850	0.0200	0.0100	0.00195
Benzo(k)fluoranthene	0.240	USEPA Region 4 sediment screening levels - freshwater	0.120	0.0200	0.0100	0.00310
Chrysene	0.166	USEPA Region 4 sediment screening levels - freshwater	0.0830	0.0200	0.0100	0.00173
Dibenz(a,h)anthracene	0.0330	USEPA Region 4 sediment screening levels - freshwater	0.0200	0.0200	0.0100	0.00184
Fluoranthene	0.423	USEPA Region 4 sediment screening levels - freshwater	0.212	0.0200	0.0100	0.00176
Fluorene	0.0770	USEPA Region 4 sediment screening levels - freshwater	0.0385	0.0200	0.0100	0.00315
Indeno(1,2,3-c,d)pyrene	0.200	USEPA Region 4 sediment screening levels - freshwater	0.100	0.0200	0.0100	0.00185
Naphthalene	0.176	USEPA Region 4 sediment screening levels - freshwater	0.0880	0.0200	0.0100	0.00257
Pentachlorophenol	0.0100	USEPA Region 4 sediment screening levels - freshwater	0.100	0.100	0.0500	0.0140
Phenanthrene	0.204	USEPA Region 4 sediment screening levels - freshwater	0.102	0.0200	0.0100	0.00177
Pyrene	0.195	USEPA Region 4 sediment screening levels - freshwater	0.0975	0.0200	0.0100	0.00210

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM B SVOC SIM Surface Soil)***
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	5.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2.65	0.100	0.0500	0.00112
2-Methylnaphthalene	24.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	12.0	0.0200	0.0100	0.00221
Acenaphthene	29.0	USEPA Eco-SSLs - lowest value	14.5	0.0200	0.0100	0.00145
Acenaphthylene	23.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	11.5	0.0200	0.0100	0.00122
Anthracene	29.0	USEPA Eco-SSLs - lowest value	14.5	0.0200	0.0100	0.00124
Benzo(a)anthracene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00193
Benzo(a)pyrene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00329
Benzo(b)fluoranthene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00244
Benzo(g,h,i)perylene	0.800	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.400	0.0200	0.0100	0.00195
Benzo(k)fluoranthene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00310
Chrysene	0.400	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.200	0.0200	0.0100	0.00173
Dibenz(a,h)anthracene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00184
Fluoranthene	1.10	USEPA Eco-SSLs - lowest value	0.550	0.0200	0.0100	0.00176
Fluorene	28.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	14.0	0.0200	0.0100	0.00315
Indeno(1,2,3-c,d)pyrene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00185
Naphthalene	0.800	RIDEM Method 1 Leachability Criteria (GA)	0.400	0.0200	0.0100	0.00257
Pentachlorophenol	1.00	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.100	0.0500	0.0140
Phenanthrene	29.0	USEPA Eco-SSLs - lowest value	14.5	0.0200	0.0100	0.00177
Pyrene	1.10	USEPA Eco-SSLs - lowest value	0.550	0.0200	0.0100	0.00210

* - The analyte list of SVOCs excluding PAHs consists of pentachlorophenol and 1,4-dioxane.

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

Matrix: **Solid**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM C SVOC SIM Subsurface Soil)***
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	5.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2.65	0.100	0.0500	0.00112
2-Methylnaphthalene	24.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	12.0	0.0200	0.0100	0.00221
Acenaphthene	43.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	21.5	0.0200	0.0100	0.00145
Acenaphthylene	23.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	11.5	0.0200	0.0100	0.00122
Anthracene	35.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	17.5	0.0200	0.0100	0.00124
Benzo(a)anthracene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00193
Benzo(a)pyrene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00329
Benzo(b)fluoranthene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00244
Benzo(g,h,i)perylene	0.800	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.400	0.0200	0.0100	0.00195
Benzo(k)fluoranthene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00310
Chrysene	0.400	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.200	0.0200	0.0100	0.00173
Dibenz(a,h)anthracene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00184
Fluoranthene	20.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	10.0	0.0200	0.0100	0.00176
Fluorene	28.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	14.0	0.0200	0.0100	0.00315
Indeno(1,2,3-c,d)pyrene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.450	0.0200	0.0100	0.00185
Naphthalene	0.800	RIDEM Method 1 Leachability Criteria (GA)	0.400	0.0200	0.0100	0.00257
Pentachlorophenol	1.00	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.100	0.0500	0.0140
Phenanthrene	40.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	20.0	0.0200	0.0100	0.00177
Pyrene	13.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	6.50	0.0200	0.0100	0.00210

* - The analyte list of SVOCs excluding PAHs consists of pentachlorophenol and 1,4-dioxane.

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

Matrix: **Solid**
Analytical Group: **GC/MS-SIM Analysis by SW8270 (BNASIM G SVOC SIM Background Soil)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,4-Dioxane (p-Dioxane)	5.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2.65	0.100	0.0500	0.00112
2-Methylnaphthalene	24.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	12.0	0.0200	0.0100	0.00221
Acenaphthene	29.0	USEPA Eco-SSLs - lowest value	14.5	0.0200	0.0100	0.00145
Acenaphthylene	23.0	RIDEM Method 1 Direct Exposure Criteria (Residential	11.5	0.0200	0.0100	0.00122
Anthracene	29.0	USEPA Eco-SSLs - lowest value	14.5	0.0200	0.0100	0.00124
Benzo(a)anthracene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential	0.450	0.0200	0.0100	0.00193
Benzo(a)pyrene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00329
Benzo(b)fluoranthene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential	0.450	0.0200	0.0100	0.00244
Benzo(g,h,i)perylene	0.800	RIDEM Method 1 Direct Exposure Criteria (Residential	0.400	0.0200	0.0100	0.00195
Benzo(k)fluoranthene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential	0.450	0.0200	0.0100	0.00310
Chrysene	0.400	RIDEM Method 1 Direct Exposure Criteria (Residential	0.200	0.0200	0.0100	0.00173
Dibenz(a,h)anthracene	0.110	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.0550	0.0200	0.0100	0.00184
Fluoranthene	1.10	USEPA Eco-SSLs - lowest value	0.550	0.0200	0.0100	0.00176
Fluorene	28.0	RIDEM Method 1 Direct Exposure Criteria (Residential	14.0	0.0200	0.0100	0.00315
Indeno(1,2,3-c,d)pyrene	0.900	RIDEM Method 1 Direct Exposure Criteria (Residential	0.450	0.0200	0.0100	0.00185
Naphthalene	0.800	RIDEM Method 1 Leachability Criteria (GA)	0.400	0.0200	0.0100	0.00257
Pentachlorophenol	1.00	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.100	0.0500	0.0140
Phenanthrene	29.0	USEPA Eco-SSLs - lowest value	14.5	0.0200	0.0100	0.00177
Pyrene	1.10	USEPA Eco-SSLs - lowest value	0.550	0.0200	0.0100	0.00210

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C A Metals Sediment)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	7700	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3850	30.0	10.0	0.710
Barium	20.0	USEPA Region 4 sediment screening levels - freshwater	10.0	0.500	0.300	0.0260
Calcium			50.0	10.0	8.00	1.80
Chromium	43.4	USEPA Region 4 sediment screening levels - freshwater	21.7	1.50	0.400	0.0260
Copper	31.6	USEPA Region 4 sediment screening levels - freshwater	15.8	2.50	1.00	0.160
Iron	5500	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2750	10.0	8.00	1.40
Lead	35.8	USEPA Region 4 sediment screening levels - freshwater	17.9	0.500	0.400	0.0900
Magnesium			50.0	10.0	8.00	0.680
Manganese	180	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	90.0	0.500	0.400	0.160
Nickel	22.7	USEPA Region 4 sediment screening levels - freshwater	11.4	4.00	0.400	0.0440
Potassium			500	100	50.0	2.90
Silver	1.00	USEPA Region 4 sediment screening levels - freshwater	1.50	1.50	0.400	0.0270
Sodium			500	100	50.0	1.50
Vanadium	39.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	19.5	2.50	0.400	0.0370
Zinc	121	USEPA Region 4 sediment screening levels - freshwater	60.5	2.50	1.00	0.200

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C B Metals Surface Soil)**

Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	7700	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3850	30.0	10.0	0.710
Barium	110	USEPA Region 4 soil screening levels - lowest value	55.0	0.500	0.300	0.0260
Calcium			50.0	10.0	8.00	1.80
Chromium	23.0	USEPA Region 4 soil screening levels - lowest value	11.5	1.50	0.400	0.0260
Copper	28.0	USEPA Eco-SSLs - lowest value	14.0	2.50	1.00	0.160
Iron	5500	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2750	10.0	8.00	1.40
Lead	11.0	USEPA Eco-SSLs - lowest value	5.50	0.500	0.400	0.0900
Magnesium			50.0	10.0	8.00	0.680
Manganese	180	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	90.0	0.500	0.400	0.160
Nickel	38.0	USEPA Eco-SSLs - lowest value	19.0	4.00	0.400	0.0440
Potassium			500	100	50.0	2.90
Silver	4.20	USEPA Eco-SSLs - lowest value	2.10	1.50	0.400	0.0270
Sodium			500	100	50.0	1.50
Vanadium	7.80	USEPA Eco-SSLs - lowest value	3.90	2.50	0.400	0.0370
Zinc	46.0	USEPA Eco-SSLs - lowest value	23.0	2.50	1.00	0.200

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C C Metals Subsurface Soil)**
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	7700	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3850	30.0	10.0	0.710
Barium	1500	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	750	0.500	0.300	0.0260
Calcium			50.0	10.0	8.00	1.80
Chromium	1400	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	700	1.50	0.400	0.0260
Copper	310	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	155	2.50	1.00	0.160
Iron	5500	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2750	10.0	8.00	1.40
Lead	150	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	75.0	0.500	0.400	0.0900
Magnesium			50.0	10.0	8.00	0.680
Manganese	180	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	90.0	0.500	0.400	0.160
Nickel	150	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	75.0	4.00	0.400	0.0440
Potassium			500	100	50.0	2.90
Silver	39.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	19.5	1.50	0.400	0.0270
Sodium			500	100	50.0	1.50
Vanadium	39.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	19.5	2.50	0.400	0.0370
Zinc	2300	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1150	2.50	1.00	0.200

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C G Metals Background Soil)**
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Aluminum	7700	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3850	30.0	10.0	0.710
Barium	110	USEPA Region 4 soil screening levels - lowest value	55.0	0.500	0.300	0.0260
Calcium			50.0	10.0	8.00	1.80
Chromium	23.0	USEPA Region 4 soil screening levels - lowest value	11.5	1.50	0.400	0.0260
Copper	28.0	USEPA Eco-SSLs - lowest value	14.0	2.50	1.00	0.160
Iron	5500	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2750	10.0	8.00	1.40
Lead	11.0	USEPA Eco-SSLs - lowest value	5.50	0.500	0.400	0.0900
Magnesium			50.0	10.0	8.00	0.680
Manganese	180	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	90.0	0.500	0.400	0.160
Nickel	38.0	USEPA Eco-SSLs - lowest value	19.0	4.00	0.400	0.0440
Potassium			500	100	50.0	2.90
Silver	4.20	USEPA Eco-SSLs - lowest value	2.10	1.50	0.400	0.0270
Sodium			500	100	50.0	1.50
Vanadium	7.80	USEPA Eco-SSLs - lowest value	3.90	2.50	0.400	0.0370
Zinc	46.0	USEPA Eco-SSLs - lowest value	23.0	2.50	1.00	0.200

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C SPLP ICP Metals)**
Concentration Level (if applicable):
Units: **µg/L**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	50.0	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	40.0	40.0	25.0	6.40
Barium	23000	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	11500	25.0	15.0	1.20
Cadmium	30.0	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	25.0	25.0	15.0	0.240
Chromium	1100	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	550	50.0	20.0	1.80
Lead	40.0	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	25.0	25.0	20.0	5.40
Nickel	1000	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	500	50.0	20.0	1.40
Selenium	600	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	300	50.0	35.0	12.0
Thallium	5.00	RIDEM Method 1 Leachability Criteria (GA) (TCLP/SPLP)	75.0	75.0	25.0	5.40

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**

Analytical Group: **Trace Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (SW6010C AVS/SEM Metals)**

Concentration Level (if applicable):

Units: **µmoles/g**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Cadmium			0.00445	0.000890	0.000530	0.0000140
Copper			0.0393	0.00787	0.00315	0.000730
Lead			0.00242	0.000483	0.000386	0.000150
Nickel			0.0171	0.00341	0.00136	0.000220
Silver			0.00925	0.00185	0.000742	0.000100
Zinc			0.0306	0.00612	0.00306	0.00110

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A A ICPMS Metals Sediment)**
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	2.00	USEPA Region 4 sediment screening levels - freshwater	1.00	0.100	0.0500	0.0200
Arsenic	0.680	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.500	0.400	0.150
Beryllium	1.50	RIDEM Method 1 Direct Exposure Criteria Be only (Residential Soil)	0.750	0.100	0.0200	0.00410
Cadmium	1.00	USEPA Region 4 sediment screening levels - freshwater	0.500	0.100	0.0200	0.00760
Cobalt	2.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.15	0.100	0.0300	0.00540
Selenium	0.800	USEPA Region 4 sediment screening levels - freshwater	0.500	0.500	0.300	0.0390
Thallium	0.0780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.100	0.100	0.0400	0.00940

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A B ICPMS Metals Surface Soil)**
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	0.270	USEPA Eco-SSLs - lowest value	0.135	0.100	0.0500	0.0200
Arsenic	0.680	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.500	0.400	0.150
Beryllium	1.50	RIDEM Method 1 Direct Exposure Criteria Be only (Residential Soil)	0.750	0.100	0.0200	0.00410
Cadmium	0.360	USEPA Eco-SSLs - lowest value	0.180	0.100	0.0200	0.00760
Cobalt	2.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.15	0.100	0.0300	0.00540
Selenium	0.520	USEPA Eco-SSLs - lowest value	0.500	0.500	0.300	0.0390
Thallium	0.0500	USEPA Region 4 soil screening levels - lowest value	0.100	0.100	0.0400	0.00940

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**

Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A C ICPMS Metals Subsurface Soil)**

Concentration Level (if applicable):

Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	3.10	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.55	0.100	0.0500	0.0200
Arsenic	0.680	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.500	0.400	0.150
Beryllium	1.50	RIDEM Method 1 Direct Exposure Criteria Be only (Residential Soil)	0.750	0.100	0.0200	0.00410
Cadmium	7.10	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3.55	0.100	0.0200	0.00760
Cobalt	2.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.15	0.100	0.0300	0.00540
Selenium	39.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	19.5	0.500	0.300	0.0390
Thallium	0.0780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.100	0.100	0.0400	0.00940

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Trace Metals by Inductively Coupled Plasma/Mass Spectrometry (SW6020A G ICPMS Metals Background Soil)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Antimony	0.270	USEPA Eco-SSLs - lowest value	0.135	0.100	0.0500	0.0200
Arsenic	0.680	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.500	0.500	0.400	0.150
Beryllium	1.50	RIDEM Method 1 Direct Exposure Criteria Be only (Residential Soil)	0.750	0.100	0.0200	0.00410
Cadmium	0.360	USEPA Eco-SSLs - lowest value	0.180	0.100	0.0200	0.00760
Cobalt	2.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.15	0.100	0.0300	0.00540
Selenium	0.520	USEPA Eco-SSLs - lowest value	0.500	0.500	0.300	0.0390
Thallium	0.0500	USEPA Region 4 soil screening levels - lowest value	0.100	0.100	0.0400	0.00940

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	20.0	RIDEM Method 1 Leachability Criteria (GA)	10.0	10.0	1.00	0.500

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

Matrix: **Solid**
Analytical Group: **Mercury in Soil (Manual Cold-Vapor Technique) (SW7471B A Mercury Sediment)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.170	USEPA Region 4 sediment screening levels - freshwater	0.0850	0.0300	0.0200	0.00520

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

Matrix: **Solid**
Analytical Group: **Mercury in Soil (Manual Cold-Vapor Technique) (SW7471B B Mercury Surface Soil)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.0130	USEPA Region 4 soil screening levels - lowest value	0.0300	0.0300	0.0200	0.00520

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Mercury in Soil (Manual Cold-Vapor Technique) (SW7471B C Mercury Subsurface Soil)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	2.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.15	0.0300	0.0200	0.00520

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

Matrix: **Solid**
Analytical Group: **Mercury in Soil (Manual Cold-Vapor Technique) (SW7471B G Mercury Background Soil)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Mercury	0.0130	USEPA Region 4 soil screening levels - lowest value	0.0300	0.0300	0.0200	0.00520

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C A VOCs Sediment Full Scan)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	0.0700	USEPA Region 4 sediment screening levels - freshwater	0.0350	0.00500	0.00250	0.000400
1,1-Dichloroethane	0.0200	USEPA Region 4 sediment screening levels - freshwater	0.0100	0.00500	0.00250	0.00170
1,1-Dichloroethene	0.100	USEPA Region 4 sediment screening levels - freshwater	0.0500	0.00500	0.00250	0.000900
1,2,3-Trichlorobenzene	0.113	USEPA Region 4 sediment screening levels - freshwater	0.0565	0.00500	0.00250	0.000800
1,2,4-Trichlorobenzene	0.0110	USEPA Region 4 sediment screening levels - freshwater	0.00550	0.00500	0.00250	0.000800
1,2,4-Trimethylbenzene	0.0970	USEPA Region 4 sediment screening levels - freshwater	0.0485	0.00500	0.00250	0.000900
1,2-Dichlorobenzene	0.0950	USEPA Region 4 sediment screening levels - freshwater	0.0475	0.00500	0.00250	0.000800
1,3,5-Trimethylbenzene	0.164	USEPA Region 4 sediment screening levels - freshwater	0.0820	0.00500	0.00250	0.000700
1,3-Dichlorobenzene	0.0890	USEPA Region 4 sediment screening levels - freshwater	0.0445	0.00500	0.00250	0.000600
2-Butanone (MEK)	7.60	USEPA Region 4 sediment screening levels - freshwater	3.80	0.0250	0.0125	0.00590
4-Methyl-2-pentanone (MIBK)	0.0730	USEPA Region 4 sediment screening levels - freshwater	0.0365	0.0250	0.0125	0.00590
Acetone	0.0650	USEPA Region 4 sediment screening levels - freshwater	0.0325	0.0250	0.0125	0.00510
Benzene	0.0100	USEPA Region 4 sediment screening levels - freshwater	0.00500	0.00500	0.00250	0.000900
Bromodichloromethane	0.210	USEPA Region 4 sediment screening levels - freshwater	0.105	0.00500	0.00250	0.000600
Bromomethane	0.00650	USEPA Region 4 sediment screening levels - freshwater	0.0100	0.0100	0.00500	0.00110
Carbon disulfide	0.00780	USEPA Region 4 sediment screening levels - freshwater	0.00500	0.00500	0.00250	0.000800
Chloroethane	1400	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	700	0.0100	0.00500	0.00130
Chloroform	0.0870	USEPA Region 4 sediment screening levels - freshwater	0.0435	0.00500	0.00250	0.000400
Chloromethane	11.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	5.50	0.0100	0.00500	0.00140
cis-1,2-Dichloroethene	0.432	USEPA Region 4 sediment screening levels - freshwater	0.216	0.00500	0.00250	0.000900
Dibromochloromethane	0.198	USEPA Region 4 sediment screening levels - freshwater	0.0990	0.00500	0.00250	0.00100
Ethylbenzene	0.290	USEPA Region 4 sediment screening levels - freshwater	0.145	0.00500	0.00250	0.000700
Isopropylbenzene (Cumene)	0.0350	USEPA Region 4 sediment screening levels - freshwater	0.0175	0.00500	0.00250	0.000900
m,p-Xylene	0.130	USEPA Region 4 sediment screening levels - freshwater	0.0650	0.0100	0.00500	0.00170

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Methylene chloride	0.0180	USEPA Region 4 sediment screening levels - freshwater	0.0250	0.0250	0.0125	0.00790
n-Butylbenzene	390	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	195	0.00500	0.00250	0.000900
n-Propylbenzene	380	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	190	0.00500	0.00250	0.000800
o-Xylene	0.130	USEPA Region 4 sediment screening levels - freshwater	0.0650	0.00500	0.00250	0.00130
p-Cymene (p-Isopropyltoluene)	0.184	USEPA Region 4 sediment screening levels - freshwater	0.0920	0.00500	0.00250	0.000800
sec-Butylbenzene	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	390	0.00500	0.00250	0.000900
tert-Butyl alcohol	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1); Value for n-Butanol	0.125	0.0250	0.0125	0.0109
tert-Butylbenzene	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	390	0.00500	0.00250	0.000900
Toluene	0.0100	USEPA Region 4 sediment screening levels - freshwater	0.00500	0.00500	0.00250	0.00140
trans-1,2-Dichloroethene	0.389	USEPA Region 4 sediment screening levels - freshwater	0.195	0.00500	0.00250	0.000700
Trichloroethene (TCE)	0.0780	USEPA Region 4 sediment screening levels - freshwater	0.0390	0.00500	0.00250	0.000600
Vinyl chloride	0.0200	RIDEM Method 1 Direct Exposure Criteria (Residential Soil)	0.0100	0.0100	0.00500	0.000900

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C B VOCs Surface Soil Full Scan)**
Concentration Level (if applicable): Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	0.0400	USEPA Region 4 soil screening levels - lowest value	0.0200	0.00500	0.00250	0.000400
1,1-Dichloroethane	0.140	USEPA Region 4 soil screening levels - lowest value	0.0700	0.00500	0.00250	0.00170
1,1-Dichloroethene	0.0400	USEPA Region 4 soil screening levels - lowest value	0.0200	0.00500	0.00250	0.000900
1,2,3-Trichlorobenzene	6.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3.15	0.00500	0.00250	0.000800
1,2,4-Trichlorobenzene	0.270	USEPA Region 4 soil screening levels - lowest value	0.135	0.00500	0.00250	0.000800
1,2,4-Trimethylbenzene	0.0900	USEPA Region 4 soil screening levels - lowest value	0.0450	0.00500	0.00250	0.000900
1,2-Dichlorobenzene	0.0900	USEPA Region 4 soil screening levels - lowest value	0.0450	0.00500	0.00250	0.000800
1,3,5-Trimethylbenzene	0.160	USEPA Region 4 soil screening levels - lowest value	0.0800	0.00500	0.00250	0.000700
1,3-Dichlorobenzene	0.0800	USEPA Region 4 soil screening levels - lowest value	0.0400	0.00500	0.00250	0.000600
2-Butanone (MEK)	1.00	USEPA Region 4 soil screening levels - lowest value	0.500	0.0250	0.0125	0.00590
4-Methyl-2-pentanone (MIBK)	3300	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1650	0.0250	0.0125	0.00590
Acetone	0.0400	USEPA Region 4 soil screening levels - lowest value	0.0250	0.0250	0.0125	0.00510
Benzene	0.120	USEPA Region 4 soil screening levels - lowest value	0.0600	0.00500	0.00250	0.000900
Bromodichloromethane	0.290	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.145	0.00500	0.00250	0.000600
Bromomethane	0.00200	USEPA Region 4 soil screening levels - lowest value	0.0100	0.0100	0.00500	0.00110
Carbon disulfide	0.00500	USEPA Region 4 soil screening levels - lowest value	0.00500	0.00500	0.00250	0.000800
Chloroethane	1400	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	700	0.0100	0.00500	0.00130
Chloroform	0.0500	USEPA Region 4 soil screening levels - lowest value	0.0250	0.00500	0.00250	0.000400
Chloromethane	11.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	5.50	0.0100	0.00500	0.00140
cis-1,2-Dichloroethene	0.0400	USEPA Region 4 soil screening levels - lowest value	0.0200	0.00500	0.00250	0.000900
Dibromochloromethane	7.60	RIDEM Method 1 Direct Exposure Criteria	3.80	0.00500	0.00250	0.00100
Ethylbenzene	0.270	USEPA Region 4 soil screening levels - lowest value	0.135	0.00500	0.00250	0.000700
Isopropylbenzene (Cumene)	0.0400	USEPA Region 4 soil screening levels - lowest value	0.0200	0.00500	0.00250	0.000900
m,p-Xylene	0.100	USEPA Region 4 soil screening levels - lowest value	0.0500	0.0100	0.00500	0.00170
Methylene chloride	0.210	USEPA Region 4 soil screening levels - lowest value	0.105	0.0250	0.0125	0.00790
n-Butylbenzene	390	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	195	0.00500	0.00250	0.000900

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
n-Propylbenzene	380	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	190	0.00500	0.00250	0.000800
o-Xylene	0.100	USEPA Region 4 soil screening levels - lowest value	0.0500	0.00500	0.00250	0.00130
p-Cymene (p-Isopropyltoluene)	0.180	USEPA Region 4 soil screening levels - lowest value	0.0900	0.00500	0.00250	0.000800
sec-Butylbenzene	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	390	0.00500	0.00250	0.000900
tert-Butyl alcohol	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1); Value for n-Butanol	0.125	0.0250	0.0125	0.0109
tert-Butylbenzene	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	390	0.00500	0.00250	0.000900
Toluene	0.150	USEPA Region 4 soil screening levels - lowest value	0.0750	0.00500	0.00250	0.00140
trans-1,2-Dichloroethene	0.0400	USEPA Region 4 soil screening levels - lowest value	0.0200	0.00500	0.00250	0.000700
Trichloroethene (TCE)	0.0600	USEPA Region 4 soil screening levels - lowest value	0.0300	0.00500	0.00250	0.000600
Vinyl chloride	0.0200	RIDEM Method 1 Direct Exposure Criteria	0.0100	0.0100	0.00500	0.000900

Highlighting indicates the specified limit is greater than the Project Action Limit for the associated analyte.

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

Matrix: **Solid**
Analytical Group: **Volatile Organic Compounds by GC/MS (SW8260C C VOCs Subsurface Soil Full Scan)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
1,1,1-Trichloroethane	11.0	RIDEM Method 1 Leachability Criteria (GA)	5.50	0.00500	0.00250	0.000400
1,1-Dichloroethane	3.60	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1.80	0.00500	0.00250	0.00170
1,1-Dichloroethene	0.200	RIDEM Method 1 Direct Exposure Criteria	0.100	0.00500	0.00250	0.000900
1,2,3-Trichlorobenzene	6.30	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3.15	0.00500	0.00250	0.000800
1,2,4-Trichlorobenzene	5.80	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2.90	0.00500	0.00250	0.000800
1,2,4-Trimethylbenzene	30.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	15.0	0.00500	0.00250	0.000900
1,2-Dichlorobenzene	41.0	RIDEM Method 1 Leachability Criteria (GA)	20.5	0.00500	0.00250	0.000800
1,3,5-Trimethylbenzene	27.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	13.5	0.00500	0.00250	0.000700
1,3-Dichlorobenzene	41.0	RIDEM Method 1 Leachability Criteria (GA)	20.5	0.00500	0.00250	0.000600
2-Butanone (MEK)	2700	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1350	0.0250	0.0125	0.00590
4-Methyl-2-pentanone (MIBK)	3300	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	1650	0.0250	0.0125	0.00590
Acetone	6100	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	3050	0.0250	0.0125	0.00510
Benzene	0.200	RIDEM Method 1 Leachability Criteria (GA)	0.100	0.00500	0.00250	0.000900
Bromodichloromethane	0.290	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.145	0.00500	0.00250	0.000600
Bromomethane	0.680	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.340	0.0100	0.00500	0.00110
Carbon disulfide	77.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	38.5	0.00500	0.00250	0.000800
Chloroethane	1400	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	700	0.0100	0.00500	0.00130
Chloroform	0.320	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	0.160	0.00500	0.00250	0.000400
Chloromethane	11.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	5.50	0.0100	0.00500	0.00140
cis-1,2-Dichloroethene	16.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	8.00	0.00500	0.00250	0.000900
Dibromochloromethane	7.60	RIDEM Method 1 Direct Exposure Criteria	3.80	0.00500	0.00250	0.00100
Ethylbenzene	5.80	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	2.90	0.00500	0.00250	0.000700
Isopropylbenzene (Cumene)	27.0	RIDEM Method 1 Direct Exposure Criteria	13.5	0.00500	0.00250	0.000900
m,p-Xylene	55.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	27.5	0.0100	0.00500	0.00170

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

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Methylene chloride	35.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	17.5	0.0250	0.0125	0.00790
n-Butylbenzene	390	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	195	0.00500	0.00250	0.000900
n-Propylbenzene	380	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	190	0.00500	0.00250	0.000800
o-Xylene	65.0	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	32.5	0.00500	0.00250	0.00130
p-Cymene (p-Isopropyltoluene)	27.0	RIDEM Method 1 Direct Exposure Criteria (Residential Soil); Value for Isopropylbenzene (Cumene)	0.0250	0.00500	0.00250	0.000800
sec-Butylbenzene	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	390	0.00500	0.00250	0.000900
tert-Butyl alcohol	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1); Value for n-Butanol	0.125	0.0250	0.0125	0.0109
tert-Butylbenzene	780	USEPA Res Soil RSL (TR=1E-6; THQ=0.1)	390	0.00500	0.00250	0.000900
Toluene	32.0	RIDEM Method 1 Leachability Criteria (GA)	16.0	0.00500	0.00250	0.00140
trans-1,2-Dichloroethene	3.30	RIDEM Method 1 Leachability Criteria (GA)	1.65	0.00500	0.00250	0.000700
Trichloroethene (TCE)	0.200	RIDEM Method 1 Leachability Criteria (GA)	0.100	0.00500	0.00250	0.000600
Vinyl chloride	0.0200	RIDEM Method 1 Direct Exposure Criteria	0.0100	0.0100	0.00500	0.000900

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

Matrix: **Solid**
Analytical Group: **Soil and Waste pH (SW9045D pH)**
Concentration Level (if applicable):
Units: **pH Units**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
pH			0.500	0.500	0.500	0.500

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

Matrix: **Solid**
Analytical Group: **Total Organic Carbon (SW9060A TOC)**
Concentration Level (if applicable):
Units: **mg/kg**

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Detection Limit (DL)
Total Organic Carbon			2000	400	300	85.0

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

Matrix: **Solid**
Analytical Group: **Grain Size (ASTM D422)**
Concentration Level (if applicable):
Units: **%**

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

Notes:

Surface Soil and Background Surface Soil:
The selected surface soil and background soil PAL is the lower of the HH PAL (based on levels protective of human health) and the Eco PAL (based on levels protective of ecological receptors), which were selected as follows:

HH PALs for surface soil were selected as the minimum of the following:

- USEPA Regional Screening Level (RSL) for Residential Soil (USEPA, 2019a). Values are based on a target cancer risk of 1E-6 and target hazard quotient (HQ) of 0.1 to account for cumulative effects per target organ. USEPA soil screening levels (SSLs) were not utilized in the selection of soil PALs since groundwater will be measured for direct assessment in the remedial investigation and risk assessments, as applicable.
- Rhode Island Department of Environmental Management (RIDEM) Method 1 Direct Exposure Criteria (Residential) (RIDEM, 2019).
- RIDEM Method 1 Leachability Criteria (GA) (RIDEM, 2019).
- The following surrogates were used for the HH surface soil and background soil PALs:
  - Value for trivalent chromium used for chromium.
  - Value for mercuric chloride used for mercury.

Ecological PALs for surface soil were selected based on the following hierarchy of sources (selecting the lowest of the available soil values within a source):

- Eco-SSL - Eco-SSLs derived by USEPA according to USEPA guidance (USEPA, 2005).
- USEPA R4 - USEPA Region 4 soil screening values (USEPA, 2018a).
- The following surrogates were used for the Ecological PALs:
  - Value for trivalent or total chromium used for chromium, as available.
  - Value for total xylenes used for xylene isomers.

Subsurface Soil and Background Subsurface Soil:
The selected subsurface soil PAL is the HH PAL (based on levels protective of human health), which is equal to the minimum of the following:

- USEPA RSL for Residential Soil (USEPA, 2019). Values are based on a target cancer risk of 1E-6 and target HQ of 0.1 to account for cumulative effects per target organ. USEPA SSLs were not utilized in the selection of soil PALs since groundwater will be measured for direct assessment in the remedial investigation and risk assessments, as applicable.
- RIDEM Method 1 Direct Exposure Criteria (Residential) (RIDEM, 2019).
- RIDEM Method 1 Leachability Criteria (GA) (RIDEM, 2019).
- The following surrogates were used for the subsurface soil PALs:
  - Value for trivalent chromium used for chromium.
  - Value for mercuric chloride used for mercury.
  - Value for total xylenes used xylene isomers.
  - Value for isopropylbenzene (cumene) used for p-cymene (p-isopropyltoluene).
  - Value for n-butanol used for tert-butyl alcohol.

Leachate:
The selected leachate PAL is the HH PAL (based on levels protective of human health), which is equal to the RIDEM Method 1 Leachability Criteria (GA) (USEPA, 2019).

Groundwater:
The selected groundwater PAL is the HH PAL (based on levels protective of human health), which is equal to the minimum of the following:

- USEPA RSL for Tapwater (USEPA, 2019). Values are based on a target cancer risk of 1E-6 and target HQ of 0.1 to account for cumulative effects on the same target organ.
- RIDEM Method 1 Groundwater Objective (GA) (RIDEM, 2019).
- USEPA Target Groundwater Concentrations for the Vapor Intrusion Pathway. Calculated using the USEPA Vapor Intrusion Screening Level (VISL) Calculator (USEPA, 2019b). Values based on a target cancer risk of 1E-6 and target HQ of 0.1 to account for cumulative effects on the same target organ.
- USEPA Maximum Contaminant Levels (MCLs) (USEPA, 2018b). Table of Regulated Drinking Water Contaminants.
- The following surrogates were used for the HH groundwater PALs:
  - Value for total chromium (or trivalent chromium if value for total is not available) used for chromium.
  - Value for mercuric chloride used for mercury.

- Value for total xylenes used for xylene isomers.
- Value for acenaphthene used for acenaphthylene.
- Value for pyrene used for benzo(g,h,i)perylene and phenanthrene.
- Value for isopropylbenzene (cumene) used for p-cymene (p-isopropyltoluene).
- Value for n-butanol used for tert-butyl alcohol.

Sediment:
The selected sediment PAL is the lower of the HH PAL (based on levels protective of human health) and the Eco PAL (based on levels protective of ecological receptors), which were selected as follows:

HH PALs for sediment were selected as the minimum of the following:

- USEPA RSL for Residential Soil (USEPA, 2019a). Values are based on a target cancer risk of 1E-6 and target HQ of 0.1 to account for cumulative effects per target organ.
- RIDEM Method 1 Direct Exposure Criteria (Residential) (RIDEM, 2019).
- RIDEM Method 1 Leachability Criteria (GA) Soil (RIDEM, 2019).
- The following surrogates were used for the HH sediment PALs:
  - Value for trivalent chromium used for chromium.
  - Value for mercuric chloride used for mercury.
  - Value for total xylenes used for xylene isomers.
  - Value for n-butanol used for tert-butyl alcohol.

Ecological PALs for sediment were selected based on the following source:

- USEPA Region 4 sediment screening values (USEPA, 2018a). Lower of freshwater and marine values selected.
- The following surrogate was used for the Ecological PALs:
  - Value for total xylenes used for xylene isomers.

Surface water:
The selected surface water PAL is the lower of the HH PAL (based on levels protective of human health) and the Eco PAL (based on levels protective of ecological receptors), which were selected as follows:

HH PALs for surface water were selected as the minimum of the following:

- USEPA National Recommended Water Quality Criteria (NRWQC) for Human Health (values for Water and Organism) (USEPA, 2019c).
- RIDEM Ambient Water Quality Criteria (AWQC) for Human Health (lower of the values for Water and Organism and Organism Only) (RIDEM, 2018).

If no surface water PAL was available from the above sources, the PAL was equal to the USEPA RSL for Tapwater (USEPA, 2019). Values are based on a target cancer risk of 1E-6 and target HQ of 0.1 to account for cumulative effects on the same target organ.

- The following surrogates were used for the surface water HH PALs:
  - Value for trivalent chromium used for chromium.
  - Value for n-butanol used for tert-butyl alcohol.
  - Value for total xylenes used for xylene isomers.

Ecological PALs for surface water were selected based on the following hierarchy of sources (selecting the lowest of the available soil values within a source):

- USEPA Region 4 Surface Water Screening Levels (value for Freshwater, Chronic) (USEPA, 2018a).
- Federal AWQC (value for Freshwater, Chronic; hardness of 100 mg/L) (USEPA, 2019d).
- RIDEM Surface Water Criteria (value for Freshwater, Chronic; hardness of 100 mg/L) (RIDEM, 2006).
- The following surrogates were used for the Ecological PALs:
  - Value for trivalent chromium used for chromium.
  - Value for total xylenes used for xylene isomers.
  - Value for benzene used for n-butylbenzene, n-propylbenzene, sec-butylbenzene, and tert-butylbenzene.

Porewater:
The selected porewater PAL is the Eco PAL (based on levels protective of ecological receptors), which is selected as the lower of the following sources of screening levels:

- USEPA Region 4 Surface Water Screening Levels (value for Freshwater, Chronic; hardness of 100 mg/L) (USEPA, 2018a).
- Federal AWQC (value for Freshwater, Chronic; hardness of 100 mg/L) (USEPA, 2019d).
- RIDEM Surface Water Criteria (value for Freshwater, Chronic; hardness of 100 mg/L) (RIDEM, 2006).
- The following surrogates were used for the porewater PALs:

- Value for trivalent chromium used for chromium.
- Value for total xylenes used for xylene isomers.
- Value for benzene used for n-butylbenzene, n-propylbenzene, sec-butylbenzene, and tert-butylbenzene.

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QAPP Worksheet #17: Sampling Design and Rationale

This worksheet describes the sampling design, basis for its selection, and field investigation details. The RI serves as the mechanism for collecting data to characterize Site conditions, determine the nature and extent of impacts, identify whether COPCs associated with former DoD activities are present in Site media at concentrations greater than risk-based human health and ecological screening levels and Site-specific background levels, and collect data to support the FS. The objective of this RI is to collect field and analytical data from each AOC to address unresolved data gaps, determine the presence and/or absence of COPCs associated with former DoD activities in Site media, and assess fate and transport of COPCs potentially impacting nearby receptors. Four AOCs have been identified at the Site and are the primary areas of investigation for the RI. Additional samples will be collected from areas outside the AOCs to determine relative background concentrations for establishing a Site-specific background dataset.

Historic reports and previous analytical results were reviewed and incorporated into the development of this QAPP. Data gaps were identified and prioritized as critical needs to update the CSM and enhance the existing dataset for use in a risk assessment and development of alternatives in the FS. As a result, the team developed a sampling approach which focused sampling locations in areas determined to contain spatial or analytical data gaps remaining from the Phase I site investigation, in areas which have not previously been investigated, and in areas of suspected sources and/or releases.

The objective of the first phase of the RI is to determine the presence-absence of analytes from former DoD activities using a biased sampling approach to identify potential source areas. It is understood that this biased sampling design does not provide representative exposure-based samples for statistical analysis as necessary for a risk assessment. However, in understanding the limitations of that dataset, the biased dataset will be evaluated in comparison to risk-based human health and ecological screening levels and BTVs to determine which constituents, if any, exceed the risk-based screening levels and BTVs, and in which areas. If biased sample results exceed risk-based screening levels and BTVs, then a second RI phase will be designed with an unbiased and exposure-based sampling approach and decision units and exposure units to be further evaluated in a risk assessment. If biased sample results do not exceed risk-based screening levels or BTVs, then no further risk assessment will be performed. **Appendix G** provides a risk assessment work plan, which describes the approach for comparison to risk-based human health and ecological screening levels and BTVs in the Phase I RI and also for conducting further risk assessment in the second phase of the RI, if necessary.

The following subsections outline the proposed sampling by media. This includes the quantity of samples to be collected, the vertical sampling interval, and the rationale for the selected sample locations. Further discussion regarding the methods and procedures for sample collection are included in **Worksheet #14**. Analytical methods are shown in **Worksheet #18**.

17.1 Soil Sampling

Discrete surface and subsurface soil samples will be collected in suspected source and/or release areas and in areas with identified data gaps within the established AOCs. The analytical methods for soils are shown in Worksheet #18, and include VOCs (subsurface soil only), SVOCs, metals, SPLP metals[1], pH, TOC, and grain size for Site soils and PAHs and metals for background soils. The locations of the proposed soil borings were determined after evaluating former DoD activities at the Site and historic sampling results from previous investigations. A total of 19 soil borings and 1 hand auger sample will be advanced across the four AOCs as indicated below. An additional four soil borings will be advanced solely for installation of monitoring wells (i.e. MW-006S, BR-002, BR-003, and BR-005). No soil sampling is proposed for these borings because these wells are located where soil will already be sampled or there are no known historic releases. The soil boring locations are shown by AOC on **Figures 17-1A through 17-1D**. Two soil samples will be collected from each soil boring: one surface soil sample (collected from the 0-2 ft bgs interval) and one subsurface soil sample (collected no deeper than 10 ft bgs). The sample intervals were specifically selected for several reasons:

- Surface soils collected during the Phase I site investigation were collected from the 0-2 ft bgs interval. For consistency and to combine the results of both datasets, the RI surface soil samples will be collected from 0-2 ft bgs. Surface soil samples will not be collected at PR79-SB-114, PR79-SB-115, and PR79-SB-116 (see **Table 17-1** for rationale).

- Due to the Site history (construction, activities, and demolition), it was agreed that sampling 0-1 ft bgs interval would likely only target reworked soils and not reach native material.

- Surface soils will be collected from the 0-2 ft bgs interval to allow for comparisons to historic datasets.

[1] The RI includes sampling of overburden and bedrock groundwater to evaluate for potential presence of COPCs that would have leached from soil over the past 50-60 years since former DoD activities ended in 1965. Due to the age of potential releases from former DoD activities and the duration of soil leaching to groundwater, it is unlikely that leachability remains a continuing source to groundwater. The RI will apply the "20 times rule" as a screening tool only for soils collected above the groundwater table, which assumes that absolutely all of the metal present in the soil sample leaches into the extract fluid.

- Subsurface soil samples will not be collected from depths greater than 10 ft bgs, which is the maximum depth to which human receptors may be exposed. Field screening will be performed to determine the exact sampling depth (highest PID, water table elevation, or lithologic interface) within the 2-10 ft bgs interval.

- Soil borings will be field screened for the entire length of the boring (to a maximum depth of boring). Additional subsurface samples will be collected if an interval of interest is observed (non-aqueous phase liquid [NAPL] present, elevated PID reading, or lithologic interface).

The proposed soil borings and hand auger sample are listed in **Table 17-1** along with the rationale for the selected location. Soil sampling procedures are summarized in **Worksheet #14**. Specific sample analyses are provided in **Worksheet #18**.

Table 17-1: Sampling Design and Rationale for Soil

AOC	Location ID	Depth	Rationale
AOC-1	PR79-SB-101 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located in front of the stairwell/ladder leading to the platform surrounding the former radar. A review of the 2015 AGC Report confirmed the orientation of the platform. After considering the historic soil and groundwater results in PZ-001, it was determined necessary to advance a second boring on the side of the former Radar Pad B to assess COPCs related to operation and maintenance of the radar pad. The upgradient side boring will cover another potential source and/or release area and be advanced deeper to assess COPCs in the overburden/weathered bedrock.
AOC-1	PR79-SB-102 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located downhill of the former Radar Pad C between it and an identified drainage feature. Based on a historic detection of 1,1-DCE in PZ-003, it is prudent to advance a third boring adjacent to the former Radar Pad C to assess COPC releases related to operation and maintenance of the radar pad. This location is proposed between the former radar pad and drainage feature to assess potential cross-gradient flow as downgradient PZ-003 had no DCE detections.
AOC-1	PR79-SB-103 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located downhill and downgradient of the former Interconnecting Corridor along a drainage feature to assess COPC releases related to operation and maintenance of the radar equipment.
AOC-1	PR79-SB-104 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located north of the slab of the former Frequency Changer/Generator Building where TCE and PAHs have been historically detected. The location was selected in an area of stressed vegetation next to the former Frequency Changer Building where drums were stored as observed from a review of the 2015 AGC Report. Historic detections of SVOCs/PAHs in soil and VOCs (TCE) in groundwater were detected below the slab, but no deep subsurface or groundwater data exists on the north side of the former building.

AOC	Location ID	Depth	Rationale
AOC-1	PR79-SB-105 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located within an area just east of AOC-1 where it is suspected that soil from the former Radar Pad may have been disposed. This area has not previously been subject to investigation and is an identified data gap. The boring location has been located in the suspected soil disposal area and will be adjusted as necessary in the field.
AOC-1	PR79-SB-106 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located adjacent to the former stairwell/ladder and platform surrounding the radar. A review of the 2015 AGC Report confirmed the orientation of the platform. After considering the historic soil and groundwater results in PZ-005, it was determined necessary to advance a second boring cross-gradient of the former Radar Pad A to assess COPCs related to operation and maintenance of the radar pad. The cross-gradient boring will cover another potential source and/or release area and be advanced deeper to assess COPCs in the overburden/weathered bedrock.
AOC-1	PR79-SB-107 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located within the former Helipad where deicing using solvents may have occurred. No previous investigations assessed within the boundary of the former unpaved helicopter pad which is an identified data gap. Historic results from nearby PZ-014 did not indicate VOC or SVOCs in soil and no chlorinated detections in groundwater. However, based on a review of shallow groundwater flow direction, this location is observed to be more cross-gradient than downgradient of the former Helipad. Further investigation is needed to assess the potential release area.
AOC-2	PR79-SB-108 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located at the end of a drainage feature which ran along the roadway. Based on an evaluation of the Site topography, this location would be a likely area of pooling and infiltration of water containing COPCs from deicing and other maintenance activities at the top of the hill. Further investigation is warranted to investigate this identified data gap.
AOC-2	PR79-SB-109 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located within an area thought to be used informally for motor pool activities. Historic boring SB-017 did not show detections of VOCs or SVOCs. However, SB-017 hit refusal at 4.5 ft bgs, so a second boring is required to assess uninvestigated deeper overburden for COPCs.
AOC-2	PR79-SB-110 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located at the confluence of two drainage ditches alongside the roadway where deicing using solvents may have occurred. No previous sampling has been performed in this area. Based on an evaluation of the topography, this location would be a likely area of pooling and infiltration of water containing COPCs from deicing and other maintenance activities at the top of the hill. Further investigation is warranted to investigate this identified data gap.
AOC-2	PR79-SB-111 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located west of the former Barracks to assess potential impacts in the deep overburden soil and weathered bedrock migrating down from AOC-1 and AOC-2. Located between potential upgradient sources on the hill and downgradient water supply wells ROU-2 and ROU-3. Within a 5-10 ft thick weathered bedrock zone which could introduce COPCs to the bedrock.

AOC	Location ID	Depth	Rationale
AOC-3	PR79-SB-112 (Figure 17-1C)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located downgradient of the former Septic Tank and adjacent to the former Distribution Box which potentially received COPCs from the sewage system. Limited soil data exists in this area from previous investigations. Additional sampling is required for horizontal and vertical data coverage. The proposed location is topographically and hydraulically downgradient of the former Septic Tank.
AOC-3	PR79-SB-113 (Figure 17-1C)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located adjacent to the former Mess Hall, which had elevated concentrations of SVOCs during previous sampling. Additional analytical results are necessary to assess potential impacts in the deep overburden soil and weathered bedrock migrating down from AOC-1.
AOC-3	PR79-SB-114 (Figure 17-1C)	Subsurface: up to 10 ft bgs	Located within the former Leachfield. Sewage from the former Septic Tank potentially containing COPCs were released here. Further investigation is warranted to investigate the deep overburden and weathered bedrock. No surface soil sampling proposed as release mechanism is subsurface only.
AOC-3	PR79-SB-115 (Figure 17-1C)	Subsurface: up to 10 ft bgs	Located along the Pipeline and Service Pathway from the Distribution Box to the Sand Pits and Chlorine Chamber. Intercept midpoint for COPCs migrating from the hilltop to Winsor Brook. Additionally, sewage leaking from the pipeline could potentially impact underlying soil and the pipeline bed could act as a preferential pathway for COPCs to flow. Further investigation is warranted to investigate this identified data gap. No surface soil sampling proposed as release mechanism is subsurface only.
AOC-3	PR79-SB-116 (Figure 17-1C)	Subsurface: up to 10 ft bgs	Located along the Pipeline and Service Pathway from the Distribution Box to the Sand Pits and Chlorine Chamber. Intercept midpoint for COPCs migrating from the hilltop to Winsor Brook. Additionally, sewage leaking from the pipeline could potentially impact underlying soil and the pipeline bed could act as a preferential pathway for COPCs to flow. Further investigation is warranted to investigate this identified data gap. No surface soil sampling proposed as release mechanism is subsurface only.
AOC-4	PR79-SB-117 (Figure 17-1D)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Positioned within the former Sand Pits to assess potential COPCs introduced to the former sewer and septic system.
AOC-4	PR79-SB-118 (Figure 17-1D)		
AOC-4	PR79-SB-119 (Figure 17-1D)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Positioned at the end of the drainage feature coming from the Chlorine Chamber. Potential release point COPCs entering the brook. Location selected to investigate potential migration of impacts from historical operations to Winsor Brook.
Hand Auger Location			
AOC-1	PR79-SB-120	Surface: 0-2 ft bgs	Hand auger location to determine if COPCs were potentially released in an area of stressed vegetation.

17.1.1 Soil Background Evaluation Sampling

An additional 20 soil borings will be advanced to provide a Site-specific background soil dataset for evaluation in comparison with Site data. The selected background study areas are located at the easternmost portion of the Property near the perimeter fence and along Theodore Foster Drive,

shown on **Figure 17-2**. Early aerial photographs from 1951 show that the areas in the easternmost portion of the Property was forested and cleared sometime between 1962 and 1963 (USAGC, 2015), but was never developed by the DoD. The area abuts a historic farmland and a solar array and was likely exposed to indirect local anthropogenic sources unrelated to DoD activities. A Site walkthrough in November 2019 observed this area as re-established forest used for game baiting. The background study area adjacent to Theodore Foster Drive has been a developed road since before DoD activities began.

Twenty soil borings (ten in each study area) will be advanced to a depth of 10 ft bgs. Soil samples will be collected from each of the advanced borings. Surface soil samples are considered to be 0 to 2 ft bgs and subsurface soil samples are considered 2-10 ft bgs. An unbiased sampling approach will be created using Pacific Northwest National Laboratory Visual Sample Plan software. Using this software, two foot sample intervals between 2-10 ft bgs will be randomly assigned to each boring for subsurface sampling. This approach was taken rather than compositing the entire 2-10 foot interval due to site conditions (presence of cobbles/boulders) requiring large bore sonic cores. This technical drilling approach would generate too much soil to adequately composite for sampling. Additional details on the soil background evaluation can be found in the Risk Assessment Work Plan located in **Appendix G**. The area where the background soil borings will be advanced is shown on **Figure 17-2**. Soil sampling procedures are summarized in **Worksheet #14**. Specific sample analyses are provided in **Worksheet #18**.

17.2 Groundwater Sampling

As part of the RI, nine overburden/weathered bedrock and five bedrock monitoring wells will be installed across the Property. Groundwater samples will also be collected from five existing piezometers. To achieve the stated groundwater goals, the monitoring well locations were established to collect groundwater samples on a Site-wide scale, as well as, on a local scale within specific AOCs that could potentially (or have demonstrated to) have localized groundwater impacts. The analytical methods for groundwater are shown in Worksheet #18, and include VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, and Ferrous Iron. Low-flow sampling will be performed following the USEPA Guidance on low stress purging and sampling (USEPA, 2017).

The predominant goal for the monitoring wells was to select locations that targeted data gaps in the overburden, weathered bedrock, and bedrock where COPCs could be acting as a continuing source to the dissolved plume observed in downgradient water supply wells. The secondary goal was to selectively position monitoring wells in downgradient locations to determine the full extent of impacts

to groundwater in the most likely flow directions based on the Site geology. These goals were achieved by evaluating the existing dataset and potential sources to identify the spatial data gaps across the Site where limited subsurface and groundwater data existed. Further evaluation of the historic surface and borehole geophysical work was done to determine the specific vertical intervals to target in order to understand fate and transport mechanics and migration of COPCs through fractured bedrock. The rationale for the locations and construction of each proposed monitoring well is provided in **Table 17-2**, and the design of the proposed monitoring well network is shown in **Figure 17-2**. **Figures 17-4, 17-5, and 17-6** show the proposed monitoring well network in comparison to the top of till, top of weathered bedrock, and top of bedrock elevations, respectively. Bedrock lineaments are shown on **Figure 17-3**. The existing piezometer construction details are provided in **Appendix B**.

To meet project goals, it is critical the proposed monitoring wells target source areas where impacts are likely to be found in the subsurface. As a result, real-time field screening and observations will be used to make decisions as to whether or not to proceed in the completion of soil borings as monitoring wells. A flowchart has been developed to guide the PDT in assessing field inputs in real-time. This iterative process will act to verify that each proposed monitoring well achieves the stated goal and rationale. The flowchart is provided in **Worksheet #14**.

Overburden and weathered bedrock monitoring wells will be constructed with the appropriate total well depth and screen interval based on field observations from the recovered material. Bedrock wells will be fitted with FLUTe sampling manifolds to specifically target transmissive fractures which could contribute to the transport of COPCs across the Site. Once monitoring well construction and development is complete, each monitoring well will be sampled via USEPA low-flow groundwater sampling guidelines (USEPA, 2017). Monitoring well installation, development, and sampling procedures are summarized in **Worksheet #14**. Specific sample analyses are provided in **Worksheet #18**.

Table 17-2: Sampling Design and Rationale for Groundwater

AOC	Location ID	Monitoring Well ID	Depth	Rationale
Overburden and Weathered Bedrock Monitoring Wells				
AOC-1	PR79-SB-101 (Figure 17-2)	PR79-MW-001	Weathered bedrock: ~40-50 ft bgs	Assessing the potential for COPC in the weathered bedrock downgradient and north of AOC-1 (Former Radar Pad B) and upgradient of PZ001.

AOC	Location ID	Monitoring Well ID	Depth	Rationale
AOC-1	PR79-SB-103 (Figure 17-2)	PR79-MW-002	Weathered bedrock: ~40-50 ft bgs	Assessing the potential for COPC in the weathered bedrock downgradient of the former Interconnecting Corridor and surface drainage feature. Placed along HGI Seismic Line 100 which identified a thicker weathered bedrock zone in this area.
AOC-1	PR79-SB-104 (Figure 17-2)	PR79-MW-003	Overburden: ~30 ft bgs	Further assessing trace VOC detections in PZ-019 beneath the slab of the former Frequency Changer/Generator Building. Coincides with fractures identified along HGI Seismic Line 100.
AOC-1	PR79-SB-106 (Figure 17-2)	PR79-MW-004	Weathered bedrock: ~ 10 ft bgs	Located on the downgradient side of the former Radar Pad A (near VOC detections in PZ-005) to assess potential releases related to operation and maintenance of the radar pad. Coincides with ANL Seismic Line 1 and potential weathered bedrock zone. Located within the zone of higher overburden hydraulic conductivity as determined by USACE in 2015.
AOC-2	PR79-SB-110 (Figure 17-2)	PR79-MW-005	Weathered bedrock: ~30-40 ft bgs	Assessing the potential for infiltration of COPC from two drainage ditches and potential upgradient deicing activities. Also assesses, potential pathway to weathered bedrock trending northeast-southwest towards ROU-2.
AOC-3	NA (Figure 17-2)	PR79-MW-006S	Overburden: ~10-15 ft bgs	Targets the shallow most water bearing unit adjacent to the former Mess Hall for human health risk assessment.
AOC-3	PR79-SB-113 (Figure 17-2)	PR79-MW-006D	Weathered bedrock: ~20-30 ft bgs	Assessing the local depression in the weathered bedrock unit to determine if weathered bedrock acts as a conduit for northeast-southwest COPC migration.
AOC-3	PR79-SB-114 (Figure 17-2)	PR79-MW-007	Overburden: ~10-15 ft bgs	Assessing potential overburden impacts from the former Leachfield and Septic Tank.
AOC-4	PR79-SB-118 (Figure 17-2)	PR79-MW-008	Overburden: ~10-15 ft bgs	Positioned at the downgradient edge of the former Sand Pits to assess whether they affected groundwater quality. Further investigation is warranted to determine the horizontal and vertical distribution of COPC due west of the Property.
Bedrock Monitoring Wells				
AOC-1	PR79-SB-107 (Figure 17-2)	PR79-BR-001	Bedrock: ~445 ft bgs, sample ports TBD	Located in close proximity to NIKE-1, the helicopter pad and the weathered bedrock migration pathway to BR. Will be advanced to intercept transmissive north-northeast dipping fractures observed in NIKE-1. Located within the higher overburden hydraulic conductivity area as determined by USACE in 2015. Will attempt to determine potential impacts to flow and migration from the overburden to bedrock.

AOC	Location ID	Monitoring Well ID	Depth	Rationale
AOC-1	NA (Figure 17-2)	PR79-BR-002	Bedrock: ~600 ft bgs, sample ports TBD	Assessing potential COPC immediately downgradient of former Frequency Changer/ Generator Building. Coincides with fractures identified along HGI Seismic Line 100.
NA	NA (Figure 17-2)	PR79-BR-003	Bedrock: ~375 ft bgs, sample ports TBD	Assessing COPCs in the vicinity of ROU-1 and downgradient of potential sources and beneath the weathered bedrock trough. Located immediately downgradient of the higher overburden hydraulic conductivity area as determined by USACE in 2015. Will attempt to determine potential impacts to flow and migration from the overburden to bedrock.
AOC-2	PR79-SB-111 (Figure 17-2)	PR79-BR-004	Bedrock: ~600 ft bgs, sample ports TBD	Located beneath northeast-southwest trending weathered bedrock zone that may provide conduit for migration into bedrock toward ROU-2. Limited downgradient bedrock coverage on western side of the Property.
AOC-3	NA (Figure 17-2)	PR79-BR-005	Bedrock: ~460 ft bgs, sample ports TBD	Located along identified lineament that may represent a migration pathway between NIKE-1 locations south of the Property. Acts as off-Property, downgradient bedrock monitoring well.
Existing Locations				
AOC-1	PZ -001 (Figure 17-2)		Screen interval: 5.5-15.5 ft bgs	Included in monitoring well network due to historic VOC detections and spatial coverage for future gauging.
AOC-1	PZ-005 (Figure 17-2)		Screen interval: 6.5-11.5 ft bgs	Included in monitoring well network due to previous 1,1-DCE detection. Additionally, will serve as a pair with the proposed weathered bedrock monitoring well MW-04.
AOC-1	PZ-007 (Figure 17-2)		Screen interval: 3.0-13.0 ft bgs	Included in monitoring well network due to previous VOC detections and spatial coverage for future gauging.
AOC-1	PZ-010 (Figure 17-2)		Screen interval: 3.9-13.9 ft bgs	Included in monitoring well network due to spatial coverage for future gauging.
AOC-1	PZ-014 (Figure 17-2)		Screen interval: 7.5-12.5 ft bgs	Included in monitoring well network due to spatial coverage for future gauging. Additionally, will serve as a pair with the proposed bedrock monitoring well BR-03.
NA	Hand Dug Well at 41 Winsor Road		TBD	Supplemental sample to replace a surface water, pore water, and sediment sample from along Winsor Brook.
NA	Steere Well		TBD	Included as a background bedrock groundwater location.

17.3 Surface Water, Pore Water, and Sediment Sampling

Surface water and co-located pore water and sediment/soil samples will be collected from streams, seeps, and delineated wetlands surrounding the Property. There are no surface water features within the boundaries of the Property; therefore, the sampling design targeted surface water exposure areas potentially impacted by upgradient sources migrating from the Property. The objective of this sampling is to provide a dataset for determining the presence and/or absence of COPCs associated with former DoD activities and to assess the nature and extent of Site impacts within nearby stream channels, seeps, and delineated wetlands as identified in a *Wetland and Waters of the US Delineation Report* (Woodard & Curran, 2019). A total of 25 collocated surface water, pore water, and sediment samples will be collected from suitable surface water locations surrounding the Property as indicated in QAPP **Worksheet #14**. The locations of the surface water, pore water, and sediment samples are shown on **Figure 17-3**. Samples within the same exposure area (e.g., stream, wetland, etc.) will be spaced approximately 100 ft apart along a linear stretch.

If sufficient water is present within the surface water feature, the surface water sample will be collocated with the pore water and sediment sample. Otherwise, only pore water and sediment will be collected. Due to flow conditions, fine grained material may not be present within the selected stretch of Winsor Brook. If a sufficient volume of sediment is not present in the proposed sampling location, the team agreed to collect sediment/soil and pore water samples from bank material along the eastern bank of Winsor Brook and surface water samples from within Winsor Brook. Pore water and sediment/soil samples will be collected from the 0-0.5 ft bgs interval and surface water will be collected from the midpoint of the water column.

The rationale for the locations of the surface water and co-located pore water and sediment/soil samples is provided in **Table 17-3**. Surface water, pore water, and sediment sampling procedures are summarized in **Worksheet #14**. Specific sample analyses are provided in **Worksheet #18**, and include VOCs, SVOCs, metals (total and filtered), and hardness. Surface water, pore water, and sediment sample locations are shown on **Figure 17-3.**

Table 17-3: Sampling Design and Rationale for Surface Water, Pore Water, and Sediment

Location ID	Depth	Rationale
PR79-WT-001	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Small pond located north of Property to assess COPCs migrating from the Property.
PR79-WT-002	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located to the west of the Property along a section of Winsor Brook to assess COPCs from surface discharge from the former septic system.
PR79-WT-003		
PR79-WT-004		
PR79-WT-005		
PR79-WT-006		
PR79-WT-007	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located within a pond to assess COPCs migrating from the Property along a two streams originating from the wetland complex located at a higher elevation south of the Property.
PR79-WT-008	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located to the south-southwest of the Property along previously delineated wetland to assess potential impacts migrating from the Property.
PR79-WT-009	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located to the south of the Property along previously identified stream within delineated wetland to assess potential impacts migrating from the Property.
PR79-WT-010		
PR79-WT-011		
PR79-WT-012		
PR79-WT-013		
PR79-WT-014		
PR79-WT-015		
PR79-WT-016	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located to the south-east of the Property along previously identified stream and wetland area to assess potential impacts migrating from the Property.
PR79-WT-017		
PR79-WT-018		
PR79-WT-019		
PR79-WT-020		
PR79-WT-021	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located at each stream culvert located upstream of the pond next to Winsor Road. Both streams appear to originate from a wetland complex south of the Property and may interact with potential seeps.
PR79-WT-022		
PR79-WT-023	SW: midpoint of water column PW: 0-1 ft bgs SD: 0-0.5 ft bgs	Located to the north-east of the Property along previously identified stream and wetland area to assess potential impacts migrating from the Property.

17.3.1 Surface Water, Pore Water, and Sediment Background Evaluation

Ten surface water and co-located pore water and sediment/soil samples will be advanced along Winsor Brook, upstream of the AOC-4 discharge point to provide a Site-specific background dataset for evaluation in comparison with Site data. The background surface water, pore water, and sediment samples will be collected and analyzed for the same suite as listed in Section 17.3. Additional details on the surface water, pore water, and sediment background evaluation can be found in the Risk Assessment Work Plan located in **Appendix G**. The location of the 10 samples is shown on **Figure 17-3**. Surface water, pore water, and sediment sampling procedures are summarized in **Worksheet #14**. Specific sample analyses are provided in **Worksheet #18.**

QAPP Worksheet #18: Sampling Locations and Methods

Surface and Subsurface Soil

Sample Location (LOCID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-SB-101	PR79-SB-101-AS-01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-101-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-102	PR79-SB-102-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-102-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-103	PR79-SB-103-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-103-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-104	PR79-SB-104-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-104-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-105	PR79-SB-105-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-105-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-106	PR79-SB-106-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-106-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-107	PR79-SB-107-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-107-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-108	PR79-SB-108-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-108-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-109	PR79-SB-109-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-109-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-110	PR79-SB-110-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-110-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-111	PR79-SB-111-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-111-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-112	PR79-SB-112-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-112-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-113	PR79-SB-113-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-113-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-114	PR79-SB-114-BS- 01	SO	TBD	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
PR79-SB-115	PR79-SB-115-BS- 01	SO	TBD	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
PR79-SB-116	PR79-SB-116-BS- 01	SO	TBD	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	

Sample Location (LOCID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-SB-117	PR79-SB-117-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-117-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-118	PR79-SB-118-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-118-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-119	PR79-SB-119-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
	PR79-SB-119-BS- 01	SO	TBD	Normal		3-21	
PR79-SB-120	PR79-SB0120-AS- 01	SO	0-2	Normal	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	
PR79-SB-104	PR79-SB-FD01- 01	SO	0-2	Field QC	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	Field Duplicate
	PR79-SB-FD02- 01	SO	TBD	Field QC		3-21	
PR79-SB-113	PR79-SB-FD03- 01	SO	0-2	Field QC	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	Field Duplicate
	PR79-SB-FD04- 01	SO	TBD	Field QC		3-21	
PR79-SB-118	PR79-SB-118-AS- 01	SO	0-2	Field QC	VOCs[4], SVOCs, metals, SPLP metals[5], pH[6], TOC[6], grain size[6]	3-21	MS/MSD
	PR79-SB-118-BS- 01	SO	TBD	Field QC		3-21	
Field QC	SO-EB- 01	AQ	NA	Field QC	VOCs		Equipment Blank
Field QC	TB- 01-01	AQ	NA	Field QC	VOCs		-02, -03, etc. if multiple collected in one day

Background Soil

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-SB-121	PR79-SB-121-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-FD05-01	SO	0-2	Field QC	SVOCs, metals	3-21	Field Duplicate
	PR79-SB-121-BS-01	SO	2-4	Normal	VOCs, SVOCs, metals	3-21	
	PR79-SB-FD06-01	SO	2-4	Field QC	VOCs[4], SVOCs, metals	3-21	Field Duplicate

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-SB-122	PR79-SB-122-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-122-BS-01	SO	8-10	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-123	PR79-SB-123-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-123-BS-01	SO	4-6	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-124	PR79-SB-124-AS-01	SO	0-2	Normal	PAHs, metals	3-21	
	PR79-SB-124-BS-01	SO	6-8	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-125	PR79-SB-125-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-125-BS-01	SO	2-4	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-126	PR79-SB-126-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-126-BS-01	SO	8-10	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-127	PR79-SB-127-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-127-BS-01	SO	2-4	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-128	PR79-SB-128-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-128-BS-01	SO	6-8	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-129	PR79-SB-129-AS-01	SO	0-2	Field QC	SVOCs, metals	3-21	MS/MSD
	PR79-SB-129-BS-01	SO	8-10	Field QC	VOCs, SVOCs, metals	3-21	MS/MSD
PR79-SB-130	PR79-SB-130-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-130-BS-01	SO	4-6	Normal	VOCs, SVOCs, metals	3-21	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-SB-131	PR79-SB-131-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-131-BS-01	SO	4-6	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-132	PR79-SB-132-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-132-BS-01	SO	2-4	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-133	PR79-SB-133-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-133-BS-01	SO	2-4	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-134	PR79-SB-134-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-134-BS-01	SO	8-10	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-135	PR79-SB-135-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-135-BS-01	SO	4-6	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-136	PR79-SB-136-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-FD07- 01	SO	0-2	Field QC	SVOCs, metals	3-21	Field Duplicate
	PR79-SB-136-BS-01	SO	4-6	Normal	VOCs, SVOCs, metals	3-21	
	PR79-SB-FD08- 01	SO	4-6	Field QC	VOCs, SVOCs, metals	3-21	Field Duplicate
PR79-SB-137	PR79-SB-137-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-137-BS-01	SO	8-10	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-138	PR79-SB-138-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-138-BS-01	SO	6-8	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-139	PR79-SB-139-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
	PR79-SB-139-BS-01	SO	4-6	Normal	VOCs, SVOCs, metals	3-21	
PR79-SB-140	PR79-SB-140-AS-01	SO	0-2	Normal	SVOCs, metals	3-21	
	PR79-SB-140-BS-01	SO	2-4	Normal	VOCs, SVOCs, metals	3-21	

Groundwater

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-MW-001	PR79-MW-001- 01	GW	Proposed Screened Interval: 45-50 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-002	PR79-MW-002- 01	GW	Proposed Screened Interval: 45-50 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-003	PR79-MW-003- 01	GW	Proposed Screened Interval: 25-30 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-004	PR79-MW-004- 01	GW	Proposed Screened Interval: 10-15 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-005	PR79-MW-005- 01	GW	Proposed Screened Interval: 35-40 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-006S	PR79-MW-006S-01	GW	Proposed Screened Interval: 10-15 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-MW-006D	PR79-MW-006D-01	GW	Proposed Screened Interval: 25-30 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-007	PR79-MW-007- 01	GW	Proposed Screened Interval: 10-15 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PR79-MW-008	PR79-MW-008- 01	GW	Proposed Screened Interval: 10-15 ft bgs	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PZ-001	PZ -001- 01	GW	Screened: 5.5-15.5	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PZ-005	PZ-005- 01	GW	Screened: 6.5-11.5	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PZ-007	PZ-007-01	GW	Screened: 3.0-13.0	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PZ-010	PZ-010-01	GW	Screened: 3.9-13.9	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
PZ-014	PZ-014-01	GW	Screened: 7.5-12.5	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden
Hand Dug Well at 41 Winsor Road	PR79-WL-001-01	GW	TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Overburden

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
Steere Well	PR79-WL-002-01	GW	TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Background bedrock
PR79-BR-001	PR79-BR-001-P1-01	GW	Sample Port Depth: TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Bedrock
	PR79-BR-001-P2-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
	PR79-BR-001-P3-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
PR79-BR-002	PR79-BR-002-P1-01	GW	Sample Port Depth: TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Bedrock
	PR79-BR-002-P2-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
	PR79-BR-002-P3-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
PR79-BR-003	PR79-BR-003-P1-01	GW	Sample Port Depth: TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Bedrock
	PR79-BR-003-P2-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
	PR79-BR-003-P3-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
PR79-BR-004	PR79-BR-004-P1-01	GW	Sample Port Depth: TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Bedrock
	PR79-BR-004-P2-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
	PR79-BR-004-P3-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
PR79-BR-005	PR79-BR-005-P1-01	GW	Sample Port Depth: TBD	Normal	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Bedrock
	PR79-BR-005-P2-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock
	PR79-BR-005-P3-01	GW	Sample Port Depth: TBD	Normal		3-14	Bedrock

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-MW-001	PR79-GW-FD01-01	GW	Proposed Screened Interval: 45-50 ft bgs	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Field Duplicate
PR79-MW-006S	PR79-GW-FD02--01	GW	Proposed Screened Interval: 10-15 ft bgs	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Field Duplicate
PR79-BR-001	PR79-GW-FD03--01	GW	Sample Port Depth: TBD	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Field Duplicate
PR79-BR-004	PR79-GW-FD04-01	GW	Sample Port Depth: TBD	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	Field Duplicate
PR79-MW-005	PR79-MW-005-01	GW	Proposed Screened Interval: 35-40 ft bgs	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	MS/MSD
PR79-BR-002	PR79-BR-002-P3-01	GW	Sample Port Depth: TBD	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron	3-14	MS/MSD
Field QC	GW-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals (filtered and non-filtered), TOC, Dissolved Gases, Nitrate, Nitrite, Sulfate, Chloride, Sulfide, Alkalinity, Ferrous Iron		Equipment Blank
Field QC	TB-01-01	AQ	NA	Field QC	VOCs		-02, -03, etc. if multiple collected in one day

Surface Water

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-001	PR79-WT-001-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-002	PR79-WT-002-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-003	PR79-WT-003-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-004	PR79-WT-004-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-005	PR79-WT-005-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-006	PR79-WT-006-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-007	PR79-WT-007-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-008	PR79-WT-008-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-009	PR79-WT-009-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-010	PR79-WT-010-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-011	PR79-WT-011-SW01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-0012	PR79-WT-012-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-013	PR79-WT-013-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-014	PR79-WT-014-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-015	PR79-WT-015-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-016	PR79-WT-016-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-017	PR79-WT-017-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-018	PR79-WT-018-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-019	PR79-WT-019-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-020	PR79-WT-020-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-021	PR79-WT-021-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-022	PR79-WT-022-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-023	PR79-WT-023-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-001	PR79-SW-FD01-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	Field Duplicate
PR79-WT-09	PR79-SW-FD02-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	Field Duplicate
PR79-WT-018	PR79-SW-FD03-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	Field Duplicate
PR79-WT-014	PR79-WT-014-SW-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	MS/MSD
PR79-WT-023	PR79-WT-023-SW-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	MS/MSD
Field QC	SW-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals (total and filtered), hardness		Equipment Blank
Field QC	SW-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals (total and filtered), hardness		Equipment Blank

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
Field QC	TB--01	AQ	NA	Field QC	VOCs		-02, -03, etc. if multiple collected in one day

Pore Water

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-001	PR79-WT-001-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-002	PR79-WT-002-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-003	PR79-WT-003-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-004	PR79-WT-004-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-005	PR79-WT-005-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-006	PR79-WT-006-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-007	PR79-WT-007-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-008	PR79-WT-008-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-009	PR79-WT-009-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-010	PR79-WT-010-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-011	PR79-WT-011-PW01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-0012	PR79-WT-012-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-013	PR79-WT-013-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-014	PR79-WT-014-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-015	PR79-WT-015-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-016	PR79-WT-016-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-017	PR79-WT-017-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-018	PR79-WT-018-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-019	PR79-WT-019-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-020	PR79-WT-020-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-021	PR79-WT-021-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-022	PR79-WT-022-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-023	PR79-WT-023-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-001	PR79-PW-FD01-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	Field Duplicate
PR79-WT-09	PR79-PW-FD02-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	Field Duplicate
PR79-WT-018	PR79-PW-FD03-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	Field Duplicate

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-014	PR79-WT-014-PW-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	MS/MSD
PR79-WT-023	PR79-WT-023-PW-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	MS/MSD
Field QC	PW-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals (total and filtered), hardness		Equipment Blank
Field QC	PW-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals (total and filtered), hardness		Equipment Blank
Field QC	TB--01	AQ	NA	Field QC	VOCs		-02, -03, etc. if multiple collected in one day

Sediment

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-001	PR79-WT-001-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-002	PR79-WT-002-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-003	PR79-WT-003-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-004	PR79-WT-004-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-005	PR79-WT-005-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-006	PR79-WT-006-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-007	PR79-WT-007-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-008	PR79-WT-008-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-009	PR79-WT-009-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-010	PR79-WT-010-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-011	PR79-WT-011-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-012	PR79-WT-012-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-013	PR79-WT-013-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-014	PR79-WT-014-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-015	PR79-WT-015-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-016	PR79-WT-016-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-017	PR79-WT-017-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-018	PR79-WT-018-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-019	PR79-WT-019-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-020	PR79-WT-020-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-021	PR79-WT-021-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-022	PR79-WT-022-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-023	PR79-WT-023-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-001	PR79-SD-FD01-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	Field Duplicate
PR79-WT-09	PR79-SD-FD02-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	Field Duplicate
PR79-WT-018	PR79-SD-FD03-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	Field Duplicate
PR79-WT-014	PR79-WT-014-SD-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC	3-22	MS/MSD
PR79-WT-023	PR79-WT-023-SD-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC	3-22	MS/MSD
Field QC	SD-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC		Equipment Blank
Field QC	SD-EB-01	AQ	NA	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC		Equipment Blank
Field QC	TB--01	AQ	NA	Field QC	VOCs		-02, -03, etc. if multiple collected in one day

Background Surface Water

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-024	PR79-WT-024-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-025	PR79-WT-025-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-026	PR79-WT-026-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-027	PR79-WT-027-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-028	PR79-WT-028-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-029	PR79-WT-029-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-030	PR79-WT-030-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-031	PR79-WT-031-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-032	PR79-WT-032-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-033	PR79-WT-033-SW-01	SW	Midpoint of Water Column	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-10	
PR79-WT-025	PR79-SW-FD04-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	Field Duplicate
PR79-WT-026	PR79-WT-026-SW-01	SW	Midpoint of Water Column	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-10	MS/MSD

Background Pore Water

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-024	PR79-WT-024-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-025	PR79-WT-025-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-026	PR79-WT-026-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-027	PR79-WT-027-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-028	PR79-WT-028-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-029	PR79-WT-029-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-030	PR79-WT-030-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-031	PR79-WT-031-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-032	PR79-WT-032-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-033	PR79-WT-033-PW-01	AQ	0-1	Normal	VOCs, SVOCs, metals (total and filtered), hardness	3-45	
PR79-WT-025	PR79-PW-FD04-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	Field Duplicate
PR79-WT-026	PR79-WT-026-PW-01	AQ	0-1	Field QC	VOCs, SVOCs, metals (total and filtered), hardness	3-45	MS/MSD

Background Sediment

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-024	PR79-WT-024-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-025	PR79-WT-025-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-026	PR79-WT-026-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-027	PR79-WT-027-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-WT-028	PR79-WT-028-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-029	PR79-WT-029-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-030	PR79-WT-030-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-031	PR79-WT-031-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-032	PR79-WT-032-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-033	PR79-WT-033-SD-01	SE	0-1	Normal	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	
PR79-WT-025	PR79-SD-FD04-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC, grain size	3-22	Field Duplicate
PR79-WT-026	PR79-WT-026-SD-01	SE	0-1	Field QC	VOCs, SVOCs, metals, AVS/SEM, pH, TOC	3-22	MS/MSD

[1]Sample IDs for samples that are field-filtered will be appended with "-F".

[2]Key: SO = soil, SE = sediment, GW = groundwater, SW = surface water, AQ=Aqueous

[3]See Worksheet #20 for full list of VOC, SVOC, PAH, metals, SPLP metals, and dissolved gases sampling suites.

[4]VOCs will only be analyzed in the subsurface soil samples

[5]The total metals results will be divided by 20 and compared to the RIDEM GA Leachability Criteria. Results greater than the RIDEM GA Leachability Criteria will be selected for SPLP analysis.

[6]pH, TOC, and grain size will only be analyzed in the surface soil samples.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
MS/MSD = matrix spike/matrix spike duplicate
PCBs = Poly
QC = quality control
SIM = selective ion monitoring
SPLP = synthetic precipitation leaching procedure
SVOCs = semi-volatile organic compounds
TOC = total organic carbon
VOCs = volatile organic compounds

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times

Laboratory (Name, sample receipt address, POC, e-mail, and phone numbers): Refer to table below

List any required accreditations/certifications: DoD ELAP[7]

Back-up Laboratory: None

Sample Delivery Method: Overnight Courier

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
Surface Soil							
Volatile Organic Compounds	SW-846 5035, 8260C/ CA-202, CA-214	3 x 40-mL VOA vials w/ H_20 1 x 40-mL VOA vial w/ MeOH 1 x 2-ounce (oz) glass	≤6°C but not frozen; freeze within 48 hours	NA	Analyze within 48 hours or analyze within 14 days if frozen (H_2O) 14 days (MeOH)	21 days	Katahdin Analytical Services, LLC[6]
Semivolatile Organic Compounds (SIM)	SW846 3540C or 3550C, 8270D SIM/ CA-213; CA-512 or CA-526	4-oz wide-mouth jar	≤6°C but not frozen	14 days	40 days from extraction	21 days	Katahdin Analytical Services, LLC[6]
Metals - 6010	SW846 3050B, 6010C/ CA-605, CA-608	2-oz wide-mouth jar	None	NA	6 months	21 days	Katahdin Analytical Services, LLC[6]
Metals - 6020	SW846 3050B, 6020A/ CA-605, CA-627				6 months		
Mercury	SW846 7471B/ CA-611				28 days		
SPLP Metals	SW-846 1312, 6010C,7470A, 3010 / CA-620, CA-604, CA-608, CA-615	8-oz wide-mouth jar	≤6°C but not frozen	28 days (Hg), 6 months (all others) to SPLP extraction	28 days (Hg), 6 months (all others) from extraction	21 days	Katahdin Analytical Services, LLC[6]
pH	SW846 9045D/ CA-709	2-oz wide-mouth jar	≤6°C but not frozen	NA	24 hours	21 days	Katahdin Analytical Services, LLC[6]

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
Total Organic Carbon	SW846 9060A (mod)/ CA-741	2-oz wide-mouth jar	≤6°C but not frozen	NA	28 days	21 days	Katahdin Analytical Services, LLC[6]
Grain Size	ASTM D422 / CA-551	8-oz wide-mouth jar	≤6°C but not frozen	NA	NA	21 days	Katahdin Analytical Services, LLC[6]
Subsurface Soil							
Volatile Organic Compounds	SW-846 5035, 8260C/ CA-202, CA-214	3 x 40-mL VOA vials w/ H_20 1 x 40-mL VOA vial w/ MeOH 1 x 2-oz glass	≤6°C but not frozen; freeze within 48 hours	NA	Analyze within 48 hours or analyze within 14 days if frozen (H_2O) 14 days (MeOH)	21 days	Katahdin Analytical Services, LLC[6]
Semivolatile Organic Compounds (SIM)	SW846 3540C or 3550C, 8270D SIM/ CA-213; CA-512 or CA-526	4-oz wide-mouth jar	≤6°C but not frozen	14 days to extraction	40 days from extraction	21 days	Katahdin Analytical Services, LLC[6]
Metals - 6010	SW846 3050B, 6010C/ CA-605, CA-608	2-oz wide-mouth jar	None	NA	6 months	21 days	Katahdin Analytical Services, LLC[6]
Metals - 6020	SW846 3050B, 6020A/ CA-605, CA-627				6 months		
Mercury	SW846 7471B/ CA-611				28 days		
SPLP Metals	SW-846 1312, 6010C,7470A, 3010 / CA-620, CA-604, CA-608, CA-615	8-oz wide-mouth jar	≤6°C but not frozen	28 days (Hg), 6 months (all others) to SPLP extraction	28 days (Hg), 6 months (all others) from extraction	21 days	Katahdin Analytical Services, LLC[6]
FACT							
Volatile Organic Compounds	SW-846 8260C	1 x 4 oz. jar w/ DI water	≤6°C but not frozen	7 days	14 days	21 days	Pace Analytical Services
Groundwater							
Volatile Organic Compounds	SW846 5030B, 8260C/ CA-202	3 x 40-mL VOA vial	≤6°C but not frozen HCl to pH < 2	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
Volatile Organic Compounds (SIM)[7]	SW846 5030B, 8260C SIM/ CA-220	3 x 40-mL VOA vial	≤6°C but not frozen HCl to pH < 2	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]
Semivolatile Organic Compounds (SIM)	SW846 3510C, 3520C, 8270D SIM/ CA-213, CA-502	2 x 1-L amber glass bottles	≤6°C but not frozen	7 days	40 days from extraction	21 days	Katahdin Analytical Services, LLC[6]
6010 Metals (total or field-filtered)	SW846 3010A, 6010C/ CA-604, CA-608	1 x 250-mL polyethylene bottle	HNO3 to pH < 2	NA	6 months	21 days	Katahdin Analytical Services, LLC[6]
6020 Metals (total or field-filtered)	SW846 3010A, 6020A/ CA-604, CA-627				6 months		
Mercury (total or field-filtered)	SW7470A/ CA-615				28 days		
Total Organic Carbon	SM 5310 B/ CA-763	2 x 40-ml VOA vials	H2SO4 to pH < 2, ≤6°C but not frozen	NA	28 days	21 days	Katahdin Analytical Services, LLC[6]
Dissolved Gases	RSK-175/ CA-336	2 x 40-mL VOA vials	HCl to pH <2, ≤6°C but not frozen, no headspace	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]
Anions (Cl, NO2, NO3, SO4)	SW846 9056A/ CA-742	1 x 100-ml polyethylene bottle	≤6°C but not frozen	NA	48 hours (nitrate, nitrite,); 28 days (chloride and sulfate)	21 days	Katahdin Analytical Services, LLC[6]
Sulfide	SM 4500S2 F / CA-722	1 x 500-ml polyethylene bottle	2N Zinc Acetate /L & NaOH, ≤6°C but not frozen	NA	7 days	21 days	Katahdin Analytical Services, LLC[6]
Alkalinity	SM 2320 B/ CA-739	1 x 100-ml polyethylene bottle	Settled, ≤6°C but not frozen	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
Surface Water, Pore Water							
Volatile Organic Compounds	SW846 5030B, 8260C/ CA-202	3 x 40-mL VOA vial	≤6°C but not frozen HCl to pH < 2	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]
Volatile Organic Compounds (SIM)[7]	SW846 5030B, 8260C SIM/ CA-220	3 x 40mL VOA vial	≤6°C but not frozen HCl to pH < 2	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]
Semivolatile Organic Compounds (SIM)	SW846 3510C, 3520C, 8270D SIM/ CA-213, CA-502	2 x 1-L amber glass bottles	≤6°C but not frozen	7 days	40 days from extraction	21 days	Katahdin Analytical Services, LLC[6]
6010 Metals (total or field-filtered)	SW846 3010A, 6010C/ CA-604, CA-608	1 x 250-mL polyethylene bottle	HNO3 to pH < 2	NA	6 months	21 days	Katahdin Analytical Services, LLC[6]
6020 Metals (total or field-filtered)	SW846 3010A, 6020A/ CA-604, CA-627				6 months	21 days	Katahdin Analytical Services, LLC[6]
Mercury (total or field-filtered)	SW7470A/ CA-615				28 days		
Hardness	SM 2340 B (calc)/ CA-608	(Calculated from 6010 metals)	HNO3 to pH < 2	NA	6 months	21 days	Katahdin Analytical Services, LLC[6]
Sediment							
Volatile Organic Compounds	SW-846 5035, 8260C/ CA-202, CA-214	3 x 40-mL VOA vials w/ H_20 1 x 40-mL VOA vial w/ MeOH 1 x 2-oz glass	≤6°C but not frozen; freeze within 48 hours	NA	Analyze within 48 hours or analyze within 14 days if frozen (H_2O) 14 days (MeOH)	21 days	Katahdin Analytical Services, LLC[6]
Semivolatile Organic Compounds (SIM)	SW846 3540C or 3550C, 8270D SIM/ CA-213; CA-512 or CA-526	4-oz wide-mouth jar	≤6°C but not frozen	14 days	40 days from extraction	21 days	Katahdin Analytical Services, LLC[6]
Metals - 6010	SW846 3050B, 6010C/ CA-605, CA-608	2-oz wide-mouth jar	None	NA	6 months	21 days	Katahdin Analytical Services, LLC[6]

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
Metals - 6020	SW846 3050B, 6020A/ CA-605, CA-627				6 months		
Mercury	SW846 7471B/ CA-611				28 days		
AVS/SEM	EPA 376.3/ CA-738	2-oz wide-mouth jar	Protected from oxygen, ≤6°C but not frozen	NA	14 days	21 days	Katahdin Analytical Services, LLC[6]
pH	SW846 9045D/ CA-709	2-oz wide-mouth jar	≤6°C but not frozen	NA	24 hours	21 days	Katahdin Analytical Services, LLC[6]
Total Organic Carbon	SW846 9060A (mod) / CA-741	2-oz wide-mouth jar	≤6°C but not frozen	NA	28 days	21 days	Katahdin Analytical Services, LLC[6]
Grain Size	ASTM D422 / CA-551	8-oz wide-mouth jar	≤6°C but not frozen	NA	NA	21 days	Katahdin Analytical Services, LLC[6]

Notes:

(1) Refer to Worksheet #15 for specific target analytes.

(2) Refer to the Analytical SOP References table (Worksheet #23).

(3) Sample containers and mass/volume required for analyses to be conducted by one laboratory may be consolidated

(4) TAT is presented in calendar days unless otherwise indicated.

(5) No backup laboratories have been identified. All laboratories are subcontracted by AECOM unless otherwise indicated.

(6) Katahdin Analytical Services, LLC, 600 Technology Way, Scarborough, Maine 04074. Point of Contact: Heather Manz, hmanz@katahdinlab.com, Direct – (207) 874-2400 x17, Fax – (207)775-4029

(7) DoD ELAP accreditation is held for all analyses and analytes in the DoD QSM v 5.1. Fixed laboratory analyses with this footnote indicated are either not included in the QSM or include analytes that are not included in the QSM. For the latter, these are VOC SIM Analyte 1,2,3-trichlorobenzene; SVOC Full Scan analyte 3,4-dimethylphenol.

QAPP Worksheet #20: Field Quality Control Summary

Matrix/Parameter	Analytical Method	FIELD SAMPLES				LABORATORY SAMPLES		
		Field Samples	Equipment Blank[1]	Trip Blanks[2]	Field Duplicates[3]	MS[4]	MSD/ MD[4]	TOTAL ANALYSES[5]
Surface Soil								
SVOCs SIM	SW-846 8270D SIM	20	0	0	2	1	1	24
Metals	SW-846 6010C/6020A/7471B	20	0	0	2	1	1	24
SPLP Metals[6]	SW-846 1312/ 6010C/7470A	4	0	0	1	1	1	7
pH	SW-846 9045D	20	0	0	2	0	0	22
TOC	SW-846 9060A Modified	20	0	0	2	1	1	24
Grain Size	ASTM D 422-63	20	0	0	0	0	0	20
Subsurface Soil								
VOCs	SW-846 8260C	19	2	10	2	1	1	35
SVOCs SIM	SW-846 8270D SIM	19	0	0	2	1	1	23
Metals	SW-846 6010C/6020A/7471B	19	0	0	2	1	1	23
SPLP Metals[6]	SW-846 1312/ 6010C/7470A	4	0	0	1	1	1	7
Fact								
VOCs	SW-846 8260C	67	0	0	7	4	4	82
Background Surface Soil								
SVOCs SIM	SW-846 8270D SIM	20	0	0	2	1	1	24
Metals	SW-846 6010C/6020A/7471B	20	0	0	2	1	1	24
Background Subsurface Soil								
VOCs	SW-846 8260C	20	0	10	2	1	1	34
SVOCs SIM	SW-846 8270D SIM	20	0	0	2	1	1	24
Metals	SW-846 6010C/6020A/7471B	20	0	0	2	1	1	24
Groundwater Overburden								
VOCs	SW-846 8260C	15	1	3	2	1	1	23
VOCs SIM	SW-846 8260C SIM	15	1	3	2	1	1	23
SVOCs SIM	SW-846 8270D SIM	15	1	0	2	1	1	20
Metals/Hg	SW-846 6010C /6020A/7470A	15	1	0	2	1	1	20
Metals/Hg (Field Filtered)	SW-846 /6020A/7470A	15	1	0	2	1	1	20
TOC	SM 5310B	15	0	0	2	1	1	19
Dissolved gasses (M, E, E)	RSK-175	15	0	0	2	1	1	19
Nitrate as N, Nitrite as N, Sulfate, Chloride	SW-846 9056A	15	0	0	2	1	1	19
Sulfide	SM 4500S2 F	15	0	0	2	1	1	19
Alkalinity	SM 2320 B	15	0	0	2	1	1	19

Matrix/Parameter	Analytical Method	FIELD SAMPLES				LABORATORY SAMPLES		
		Field Samples	Equipment Blank[1]	Trip Blanks[2]	Field Duplicates[3]	MS[4]	MSD/ MD[4]	TOTAL ANALYSES[5]
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	15	0	0	0	0	0	15
Ferrous Iron (Field Analysis)	HACH Field Test Kit	15	0	0	0	0	0	15
Groundwater Bedrock								
VOCs	SW-846 8260C	15	1	3	2	1	1	23
VOCs, SIM	SW-846 8260C SIM	15	1	3	2	1	1	23
SVOC SIM	SW-846 8270D SIM	15	1	0	2	1	1	20
Metals/Hg	SW-846/6020A/7470A	15	1	0	2	1	1	20
Metals/Hg (Field Filtered)	SW-846 6010C/6020A/7470A	15	1	0	2	1	1	20
TOC	SM 5310B	15	0	0	2	1	1	19
Dissolved gasses (M, E, E)	RSK-175	15	0	0	2	1	1	19
Nitrate as N, Nitrite as N, Sulfate, Chloride	SW-846 9056A	15	0	0	2	1	1	19
Sulfide	SM 4500S2 F	15	0	0	2	1	1	19
Alkalinity	SM 2320 B	15	0	0	2	1	1	19
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	15	0	0	0	0	0	15
Ferrous Iron (Field Analysis)	HACH Field Test Kit	15	0	0	0	0	0	15
Background Groundwater Bedrock[7]								
VOCs	SW-846 8260C	1	0	0	0	0	0	1
VOCs, SIM	SW-846 8260C SIM	1	0	0	0	0	0	1
SVOC SIM	SW-846 8270D SIM	1	0	0	0	0	0	1
Metals/Hg	SW-846/6020A/7470A	1	0	0	0	0	0	1
Metals/Hg (Field Filtered)	SW-846 6010C/6020A/7470A	1	0	0	0	0	0	1
TOC	SM 5310B	1	0	0	0	0	0	1
Dissolved gasses (M, E, E)	RSK-175	1	0	0	0	0	0	1
Nitrate as N, Nitrite as N, Sulfate, Chloride	SW-846 9056A	1	0	0	0	0	0	1
Sulfide	SM 4500S2 F	1	0	0	0	0	0	1
Alkalinity	SM 2320 B	1	0	0	0	0	0	1
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	1	0	0	0	0	0	1
Ferrous Iron (Field Analysis)	HACH Field Test Kit	1	0	0	0	0	0	1
Surface Water								
VOCs	SW-846 8260C	23	2	5	3	2	2	37

Matrix/Parameter	Analytical Method	FIELD SAMPLES				LABORATORY SAMPLES		
		Field Samples	Equipment Blank[1]	Trip Blanks[2]	Field Duplicates[3]	MS[4]	MSD/ MD[4]	TOTAL ANALYSES[5]
VOCs, SIM	SW-846 8260C SIM	23	2	5	3	2	2	37
SVOC SIM	SW-846 8270D SIM	23	2	0	3	2	2	32
Metals/Hg	SW-846 6010C/6020A/7470A	23	2	0	3	2	2	32
Metals/Hg (Field Filtered)	SW-846 6010C/6020A/7470A	23	2	0	3	2	2	32
Hardness	SM 2340 B (Calculated)	23	2	0	3	2	2	32
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	23	0	0	0	0	0	23
Background Surface Water								
VOCs	SW-846 8260C	10	1	3	1	1	1	17
VOCs, SIM	SW-846 8260C SIM	10	1	3	1	1	1	17
SVOC SIM	SW-846 8270D SIM	10	1	0	1	1	1	14
Metals/Hg	SW-846 6010C/6020A/7470A	10	1	0	1	1	1	14
Metals/Hg (Field Filtered)	SW-846 6010C/6020A/7470A	10	1	0	1	1	1	14
Hardness	SM 2340 B (Calculated)	10	1	0	1	1	1	14
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	10	0	0	0	0	0	10
Pore Water								
VOCs	SW-846 8260C	23	2	5	3	2	2	37
VOCs, SIM	SW-846 8260C SIM	23	2	5	3	2	2	37
SVOC SIM	SW-846 8270D SIM	23	2	0	3	2	2	32
Metals/Hg	SW-846 6010C/6020A/7470A	23	2	0	3	2	2	32
Metals/Hg (Field Filtered)	SW-846 6010C/6020A/7470A	23	2	0	3	2	2	32
Hardness	SM 2340 B (Calculated)	23	2	0	3	2	2	32
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	23	0	0	0	0	0	23
Background Pore Water								
VOCs	SW-846 8260C	10	1	3	1	1	1	17
VOCs, SIM	SW-846 8260C SIM	10	1	3	1	1	1	17
SVOC SIM	SW-846 8270D SIM	10	1	0	1	1	1	14
Metals/Hg	SW-846 6010C/6020A/7470A	10	1	0	1	1	1	14
Metals/Hg (Field Filtered)	SW-846 6010C/6020A/7470A	10	1	0	1	1	1	14
Hardness	SM 2340 B (Calculated)	10	1	0	1	1	1	14
Conductivity, DO, ORP, pH, Temp (Field Measurement)	Multi-Sonde Water Quality Meter	10	0	0	0	0	0	10

Matrix/Parameter	Analytical Method	FIELD SAMPLES				LABORATORY SAMPLES		
		Field Samples	Equipment Blank[1]	Trip Blanks[2]	Field Duplicates[3]	MS[4]	MSD/ MD[4]	TOTAL ANALYSES[5]
Sediment								
VOCs	SW-846 8260C	23	2	10	3	2	2	42
SVOC SIM	SW-846 8270D SIM	23	0	0	3	2	2	30
Metals	SW-846 6010C/6020A/7471B	23	0	0	3	2	2	30
AVS/SEM	EPA 376.3	23	0	0	3	2	2	30
pH	SW-846 9045D	23	0	0	3	2	2	30
TOC	SW-846 9060A Modified	23	0	0	3	2	2	30
Grain Size	ASTM D 422-63	23	0	0	0	0	0	23
Background Sediment								
VOCs	SW-846 8260C	10	1	5	1	1	1	19
SVOC SIM	SW-846 8270D SIM	10	0	0	1	1	1	13
Metals	SW-846 6010C/6020A/7471B	10	0	0	1	1	1	13
AVS/SEM	EPA 376.3	10	0	0	1	1	1	13
pH	SW-846 9045D	10	0	0	1	1	1	13
TOC	SW-846 9060A Modified	10	0	0	1	1	1	13
Grain Size	ASTM D 422-63	10	0	0	0	0	0	10

Notes:
MD - Matrix Duplicate
MS - Matrix Spike
MSD - Matrix Spike Duplicate
SIM - Selected Ion Monitoring
SVOCs - Semivolatile Organic Compounds
TOC - Total Organic Carbon
VOCs - Volatile Organic Compounds
1 - Five percent of field samples per type of equipment for analyses and matrices shown. Equipment blanks will not be collected or analyzed if disposable equipment is used. With the possible exception of VOCs, it is unlikely that significant contamination will be detected in the aqueous equipment blanks for soil samples relative to the concentrations. Even if significant contamination were to be detected, the positive bias would likely be small relative to the bias introduced by the biased sampling designs. As such, non-VOC analysis will not be analyzed in the aqueous equipment blanks.
2 - One per cooler containing VOC samples. Assumes 1 cooler of VOC samples shipped per day of sampling.
3 - Ten percent of field samples, not including field QC samples, for the analyses and matrices shown.
4 - Five percent of field samples (not including field QC samples) per medium for methods required.
5 - Total includes MS, MSD, or MD.
6 - The total metals results will be divided by 20 and compared to the RIDEM GA Leachability Criteria. Results greater than the RIDEM GA Leachability Criteria will be selected for SPLP analysis. It is estimated that twenty percent of the samples will require SPLP metals analysis.
7 – Additional field QC samples are not required for the background bedrock groundwater sample because the number of QC samples planned for bedrock groundwater accounts for the additional background sample.

QAPP Worksheet #21: Field Standard Operating Procedures Table

Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Yes/No)	Comments
SOP 3-01	Utility Clearance	AECOM	Flagging for locations, utilities; Additional equipment provided by the subcontractor/agency	No	
SOP 3-02	Logbooks	AECOM	Field logbook and field forms	No	
SOP 3-03	Recordkeeping, Sample Labeling, and Chain-of-Custody	AECOM	Sample labels, pen with indelible ink, and sample attribute forms, CoC forms,	No	
SOP 3-04	Sample Handling, Storage, and Shipping	AECOM	CoCs, custody seals, ice, cooler, resealable bags, bubble wrap, air bills	No	
SOP 3-05	Investigation-Derived Waste Management	AECOM	DOT-approved drums or other containers, 5 gallon buckets, PID, labeling material	No	
SOP 3-06	Equipment Decontamination	AECOM	Plastic sheeting, buckets, potable water, DI water, isopropanol, Alconox/Liquinox	No	
SOP 3-07	Land Surveying	AECOM	Total station, GPS receivers, handheld tablets, digital levels, subsurface locators, GIS and surveying software	No	
SOP 3-10	Surface Water Sampling	AECOM	Dipper and extension pole and laboratory-supplied sample containers	No	
SOP 3-12	Monitoring Well Installation	AECOM	DPT or Sonic drill rig, PID, water level meter, water quality meter, well screen, casing, riser, submersible pump	No	
SOP 3-13	Monitoring Well Development	AECOM	PID, submersible pump, tubing, water level meter, water quality meter, power source, 55-gallon drums	No	
SOP 3-14	Monitoring Well Sampling	AECOM	PID, submersible pump, tubing, water level meter, water quality meter, power source, laboratory-supplied sample containers	No	
SOP 3-15	Monitoring Well and Borehole Abandonment	AECOM	Plastic sheeting, buckets, water	No	
SOP 3-16	Soil and Rock Classification	AECOM	DPT or Sonic drill rig, field logbook, ruler, tape measure, grain size chart, Munsell color chart	No	

Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Yes/No)	Comments
SOP 3-18	Field Analysis of Ferrous Iron Using the HACH DR890 Colorimeter and HACH Method 8146	AECOM	HACH DR/890 colorimeter, HACH "AccuVac ampuls," HACH colorimeter sample cell with cap, Plastic cups and lids, Graduated cylinders, Kimwipes®, Pipette, Deionized water for dilutions	No	
SOP 3-20	Operation and Calibration of a Photoionization Detector	AECOM	PID, calibration gas, tedlar bag	No	
SOP 3-21	Surface and Subsurface Soil Sampling Procedures	AECOM	Sampling trowels, PID stainless steel bowls, field logbook, flagging, laboratory-supplied sample containers	No	
SOP 3-22	Sediment Sampling	AECOM	Stainless steel trowel, spoon, spatula, or scoop; hand corer, wetland sampler	No	
SOP 3-24	Water Quality Parameter Testing for Groundwater Sampling	AECOM	submersible pump, tubing, water level meter, water quality meter, power source	No	
SOP 3-35	In-Situ Hydraulic Conductivity Testing via Rising or Falling Head Slug Testing	AECOM	Boring logs, Well construction diagrams, Well development logs, Water level meter, Slug (bailer or solid cylinder), Nylon string, Water level indicator, Pressure transducer(s), Data logger(s), Computer with appropriate software, Plastic sheeting	No	
SOP 3-45	Pore Water Sampling	AECOM	Pore water push-point sampler (e.g., Henry Sampler), peristaltic pump, Teflon and silicone tubing, syringe	No	

Notes:
FID = Flame ionization detector
GPS = Global Positioning System
PID = Photoionization detector
POP = Project Operating Procedure
SOP = Standard Operating Procedure
USEPA = United States Environmental Protection Agency
VOC = Volatile Organic Compound

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
PID	Inspection	Visually inspect upon receipt for damage, cleanliness, missing parts, etc. Basic functional checks to ensure proper calibration and operating capacity.	Field Team Leader (FTL) or designee	Upon receipt and daily as needed	Undamaged; no missing parts; calibration record present; functions properly	Replace or service instrument
	Calibration	SOP 3-20		Initial: Each time the instrument is turned on or if the instrument gives erratic results. Check: At the end of the day	± 10% of standard	Replace or service instrument; data may be flagged
	Maintenance	Charge batteries. Refer to manufacturer's instructions.		As needed	Functions properly/ acceptable calibration	Replace or service instrument
	Testing	Check functionality		Daily, prior to use, and as needed	Functions properly	Replace or service instrument
Multi-probe water quality meter	Inspection	Visually inspect upon receipt for damage, cleanliness, missing parts, etc. Basic functional checks to ensure proper calibration and operating capacity. Probes inspected to ensure free of debris and that membranes are free of tears.	FTL or designee	Daily, prior to use	Undamaged; no missing parts; calibration record present; functions properly; DO membrane free of tears	Clean; replace DO membrane
	Calibration	SOP 3-24		Initial: Each time the instrument is turned on or if the instrument gives erratic results. Check: At the end of the day	As described in SOP	Replace or service instrument; data may be flagged
	Maintenance	Charge batteries. Refer to manufacturer's instructions.		As needed	Functions properly/ acceptable calibration	Replace or service instrument
	Testing	Check functionality		Daily prior to use	Functions properly	Replace or service instrument
Turbidity meter	Inspection	Visually inspect upon receipt for damage, cleanliness, missing parts, etc. Basic functional checks to ensure proper calibration and operating capacity.	FTL or designee	Upon receipt and daily as needed	Undamaged; no missing parts; calibration record present; functions properly	Replace or service instrument
	Calibration	SOP 3-24		Initial: Each time the instrument is turned on or if the instrument gives erratic results. Check: At the end of the day	As described in SOP	Replace or service instrument; data may be flagged
	Maintenance	Charge batteries. Refer to manufacturer's instructions.		As needed	Functions properly/ acceptable calibration	Replace or service instrument

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
	Testing	Check functionality		Daily, prior to use, and as needed	Functions properly	Replace or service instrument
Water level meter	Inspection	Visually inspect upon receipt for damage, cleanliness, missing parts, etc. Basic functional checks to ensure proper calibration and operating capacity.	FTL or designee	Upon receipt and daily as needed	Undamaged, no missing parts, functions properly	Replace or service instrument
	Accuracy Check	Measure length against a measuring tape and line. Ensure no segments have been cut and removed.		Upon receipt	Water level meter tape is not off by more than 0.1 foot.	Replace or service instrument; data may be flagged
	Maintenance	Charge batteries. Refer to manufacturer's instructions.		As needed	Functions properly/ acceptable calibration	Replace or service instrument
	Testing	Check functionality		Daily, prior to use, and as needed	Functions properly	Replace or service instrument
Global Positioning System Trimble Geo XT (or similar)	Inspection	Visually inspect upon receipt for damage. Ensure TerrSync software (or similar) is installed and operating.	FTL or designee	Upon receipt	Undamaged, no missing parts, functions properly	Replace or service instrument. If accuracy is not met with Trimble Geo XT, then Carlson GPS Network Rovers may in used to improve accuracy.
	Calibration	Manufacturer's Manual and SOP 3-07		As needed	As described in SOP	
	Maintenance	As needed		As needed	Functions properly	
	Testing	As needed		As needed	Functions properly	
Ferrous Iron Test Kits	Inspection	Visually inspect upon receipt for damage, cleanliness, missing parts, etc. Basic functional checks to ensure proper calibration and operating capacity.	FTL or designee	Upon receipt and daily as needed	Undamaged, no missing parts, functions properly	Replace or service instrument
	Calibration	SOP 3-18		Initial: Each time the instrument is turned on or if the instrument gives erratic results. Check: At the end of the day	As described in SOP	Replace or service instrument; data may be flagged
	Maintenance	Charge batteries. Refer to manufacturer's instructions.		As needed	Functions properly/ acceptable calibration	Replace or service instrument
	Testing	Check functionality		As needed	Functions properly	Replace or service instrument

QAPP Worksheet #23: Analytical Standard Operating Procedures Table

Laboratory/ SOP Number	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM[1]	Modified for Project Work? (Y/N)
Katahdin Analytical Services						
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, 10/18, Revision 19.	Definitive	Water and Sediment / VOCs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	No Variance	N
CA-213	Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified for Selected Ion Monitoring (SIM), 01/19, Revision 15.	Definitive	Water and Sediment / SVOCs and PAHs	GC/MS	No Variance	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	Sediment / VOCs	Not applicable (extraction)	No Variance	N
CA-220	Analysis of Volatile Organic Compounds by Purge And Trap GC/MS SW-846 Method 8260 – Modified For Selected Ion Monitoring (SIM), 03/19, Revision 15.	Definitive	Aqueous / VOCs	GC/MS	No Variance	N
CA-226	Analysis of SVOAs by Capillary Column GC/MS: SW-846 Method 8270D, 06/17, Revision 10. (Reviewed 01/19)	Definitive	Water and Sediment / SVOCs	GC/MS	No Variance	N
CA-336	Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Technique EPA SOP RSK-175, 03/19, Revision 7.	Definitive	Water / MEE	Gas Chromatography (GC)/ Flame Ionization Detector (FID)	No Variance	N
CA-502	Preparation of Aqueous Samples for Extractable Semivolatile Analysis, 03/19, Revision 12.	Definitive	Water / SVOCs and PAHs	Not applicable (extraction)	No Variance	N
CA-512	Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis, 04/19, Revision 14.	Definitive	Sediment / SVOCs and PAHs	Not applicable (extraction)	No Variance	N

Laboratory/ SOP Number	**Title, Revision Date, and Number**	**Definitive or Screening Data**	**Matrix and Analytical Group**	**Instrument**	**Variance to QSM[1]**	**Modified for Project Work?** (Y/N)
CA-526	Preparation of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis, 04/19, Revision 12.	Definitive	Sediment / SVOCs and PAHs	Not applicable (extraction)	No Variance	N
CA-551	Grain Size Analysis, 04/19, Revision 2.	Definitive	Solid / grain size	Sieves/Hydrometer	No Variance	N
CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals, 01/19, Revision 9.	Definitive	Water / TAL Metals and TAL Metals + Boron	Not applicable (digestion)	No Variance	N
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA, 01/19, Revision 8.	Definitive	Sediment / TAL Metals	Not applicable (digestion)	No Variance	N
CA-608	Trace Metals Analysis By ICP-AES Using EPA Method 6010, 01/19, Revision 19.	Definitive	Water and Sediment / TAL Metals	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	No Variance	N
CA-611	Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471, 01/19, Revision 12.	Definitive	Sediment / Mercury	Mercury Analyzer	No Variance	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470, 01/19, Revision 11.	Definitive	Water / Mercury	Mercury Analyzer	No Variance	N
CA-620	Synthetic Precipitation Leaching Procedure (SPLP) for Inorganic and Non-Volatile Organic Analytes	Definitive	Soil / Metals	Not Applicable (leaching)	No Variance	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020, 01/19, Revision 13.	Definitive	Water and Sediment / TAL Metals and TAL Metal	ICP-MS	No Variance	N
CA-709	pH Concentration Measurements In Soil Matrices – SW 846 Method 9045, 08/16, Revision 11. (Reviewed 07/18)	Definitive	IDW / Corrosivity	pH Meter	No Variance	N
CA-722	Trimetric Determination of Sulfide Using EPA Method 376.1, SM4500S2- F, SW846 9034 and SW846 7.3.4, 12/17, Revision 8. (Reviewed 01/19)	Definitive	Water / Sulfide	Buret	No Variance	N

Laboratory/ SOP Number	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM[1]	Modified for Project Work? (Y/N)
CA-738	Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments, 01/19, Revision 4.	Definitive	Sediments / Acid Volatile Sulfide and Simultaneously Extractable Metals	Buret – Acid Volatile sulfides ICP – Simultaneously Extractable Metals	No Variance	N
CA-739	Titrimetric Determination of Total Alkalinity by EPA Method 310.1 and SM 2320 B using the Mettler Dl25 Autotitrator, and Calculation of the Component Forms of Alkalinity by SM 4500–$C0_2$ D, 07/18, Revision 13.	Definitive	Water / Alkalinity	Autotitrator	No Variance	N
CA-741	Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method and SW846 9060 Mod, 01/19, Revision 8.	Definitive	Water / Total Organic Carbon	Total Organic Carbon Analyzer	No Variance	N
CA-742	Anions by Ion Chromatography (IC) – Method 300.0, 12/17, Revision 11. (Reviewed 02/19)	Definitive	Water / Anions	Ion Chromatograph	No Variance	N
CA-763	Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B, 06/17, Revision 10. (Reviewed 02/19)	Definitive	Water / Total Organic Carbon	Total Organic Carbon Analyzer	No Variance	N
SD-902	Sample Receipt and Internal Control, 01/19, Revision 13.	NA	NA	NA	NA	N
SD-903	Sample Disposal, 09/17, Revision 6. (Reviewed 01/19)	NA	NA	NA	NA	N

Notes:
1. Laboratory address, point of contact and contact information is presented in the footnotes of Worksheet #19 & 30.
2. The laboratory holds current DoD ELAP accreditation for all definitive analyses presented that are included in the DoD QSM. Certificates are presented in Appendix D.

GC/MS = gas chromatography;/mass spectrometry

QAPP Worksheet #24: Analytical Instrument Calibration Table - Katahdin Analytical Services, LLC (1,2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS-VOCs	Initial Calibration (ICAL) - A minimum 5-point initial calibration is required for all VOCs.	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.	Analyst, Department Manager	CA-202
	Establish Retention Time 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.	Analyst, Department Manager	
	Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	Second Source ICAL 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.	Analyst, Department Manager	
	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.	Analyst, Department Manager	
	4-Bromofluorobenze (BFB) Tune	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or decafluorotriphenylphosphine (DFTPP) from method.	Retune instrument and verify.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS (full scan) SVOCs GC/MS (SIM) PAHs, Pentachlorophenol and 1,4-Dioxane	ICAL - A minimum 5-point calibration is required for all SVOCs.	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.	Analyst, Department Manager	CA-213, CA-226
	Establish Retention Time 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.	Analyst, Department Manager	
	Evaluation of RRT	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	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.	Analyst, Department Manager	
	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.	Analyst, Department Manager	
	DFTPP Tune	Every 12 hours	Criteria listed in Section 7.4, current revision of SOPs CA-213 and CA-226	Retune and/or clean source.	Analyst, Department Manager	
GC/FID-Methane, Ethane, Ethene	ICAL	Instrument receipt, major instrument change, when CCV does not meet criteria	Average %RSD must be ≤ 20	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	CA-336
	ICV	Immediately following calibration.	The %D of the expected value must be ≤20%for all analytes.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	CCV	If initial calibration analyzed, daily and after 20 samples, and at end of sequence.	All reported analytes within ± 20% of true value.	Assess the samples: If the %RPD >20% and sample results are < LOQ, narrate. If %RPD >20% and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after last acceptable CV.		
ICP-AES –Metals	ICAL - 1 point calibration plus blank	Daily ICAL prior to sample analysis.	One point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-608
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110% for all project constituents.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Calibration Blank (CB)	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze CB. All samples following the last acceptable CB must be reanalyzed.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project constituents.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	Low-level Calibration Check Standard (if using one-point ICAL)	Daily after one-point ICAL.	%R must within 80%-120% for all project constituents.	Correct problem, then reanalyze.	Analyst, Department Manager	
	ICS - ICSA & ICSB	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICSB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	
ICP-MS –Metals	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.	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.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	ICV	Once after each ICAL, and before beginning a sample run.	%R must be within 90-110% for all project constituents.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project constituents.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	ICS - ICSA & ICSB	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICS-AB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	
Mercury analyzer	ICAL - 5 points plus a CB	Upon instrument receipt, major instrument change, at the start of each day.	Correlation coefficient (r) must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-611, CA-615
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110%	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze CB. All samples following the last acceptable CB must be reanalyzed.	Analyst, Department Manager	
	CCV	Beginning and end of each run sequence and every 10 samples.	%R must be within 90-110%	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Ion Chromatography / Chloride, Nitrate, Nitrite, Sulfate	ICAL – A minimum of a 5-point calibration is prepared.	Prior to sample analysis.	Correlation coefficient (r) must be ≥0.995.	Correct problem and rerun calibration.	Analyst, Department Manager	CA-742
	ICV	Once after each ICAL prior to sample analysis.	The %R must be within 90-110% of true value and retention times (RTs) must be within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of the sequence.	The %R must be within 90-110% of true value and all project analytes must be within established RT windows.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
Buret – Sulfide	Standardization	Daily prior to sample analysis.	Standardized using 0.25 N Sodium thiosulfate	An acceptable titrant is compared against an independent source identified as an LCS/ICV (see next line)	Analyst, Department Manager	CA-722
	CCV	At beginning and end of each run sequence and every 10 samples	80-120 %	If the Continuing Calibration Verification fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable Continuing Calibration Verification recovery.	Analyst, Department Manager	
Autotitrator / Alkalinity, Bicarbonate	CCV	One after every 10 samples	%R must within 80%-120%	(1) If the CCV fails high, report samples that are <LOQ (2) Recalibrate and/or reanalyze other samples.	Analyst, Department Manager	CA-739
pH Meter (Corrosivity	ICAL	Three to five point calibration with pH buffers daily	N/A	N/A	Analyst, Department Manager	CA-709
	CCV	At beginning of run, after every 10 samples and at end of run.	90% - 110%	Recalibrate and reanalyze samples back to last acceptable continuing calibration.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Total Organic Carbon Analyzer / Total Organic Carbon	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) must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	CA741, CA-763
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must within 80%-120% (soil), 90%-110% (water)	(1) If the ICV fails high, report samples that are <LOQ. (2) Redigest, recalibrate and/or reanalyze other samples.	Analyst, Department Manager	
	CCV	Every 10 samples and at the end of the run	%R must within 80%-120% (soil), 90%-110% (water)	If the CCV fails high, report samples that are <LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.	Analyst, Department Manager	
Spectrophotometer / AVS	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	Prior to sample analysis	Correlation coefficient (r) must be ≥ 0.995.	Investigate source of problem, Recalibrate	Analyst, Department Manager	CA-738
	ICV	One of each per prep batch	80-120 %R	Recalibrate and reanalyze sample batch	Analyst, Department Manager	
	CCV	At beginning of run, after every 10 samples and at the end of the run	80-120 %R	Reanalyze all samples back to last acceptable CCV recovery	Analyst, Department Manager	
Grain Size	Sieve – Visual inspection	Every use	No clogging or tears in mesh	Remove from service	Analyst, Department Manager	CA-551

Notes:

1. Refer to the Analytical SOP References table (Worksheet 23#).
2. LOD/LOQ verification procedures presented in the DoD QSM will be modified for this project in the following manner: Katahdin will make an effort to prioritize analysis of the LOD and LOQ verifications for the analyses in this project such that LODs and LOQs are verified prior to sample analysis. If LOD/LOQ verification cannot be analyzed for the quarter prior to sample receipt in that quarter, the required LOD/LOQ verification will be analyzed with the project samples. If LOD/LOQ verification is not successful but batch QC requirements are otherwise met, the data will be reported with and the failed LOD/LOQ verification will be identified in the case narrative. AECOM will then consider the verification failure as part of data validation (Refer to WS #36).

ICS = Interference Check Solution
LOD – Limit of Detection

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table- Katahdin Analytical Services, LLC

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS VOCs	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in lab Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-202 Full Scan; CA-220 SIM
GC/MS SVOCs (full scan and SIM)	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	SVOCs; 1,4-dioxane, pentachlorophenol and PAHs	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-213, CA-226
Sieves	Cleaning	grain size	Visual inspection for clogs or tears	Each use	N/A	Remove from service	Analyst, Department Manager	CA-551
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TAL Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-608
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-611, CA-615

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP-MS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TAL Metals	Torch, nebulizer, spray chamber, pump tubing.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-627
Probe - Ion-Selective Electrode, pH,	Clean, drain, and refill reference electrode as needed.	pH	Reference electrode for white crystals, Inspect electrode for damage.	Before use	pH 7 ± 0.05 pH units (pH)	Correct problem and repeat calibration.	Analyst, Department Manager	CA-709
Buret - Sulfide,	N/A	Sulfide	Visual inspection for cracks or chips	Each use	N/A	Remove from service	Analyst, Department Manager	CA-722
Autotitrator	Fill pH electrode as needed, clean stirring paddle weekly, fill rinse as needed.	Alkalinity	pH electrode, stirring paddle, reagent bottles.	As necessary	Acceptable CCV	Correct the problem reanalyze CCV.	Analyst, Department Manager	CA-739
TOC Combustion Analyzer	Check level of dilution water, drain vessel water, humidifier water, auto sampler rinse water and phosphoric acid vessel and fill as needed. Replace oxygen cylinder.	Total Organic Carbon	Tubing, sample boat, syringe, humidifier, rinse reservoir, phosphoric acid vessel, oxygen pressure	Prior to initial calibration and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-741, CA-763
Ion Chromatograph	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.	Chloride, Sulfate, Ortho-Phosphorus, Nitrite, Nitrate	Tubing, column, suppressor.	Prior to initial calibration and/or as necessary.	Passing ICAL or CCV.	Correct problem and repeat calibration or CCV.	Analyst, Department Manager	CA-742

Notes:
1. Refer to the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action, Volatile Organic Compounds, Full Scan

Matrix	Aqueous and Solids
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260C/ CA-202

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	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.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoro-methane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluoro-benzene	QSM Appendix C limits	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	QSM Appendix C Limits	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	QSM Appendix C Limits RPD of all analytes ≤ 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260C/ CA-202

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Internal Standard (IS)	Four per sample: Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank

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QAPP Worksheet #28-2: Analytical Quality Control and Corrective Action – Volatile Organic Compounds, SIM

Matrix	Aqueous
Analytical Group	VOCs SIM
Analytical Method/ SOP Reference	SW-846 8260CSIM/ CA-220

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	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.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoromethane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene (BFB)	Laboratory Limits: 70-130%R	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	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	Laboratory Limits: 70-130%R	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	Same as LCS RPD of all analytes ≤20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous
Analytical Group	VOCs SIM
Analytical Method/ SOP Reference	SW-846 8260CSIM/ CA-220

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Internal Standard (IS)	Four per sample: Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank

QAPP Worksheet #28-3: Analytical Quality Control and Corrective Action – Semivolatile Organic Compounds, Full Scan and SIM

Matrix	Aqueous and Solids
Analytical Group	SVOCs and SVOCs SIM
Analytical Method/ SOP Reference	SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	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.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids
Analytical Group	SVOCs and SVOCs SIM
Analytical Method/ SOP Reference	SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Surrogate	Full Scan - 6 per sample: 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14 SIM 2-Methylnaphthalene-d10 Fluorene-d10 Pyrene-d10 2,4-dibromophenol-d3	QSM Appendix C limits and in-house limits for 2-Methylnaphthalene, Fluorene-d10, Pyrene-d10, and 2,4-Dibromophenol-d3: 10-130 (Aq); 20-116 (Soil)	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	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	QSM Appendix C Limits and in-house limits for 1,4-Dioxane (30-150% Soil); (10-93% Aq), Benzoic acid (10-70% Soil); (10-151% Aq), Phenol (10-78% Aq)	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	QSM Appendix C Limits , and in-house LCS limits For 1,4-Dioxane, Benzoic acid, and Phenol (Aq) RPD of all analytes ≤ 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids
Analytical Group	SVOCs and SVOCs SIM
Analytical Method/ SOP Reference	SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
IS	Six per sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	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 or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank
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QAPP Worksheet #28-4: Analytical Quality Control and Corrective Action – Dissolved Gases

Matrix	Aqueous
Analytical Group	Methane, Ethane, Ethene
Analytical Method/ SOP Reference	RSK-175 / CA-336

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of 20 or fewer samples.	No analytes detected >1/2 LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	Analyst, Department Manager	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	Limits in QSM Appendix C	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.	Analyst, Department Manager	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One MS/MSD as requested by clients.	Recovery: Same as LCS RPD ≤ 30% between MS and MSD.	Assess the samples and associated QC. If the LCS is acceptable, narrate. If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.	Analyst, Department Manager	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.

MB – method blank
QSM – Quality Systems Manual

QAPP Worksheet #28-5: Analytical Quality Control and Corrective Action – Metals and SPLP Metals (ICP-AES)

Matrix	Aqueous and Solids
Analytical Group	Metals, SPLP Metals (ICP-AES)[1]
Analytical Method/ SOP Reference	SW-846 6010C / CA-608

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	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.	Analyst, Laboratory Department Manager and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	QSM Appendix C Limits	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	QSM Appendix C Limits RPD of all analytes ≤ 20% (between MS and MSD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids
Analytical Group	Metals, SPLP Metals (ICP-AES)[1]
Analytical Method/ SOP Reference	SW-846 6010C / CA-608

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

1. Hardness is calculated from results generated by this analysis. No additional QC acceptance limits or MPCs apply to hardness.

QSM – Quality Systems Manual

QAPP Worksheet #28-6: Analytical Quality Control and Corrective Action – Metals (ICP-MS)

Matrix	Aqueous
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020A / CA-627

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	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.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	QSM Appendix C Limits	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	QSM Appendix C Limits RPD of all analytes ≤ 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution (not applicable for rinsate blanks)	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.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020A / CA-627

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
IS	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity must be within 30-120% of that of initial calibration standard.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Per the method, for each sample, IS intensity must be ≥ 70% of that of initial calibration standard.

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QAPP Worksheet #28-7: Analytical Quality Control and Corrective Action – Metals and SPLP (Mercury)

Matrix	Aqueous and Solids
Analytical Group	Metals, SPLP Metals (Mercury)
Analytical Method/ SOP Reference	SW-846 7470A/7471B/ CA-611, CA-615

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	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.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	AQ: 82-119 %R SL: 80-124	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	Same as LCS RPD of all analytes ≤ 20% (between MS and MSD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

MB – method blank

QAPP Worksheet #28-8: Analytical Quality Control and Corrective Action - Anions

Matrix	Aqueous
Analytical Group	Anions – Chloride, Nitrite, Nitrate, Sulfate
Analytical Method/ SOP Reference	SW-846 9056A / CA-742

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per analytical batch of 20 or fewer samples.	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 the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within: Cl 87-111 NO3 88-111 NO2 87-111 SO4 87-112	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 (see full explanation in Appendix F).	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One set for every set 20 samples	Same as LCS	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤ 20%	Correct problem and reanalyze sample and duplicate. Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

LOD – Limit of Detection

QAPP Worksheet #28-9: Analytical Quality Control and Corrective Action - Sulfide

Matrix	Aqueous
Analytical Group	SM4500-S2-F
Analytical Method/ SOP Reference	Sulfide / CA-722

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager, QA Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, QA Manager and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	%R must be within: 75-125	(1) Assess the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, QA Manager and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per ten samples	RPD <10 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	(1) If RPD is outside criteria report original result with notation or narration.	Analyst, Laboratory Department Manager, QA Manager and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

LOD – Limit of Detection

QAPP Worksheet #28-10: Analytical Quality Control and Corrective Action - Alkalinity

Matrix	Aqueous
Analytical Group	Alkalinity
Analytical Method/ SOP Reference	SM2320B / CA-739

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > LOQ and > 1/10 the amount measured in any sample.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	Investigate source of problem. If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	%R must be within: 80-120	Assess the samples and associated QC: i.e. If the LCS results are acceptable, narrate. If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	Investigate problem and reanalyze sample in duplicate If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

LOD – Limit of Detection

QAPP Worksheet #28-11: Analytical Quality Control and Corrective Action – pH

Matrix	Solids
Analytical Group	pH
Analytical Method/ SOP Reference	SW-846 9045D/ CA-709

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
LCS	One per analytical batch of 20 or fewer samples.	%R must be ± 0.1 pH units	Investigate source of problem. If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD $\leq$20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	Investigate problem and reanalyze sample in duplicate If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

QAPP Worksheet #28-12: Analytical Quality Control and Corrective Action – Total Organic Carbon in Soil

Matrix	Solids
Analytical Group	Total Organic Carbon
Analytical Method/ SOP Reference	SW846 9060A / CA-741

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	Investigate source of problem. If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	Same as LCS	Assess the samples and associated QC: i.e. If the LCS results are acceptable, narrate. If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Quadruplicate	One sample quadruplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	Investigate problem and reanalyze sample in quadruplicate If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

LOD – Limit of Detection

QAPP Worksheet #28-13: Analytical Quality Control and Corrective Action – Total Organic Carbon in Water

Matrix	Water
Analytical Group	Total Organic Carbon
Analytical Method/ SOP Reference	SW846 9060 / CA-763

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	Investigate source of problem. If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	Same as LCS	Assess the samples and associated QC: i.e. If the LCS results are acceptable, narrate. If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	Investigate problem and reanalyze sample in duplicate If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

LOD – Limit of Detection

QAPP Worksheet #28-14: Analytical Quality Control and Corrective Action – AVS/SEM

Matrix	Solids
Analytical Group	AVS/SEM
Analytical Method/ SOP Reference	EPA 376.3 / CA-738

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per prep batch of 20 or fewer samples	No analyte detected >LOQ	Investigate source of contamination. Reprep and analyze method blank and all samples processed with the contaminated blank	Analyst, Laboratory Department Manager, and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One of each per prep batch	80-120 %R	Recalibrate and reanalyze sample batch	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.
MS	One for every set of 10 samples	75-125 %R	Notate sample result in raw data with Notation I-1	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per twenty samples or per batch	RPD $\leq$20	If lab QC in criteria and matrix interference suspected, flag data. Else, reanalyze	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

QAPP Worksheet #28-15: Analytical Quality Control and Corrective Action – Grain Size

Matrix	Solid
Analytical Group	Grainsize
Analytical Method/ SOP Reference	ASTM D422 / CA-551

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
N/A: Laboratory QC samples are not planned for grain size analysis.						

QAPP Worksheet #29: Project Documents and Records Table

Document	Where Maintained
Sample Collection Documents and Records Project personnel sign-off record Field logbook (and sampling notes) Field sample forms (e.g., soil gas, sub-slab and indoor air sample log sheets, etc.) Chain-of-custody records Equipment calibration logs Photographs QAPPs including field sampling SOPs Safe work assessment and permit forms	Sample collection documents and records (may include printed copy as well as electronic information) will be maintained at the AECOM office at 250 Apollo Drive, Chelmsford, Massachusetts 01824.
Analytical Results Documents and Records Sample receipt/log-in forms Sample preparation logs Equipment calibration logs Sample analysis run logs Reported field sample results Reported results for standards, quality control checks Reported results for standards, quality control samples Data validation memoranda	Analytical results, documents and records will be provided by the laboratory in electronic formats. Although available in the Administrative Record (AR) file, laboratory reports are typically filed at a separate location and are available upon request. Electronic analytical results will also be verified, entered, and maintained in a password protected database.
Other Documents Personnel training records Health and Safety certifications Health and Safety Plan Field Sampling Audit Checklist Letter reports, technical memos Analytical Audit Checklist	Personnel training records and health and safety certificates will be stored in personnel records and electronically in the AECOM training database in the project file at 250 Apollo Drive, Chelmsford, Massachusetts 01824. Field Audit Checklists are not considered part of the AR file and will be stored in the AECOM office at 250 Apollo Drive, Chelmsford, Massachusetts 01824, and electronically in the server library. Analytical Audit Checklists will be retained by the respective accreditation authorities.
Final Document/Records Repository AR files Site files Post decision files Analytical data Spatial data Maps	EPA is responsible for storing and maintaining final documents in accordance with requirements under the Superfund Program.

QAPP Worksheets #31, 32 & 33: Assessments and Corrective Actions Table

Assessments:

Assessment Type	Responsible Party & Organization	Number / Frequency	Estimated Dates	Assessment Deliverable[1]	Deliverable[1] Due Date
On-Site Laboratory Systems Audit (External TSA)	DoD ELAP Accrediting Body	Every 18 months	TBD	Verbal debriefing, Written audit report	Specified by DoD ELAP Accrediting Body
On-Site Laboratory Systems Audit (External TSA)	NELAP Auditor	Every 2 years	TBD	Verbal debriefing, Written audit report	Specified by NELAP Program
Proficiency Testing (External TSA)	NELAP Program	Every 6 months; passing 2 of every 3 consecutive PTs	TBD	PT Scores Change to Accreditation Status	21 days after study close date 60 days after release of PT scores
On-Site Laboratory Systems Audit (Internal TSA)	Laboratory QA Manager	Annually	TBD	Corrective Action Reports	30 days after completion of Internal Audit
Data Validation (External TSA)	AECOM Data Validation Coordinator	Per SDG Scheduled for Validation	7 weeks after completion of the sampling event[1]	Validation Deliverable uploaded to ADR Module in FUDSChem	**7 weeks after completion of the sampling event[1]**
Data Package (External TSA)	AECOM Project Chemist	Per SDG Scheduled for Validation	10 weeks after completion of the sampling event[1]	Data Approved in FUDSChem	**10 weeks after completion of the sampling event[1]**
Technical Quality Review (TQR, Internal TSA)	AECOM TOM	Per formal deliverable	Prior to deliverable release	Deliverable Review and Approval Records	Prior to deliverable release
Quality Management System Audit (Internal TSA)	AECOM Program QA/QC Manager (or designee)	TBD	TBD	Quantitative Assessment Ranking	TBD
On-Site Field Sampling Systems Audit (Internal TSA)	AECOM Task Order Quality Representative (or designee)	TBD	TBD	Verbal debrief and written memo	Debrief immediately following audit; memo within 7 days of audit completion

Assessment Type	Responsible Party & Organization	Number / Frequency	Estimated Dates	Assessment Deliverable[1]	Deliverable[1] Due Date
Field Progress Report[2]	AECOM Field Team Lead	Daily (verbal); Summary every 1-2 days (email)	During field work	None	Not applicable
Status Report/Status Conference Call[2]	AECOM TOM	Monthly	June 30, 2019 and the 30th of following months until December 26, 2020	Status Report	Every 4 business weeks from Notice to Proceed
Project Schedule[2]	AECOM TOM	Initial then Monthly	June 29, 2019; and the 30th of following months until December 26, 2020	Project Schedule	**2 business weeks from Notice to Proceed, monthly thereafter**

Notes:

1. The term "deliverable" in this case refers to documentation and not necessarily to a deliverable required as part of the task order (TO) project work scope. Items in **bold** are deliverables to be submitted to CENAE.
2. Although not assessments, these status reports provide information to internal and external management on project schedule, scope and budget.

Assessment Response and Corrective Action:

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
DoD ELAP On-Site Laboratory Audit	Laboratory Quality Assurance Manager	Dependent on accrediting agency	Dependent on accrediting agency	Laboratory Quality Assurance Manager	Accrediting body
NELAP On-Site Laboratory Audit	Laboratory Quality Assurance Manager	Dependent on accrediting agency	Dependent on accrediting agency	Laboratory Quality Assurance Manager	Accrediting body
NELAP Proficiency Testing	Laboratory Quality Assurance Manager	Dependent on accrediting agency	Dependent on accrediting agency	Laboratory Quality Assurance Manager	Accrediting body
Internal On-Site Laboratory Systems Audit	Annually	TBD	Corrective Action Reports	30 days after completion of Internal Audit	Annually
Data Validation	Laboratory Project Manager	Resubmittal and/or reanalysis and/or Corrective Action Report (depending on nature and severity of issue)	CAR 7 calendar days	Laboratory Quality Assurance Manager	AECOM Project Chemist
Data Approval	Laboratory Project Manager	Resubmittal and/or reanalysis and/or Corrective Action Report (depending on nature and severity of issue)	7 calendar days from notification	Laboratory Quality Assurance Manager	AECOM Project Chemist
Quality Management System Audit (Internal TSA)	AECOM TOM	Quantitative Assessment Ranking	TBD	AECOM TOM	AECOM Task Order Quality Representative

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
On-Site Field Sampling Systems Audit (Internal TSA)	AECOM TOM	Corrective actions as needed	Dependent on nature of finding; Within 7 days of audit completion	Field Team Lead	AECOM Task Order Quality Representative

Note: Delivery dates assume receipt of laboratory data no later than 21 days after sample receipt

QAPP Worksheet #34: Data Verification and Validation Inputs

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	X	
2	Contract	X	
3	Subcontract agreements	X	
4	Field SOPs	X	
5	Laboratory SOPs	X	
Field Records			
6	Field logbooks/ sample collection forms/ daily reports	X	X
7	Equipment calibration records	X	
8	Instrument testing and inspection records	X	
9	Chain-of-custody forms	X	X
10	Sampling diagrams/maps	X	
11	Surveys	X	
12	Drilling logs	X	
13	Well Installation logs	X	
14	Well Development forms	X	
15	Geophysics reports	X	
16	Photographs	X	
17	Subcontractor reports	X	
18	Relevant correspondence	X	
19	Change orders/deviations	X	
20	Field corrective action reports	X	
Analytical Data Package			
21	Cover sheet (laboratory identifying information)	X	
22	Case narrative	X	X
23	Internal laboratory chain-of-custody	X	
24	Sample receipt records	X	X
25	Sample chronology (i.e., dates and times of receipt, preparation, and analysis)	X	X
26	Communication records	X	X
27	Limit of detection/limit of quantification establishment and verification	X	X[1]
28	Standards traceability	X	
29	Instrument calibration records	X	X[1]
30	Definition of laboratory qualifiers	X	X
31	Sample results	X	X
32	QC sample results	X	X[1]
33	Corrective action reports	X	X
34	Raw data	X	X[2]
35	Electronic data deliverable	X	X

Note:
1. Applies to Stage 2B validation and higher.
2. Applies to Stage 4 validation only

QAPP Worksheet #35: Data Verification Procedures

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbooks / sample collection forms / daily reports / photographs	QAPP, field SOPs, subcontractor contracts	Verify that records are present and complete for each day of field activities. Verify that planned samples including field QC samples were collected and that sample collection dates, times, and locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that required field monitoring was performed and results are documented. Verify that data electronically captured, including photographs, have been downloaded to a centralized location and a backup copy exists. Verify that photographs are named using a consistent system and contain sufficient subject information to be useful to a future third- party observer (e.g., sampling location, direction, date).	AECOM FTL or designee (daily) AECOM Task Order Manager AECOM Senior Geologist, Chemist, or Technical Lead (depending on event focus) AECOM Database Manager
Equipment calibration records / Instrument testing and inspection records	QAPP, field SOPs, subcontractor contracts	Verify that records are present and complete for each type of equipment and the duration of field activities. Verify that calibration and testing were performed in accordance with the QAPP.	AECOM FTL or designee (daily) AECOM Senior Geologist, Chemist, or Technical Lead (depending on event focus)
Chain-of-custody forms	QAPP, field SOPs, laboratory SOPs	Verify the completeness of chain-of-custody records and entries for consistency with field records. Verify that custody forms are complete and correct, including sample IDs, preservation, analyses, dates, times, and signatures Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that required signatures and dates are present. Check for transcription errors.	AECOM FTL or designee (daily) Laboratory Sample Management (upon sample receipt) AECOM Project Chemist AECOM Data Validator

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Surveys	QAPP, field SOPs, subcontractor contracts	Verify the coordinate data for surveyed sample locations is present and complete. Check to make sure coordinate data has been presented in the correct coordinate system and referenced to the appropriate datum. Plot the coordinate data and compare against fieldnotes, drilling logs, and other field documentation to confirm the coordinates accurately show the sampling location.	AECOM FTL or designee AECOM GIS Specialist
Drilling logs/Well Installation logs / Well Development forms	QAPP, field SOPs, subcontractor contracts	Check that records (digital and hardcopy) are present for each day of field activities. Examine information recorded in the log sheets, forms, and notes to ensure documentation is accurate and complete. Verify geologic and lithologic information has been recorded following the USCS protocols for logging soils. Verify that changes/exceptions/deviations were reported in accordance with requirements.	AECOM FTL (or designee)
Geophysics Reports	QAPP, field SOPs, subcontractor contracts	Review the procedures intended to be implemented during each downhole test. Confirm the data generated by the subcontractor matches the field notes.	AECOM Technical Lead
Laboratory Deliverable	QAPP	Verify that the laboratory deliverable contains records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and missing/broken sample containers were noted and reported according to plan. Compare the data package with the CoCs to verify that results were provided for collected samples. Review the narrative to ensure QC exceptions are described. Check for evidence that required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Laboratory Project Manager (before release) AECOM Data Validator (during validation or review)
Subcontractor Reports	QAPP, field SOPs, subcontractor contracts	Check to make sure that transmissivity profile and reverse head profile plots generated by FLUTe match the field notes. Check for proper units and ensure that the dates and depths of FLUTe measurements match the well that was tested.	AECOM Technical Lead

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Corrective Action Reports	QAPP	Verify that planned audits were conducted. Examine audit reports. For deficiencies noted, verify that corrective action was implemented according to plan.	AECOM Task Order Quality Representative

QAPP Worksheet #36: Data Validation Procedures

Data validation will be conducted on all fixed laboratory data with the exception of VOC analysis of FLUTe FACT and that generated for characterization of IDW. VOC analysis of FACT is considered screening-level data. Details for validation of the remaining data are presented in the tables below. The validation code and tier are defined at the end of this WS.

For the data loaded to FUDSChem using SEDD Stage 2a deliverables, validation will be performed using the FUDSChem ADR module. Supplemental manual validation will be conducted as needed to meet the required validation stage. Validation for the remaining data will be conducted manually.

Data for all analytes will be validated to a combination of Stage 2bVEM and Stage 2aVEM. Supporting analyses used for matrix characterization will be validated to Stage 2aVEM unless otherwise indicated. For the analyses for which a combination of validation stages is planned, the first SDG of each matrix and analysis will undergo the greater level of validation, Stage 2bVEM. Subsequent SDGs will be validated to a lesser level, Stage 2aVEM. If data quality issues identified during the initial validation indicate that a greater level of review is warranted for subsequent data, the validation level may be increased to Stage 2b for those data.

FACT data is considered screening level. The subcontractor to FLUTe is not able to generate SEDD 2a deliverables.

Validation will be performed in accordance with the requirements of this UFP-QAPP, the DoD General DV Guidelines (EDQW, 2018), the applicable DoD QSM version (or laboratory SOP), the *EPA New England Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Guidance/Procedures* (EPA-NE, 2018), and the EPA National Functional Guidelines, [*USEPA Contract Laboratory Program National Functional Guidelines for Organic Superfund Methods Data Review (SOM02.4) (January 2017); USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (ISM02.4) (January 2017)*]. Guidelines applicable to each method will be considered in the order presented with this UFP-QAPP taking precedence over the documents that follow.

EPA validation guidelines will be adapted as necessary for the non-EPA Contract Laboratory Program (CLP) methods. In addition to the DoD General Guidelines, method-specific DoD validation modules that are released as final prior to the start of the sampling program will also be applied.

Per client request, validators will add review of the LOD and LOQ verification to the validation procedures. Validators will review the case narratives of the analytical data reports to determine whether LOD and LOQ verification for the applicable method and matrix was successful for the quarter

in which the samples were analyzed. If verification was not successful, non-detected results at the LOD will be qualified as estimated with unknown bias. Professional judgement may be applied to qualify additional results.

Matrix: Soil

Analytical Group/Method:	VOCs, SVOCs, SVOCs SIM	Metals, SPLP Metals	pH, TOC[2]	Grain Size
Data deliverable requirements:	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report uploaded to FUDSChem
Analytical specifications[1]:	DoD QSM v. 5.1	DoD QSM v. 5.1; laboratory SOP for SPLP leaching (Refer to WS 23)	Laboratory SOPs (Refer to WS 21)	Laboratory SOP (Refer to WS 21)
Measurement performance criteria:	WS12	WS12	WS12	WS12
Percent of data packages to be validated:	100%	100%	100%	100%
Percent of raw data reviewed:	0%	0%	0%	0%
Percent of results to be recalculated:	0%	0%	0%	0%
Validation procedure:	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD DV Module 1, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines, DoD QSM (laboratory SOP for leaching procedure), EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines and the laboratory SOP. UFP-QAPP will take precedence over the documents that follow.	Manual verification and validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines and the laboratory SOP. UFP-QAPP will take precedence over the documents that follow.
*Validation code (*see attached table):*	S2aVEM, S2bVEM	S2aVEM, S2bVEM	S2aVEM	S1VM
Electronic validation program/version:	ADR Module, FUDSChem	ADR Module, FUDSChem	ADR Module, FUDSChem	Not Applicable

Notes:

1. Laboratory analytical SOPs for each matrix and analysis are listed in WS 19&30. Laboratory SOPs are listed on WS 23.
2. Validation tier and procedures are dependent on FUDSChem capabilities. Refer to the text at the beginning of this WS.

QSM – Quality Systems Manual

Matrix: Groundwater

Analytical Group/Method:	VOCs, VOCs SIM, SVOCs, SVOC SIM	TAL Metals/Hg (Total and Field Filtered)	TOC, Sulfide, Alkalinity[2]
Data deliverable requirements:	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem
Analytical specifications[1]:	DoD QSM v. 5.1	DoD QSM v. 5.1	Laboratory SOPs (Refer to WS 21)
Measurement performance criteria:	WS12	WS12	WS12
Percent of data packages to be validated:	100%	100%	100%
Percent of raw data reviewed:	0%	0%	0%
Percent of results to be recalculated:	0%	0%	0%
Validation procedure:	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD DV Module 1, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines, and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	Manual verification and validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines and the laboratory SOP. UFP-QAPP will take precedence over the documents that follow.
*Validation code (*see attached table):*	S2aVEM, S2bVEM	S2aVEM, S2bVEM	S2aVEM
Electronic validation program/version:	ADR Module, FUDSChem	ADR Module, FUDSChem	ADR Module, FUDSChem

Notes:

1. Laboratory analytical SOPs for each matrix and analysis are listed in WS19&30. Laboratory SOPs are listed on WS 23.
2. Validation tier and procedures are dependent on FUDSChem capabilities. Refer to the text at the beginning of this WS.

QSM – Quality Systems Manual

Matrix: Groundwater (Continued)

Analytical Group/Method:	**Dissolved gasses (M, E, E), Nitrate as N, Nitrite as N, Sulfate, Chloride**
Data deliverable requirements:	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem
Analytical specifications[1]:	DoD QSM v. 5.1
Measurement performance criteria:	WS12
Percent of data packages to be validated:	100%
Percent of raw data reviewed:	0%
Percent of results to be recalculated:	0%
Validation procedure:	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines, and DoD QSM. UFP-QAPP will take precedence over the documents that follow.
*Validation code (*see attached table):*	S2aVEM
Electronic validation program/version:	ADR Module, FUDSChem

Note:
1. Laboratory analytical SOPs for each matrix and analysis are listed in WS19&30. Laboratory SOPs are listed on WS 23.

QSM – Quality Systems Manual

Matrix: Surface Water and Pore Water

Analytical Group/Method:	VOCs, VOCs SIM, SVOCs, SVOC SIM	TAL Metals/Hg (Total and Field Filtered)	Hardness
Data deliverable requirements:	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem
Analytical specifications[1]:	DoD QSM v. 5.1	DoD QSM v. 5.1	DoD QSM v. 5.1 and Laboratory SOPs (Refer to WS 21)
Measurement performance criteria:	WS12	WS12	WS12 (refer to TAL metals, Total)
Percent of data packages to be validated:	100%	100%	100%
Percent of raw data reviewed:	0%	0%	0%
Percent of results to be recalculated:	0%	0%	0%
Validation procedure:	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD DV Module 1, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines, and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	Harness will be calculated from the total metals analytical results. Validation qualifiers applied to the calcium and magnesium results will be carried over to the hardness data as appropriate.
*Validation code (*see attached table):*	S2aVEM, S2bVEM	S2aVEM, S2bVEM	S2aVEM, S2bVEM
Electronic validation program/version:	ADR Module, FUDSChem	ADR Module, FUDSChem	ADR Module, FUDSChem

Note:
1. Laboratory analytical SOPs for each matrix and analysis are listed in WS19&30. Laboratory SOPs are listed on WS 23.

QSM – Quality Systems Manual

Matrix: Sediment

Analytical Group/Method:	VOCs, SVOCs, SVOCs SIM	Metals	AVS/SEM[2]	pH, TOC[2]
Data deliverable requirements:	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem	Pdf of complete data report and SEDD Stage 2a uploaded to FUDSChem
Analytical specifications[1]:	DoD QSM v. 5.1	DoD QSM v. 5.1	DoD QSM v. 5.1 and Laboratory SOPs (Refer to WS 21)	Laboratory SOPs (Refer to WS 21)
Measurement performance criteria:	WS12	WS12	WS12	WS12
Percent of data packages to be validated:	100%	100%	100%	100%
Percent of raw data reviewed:	0%	0%	0%	0%
Percent of results to be recalculated:	0%	0%	0%	0%
Validation procedure:	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD DV Module 1, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines, DoD QSM, EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines, DoD QSM and laboratory SOP, EPA Region 1 guidelines and NFG. UFP-QAPP will take precedence over the documents that follow.	FUDSChem ADR with manual verification and supplemental validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines and the laboratory SOP. UFP-QAPP will take precedence over the documents that follow.
*Validation code (*see attached table):*	S2aVEM, S2bVEM	S2aVEM, S2bVEM	S2aVEM, S2bVEM	S2aVEM
Electronic validation program/version:	ADR Module, FUDSChem	ADR Module, FUDSChem	ADR Module, FUDSChem	ADR Module, FUDSChem

Note:
1. Laboratory analytical SOPs for each matrix and analysis are listed in WS 19&30. Laboratory SOPs are listed on WS 23.
2. Validation tier and procedures are dependent on FUDSChem capabilities. Refer to the text at the beginning of this WS.

QSM – Quality Systems Manual

Matrix: Sediment (Continued)

Analytical Group/Method:	Grain Size
Data deliverable requirements:	Pdf of complete data report uploaded to FUDSChem
Analytical specifications[1]:	Laboratory SOP (Refer to WS 21)
Measurement performance criteria:	WS12
Percent of data packages to be validated:	100%
Percent of raw data reviewed:	0%
Percent of results to be recalculated:	0%
Validation procedure:	Manual verification and validation as required by Stage; Validation will be performed in accordance with the requirements of this UFP-QAPP, DoD General DV Guidelines and the laboratory SOP. UFP-QAPP will take precedence over the documents that follow.
*Validation code (*see attached table):*	S1VM
Electronic validation program/version:	Not Applicable

Notes:
1. Laboratory analytical SOPs for each matrix and analysis are listed in WS 19&30. Laboratory SOPs are listed on WS 23.
2. Validation tier and procedures are dependent on FUDSChem capabilities. Refer to the text at the beginning of this WS.

Validation Tiers and Selection of Data for Each Tier

Validation Tiers are as defined in the *United States Department of Defense General Data Validation Guidelines* (EDQW, 2018).

Validation Code and Label Identifier Table

Validation Code*	Validation Label
S1VE	Stage 1 Validation Electronic
S1VM	Stage 1 Validation Manual
S1VEM	Stage 1 Validation Electronic and Manual
S2aVE	Stage 2a Validation Electronic
S2aVM	Stage 2a Validation Manual
S2aVEM	Stage 2a Validation Electronic and Manual
S2bVE	Stage 2b Validation Electronic
S2bVM	Stage 2b Validation Manual
S2bVEM	Stage 2b Validation Electronic and Manual
S3VE	Stage 3 Validation Electronic
S3VM	Stage 3 Validation Manual
S3VEM	Stage 3 Validation Electronic and Manual
S4VE	Stage 4 Validation Electronic
S4VM	Stage 4 Validation Manual
S4VEM	Stage 4 Validation Electronic and Manual
NV	Not Validated

Validation Qualifiers

Data validation qualifiers that will be used for validation of fixed-laboratory data are those presented in the DoD General DV Guidelines (EDQW, 2018), presented below:

Qualifier	Definition
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.

LOD – Limit of Detection

QAPP Worksheet #37: Data Usability Assessment

37.1 Data Review

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used

AECOM will validate the fixed-laboratory data for all definitive analyses conducted. Validation will be conducted in accordance with the protocols described in Worksheets #34-36. The Project Chemist, in conjunction with the project team, will determine whether the analytical data meet the requirements to support the investigation. The results of laboratory measurements will be compared to the DOQs described in Worksheet #11.

At the completion of validation, data qualified by the validators as "X" (affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria) will be reviewed by the Project Chemist. The Project Chemist, with input from the project team, will determine which data should be accepted or rejected (R), and excluded from the data set.

Describe the evaluative procedures used to assess overall measurement error associated with the project

A data assessment will be performed in accordance with USEPA guidance QA/G-9R, *Data Quality Assessment, A Reviewer's Guide* (EPA/240/B-06/002) dated February 2006. In accordance with USEPA guidance, a data assessment is intended to provide documentation to clearly demonstrate that the collected data are of the right type, quality, and quantity to meet the objectives of the project. A comprehensive evaluation of how the data meet precision, accuracy, representativeness, comparability, and completeness (PARCC) objectives will also be performed.

The data usability assessment will reconcile the DQOs of this UFP-QAPP to the results of the data collection and analytical results, data validation evaluation (as applicable), and field QC results.

Data quality indicators, such as precision, accuracy, completeness, representativeness, and comparability measurements, aid in the evaluation process and are discussed in the following subsections.

<u>Precision</u>

The most commonly used estimates of precision are the RPD for cases in which only two measurements are available and the percent relative standard deviation (%RSD) when three or more measurements are available. The latter is especially useful in normalizing environmental measurements to determine acceptability ranges for precision because it effectively corrects for the wide variability in sample analyte concentration indigenous to samples.

Precision is represented as the RPD between measurement of an analyte in duplicate samples or in duplicate spikes. RPD is defined as follows:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where:

C1 = First measurement value
C2 = Second measurement value

The %RSD is calculated by the standard deviation of the analytical results of the replicate determinations relative to the average of those results for a given analyte. This method of precision measurement can be expressed by the formula:

$$\%RSD = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - [\sum_{i=1}^{n} x_i]^2 / n}{n-1}} \times 100 / RF$$

Where:

RF = Response factor

N = Number of measurements

Precision control limits for evaluation of sample results are established by the analysis of control samples. The control samples can be method blanks fortified with surrogates (e.g., for organics), or LCS purchased commercially or prepared at the laboratory. The LCS is typically identified as blank spikes (BS) for organic analyses. For multi-analyte methods, the LCS or BS may contain only a representative number of target analytes rather than the full list.

The RPD for duplicate investigative sample analysis provides a tool for evaluating how well the method performed for the respective matrices.

Accuracy/Bias

Accuracy control limits are established by the analysis of control samples, which are water and/or solid/waste matrices. For organic analyses, the LCS may be a surrogate constituent in the blank or a select number of target analytes in the BS. The LCS is subjected to all sample preparation steps. When available, a solid LCS may be analyzed to demonstrate control of the analysis for soil. The

amount of each analyte recovered in an LCS analysis is recorded and entered into a database to generate statistical control limits. These empirical data are compared with available method reference criteria and available databases to establish control criteria.

The % R for spiked investigative sample analysis (e.g., matrix spike) provides a tool for evaluating how well the method worked for the matrix. These values are used by the USACE to assess a reported result within the context of the project DQOs. For results that are outside control limits provided as requirements in the QAPP, corrective action appropriate to the project will be taken and the deviation will be noted in the case narrative accompanying the sample results. The %R is defined as follows:

$$\% R = \frac{(A_T - A_{O)} \times 100)}{A_F}$$

Where:

AT = Total amount recovered in fortified sample

A0 = Amount recovered in unfortified sample

AF = Amount added to sample

Accuracy for some procedures is evaluated as the degree of agreement between a new set of results and a historical database or a table of acceptable criteria for a given parameter. This degree of agreement is measured as %D from the reference value and is primarily used by the laboratory as a means for documenting acceptability of continuing calibration.

The %D is calculated by expressing, as a percentage, the difference between the original value and new value relative to the original value. This method for precision measurement can be expressed by the formula:

$$\% D = \frac{C1 - C2}{C_1} \times 100$$

Where:

C1 = Concentration of analyte in the initial aliquot of the sample.

C2 = Concentration of analyte in replicate.

Completeness

Site-wide completeness goals account for all aspects of sample handling, from collection through data reporting. The level of completeness can be affected by loss or breakage of samples during transport, as well as external problems that prohibit collection of the sample. The following calculation is used for determining the percent complete:

$$\text{Completeness} = \frac{A}{B} \times 100$$

Where:

A = Number of usable data points.

B = Total number of data points collected.

The formula for sampling completeness is:

$$\text{Sampling Completeness} = \frac{\text{Number of locations sampled}}{\text{Number of planned sample locations}} \times 100$$

An example formula for analytical completeness is:

Trichloroethene (TCE) in Groundwater Analytical Completeness

$$= \frac{\text{Number of Usable Data Points}}{\text{Expected Number of Usable Data Points}} \times 100$$

The ability to meet or exceed completeness objectives is dependent on the nature of samples submitted for analysis.

Determining whether the data set obtained is sufficiently complete to accomplish project objectives must consider the multiple objectives for each data point in the context of the whole data set- both current and historical. A goal based on a simple numerical percentage of results does not capture this complexity. Consideration in the context of determination of risk, extent, nature and fate and transport must all be weighed. Whether the analyte is likely Site-related, whether it is a potential risk-driver, whether the location of the missing data presents a critical spatial data gap, whether there are sufficient points remaining in the exposure area to calculate 95% UCLs all are questions that must be answered. Determination of whether completeness is sufficient must be made by multiple disciplines within the project team.

If the completeness goal is not met because of controllable circumstances, then the samples will be recollected and reanalyzed, as necessary, to meet the completeness objectives. If the completeness goal is not met because of uncontrollable circumstances, such as inaccessible sample points, matrix interferences, etc., then the deficiency will be evaluated by the project team and resulting limitations will be discussed in data usability.

Representativeness

Data representativeness for a project is accomplished by implementing approved sampling procedures and analytical methods that are appropriate for the intended data uses, and which are established within this project-specific QAPP.

Comparability

Comparability of data sets generated for a project will be obtained through the implementation of standard sampling and analysis procedures, by the use of traceable reference materials for laboratory standards, and by expressing the results in comparable concentration units.

Sensitivity

Sensitivity is the ability of the method or acceptable sensitivity instrument to detect the COPC and other target constituents at the level of interest. Quantitative MPC need to be determined for acceptable sensitivity to ensure that the quantitation limits can be routinely achieved for each matrix, analytical parameter, and concentration level. The use of standards and instrument calibration will enable the instrument to identify and differentiate between various constituents/analytes of interest and interferences.

Assessment of Data Usability

In addition, data assessment is considered the final step in the data evaluation process and can be performed only on data of known and documented quality. For a project, all data will be assessed for usability, regardless of the data evaluation/validation process implemented. As mentioned previously, data usability goes beyond validation because it evaluates the achievement of the DQOs based on the comparison of the specific WPs with the obtained results. The results of the data usability assessment, and particularly changes to the DQOs necessitated by the data not meeting usability criteria, will be included in each final data quality assessment report. As noted in Worksheet #15, PALs for some analytes are below the limit of quantitation (LOQ); uncertainties associated with LOQs greater than the associated PALs for Site-related analytes will be discussed qualitatively in the RI and/or risk assessments, as applicable.

Primarily, the assessment of the usability will follow procedures described in appropriate USEPA guidance documents, particularly *Guidance for Data Usability in Risk Assessment* (Publication No. 9285.7-05FS, September 1992), and will be conducted according to the process outlined below.

Sampling and Analysis Activities Evaluation

The first step of the data usability evaluation will include a review of the sampling and analysis activities in comparison to Site-Wide DQIs and study-specific WPs. Specific limitations to the data, i.e., results that are qualified as estimated (J/UJ), or rejected (R), will be determined and documented in the database. The data acquisition and evaluation process consists of a series of procedures that were designed to maximize final data quality.

Assessment of DQIs

The second part of data usability pertains to the assessment of the program-specific DQIs. Each investigator will compare the performance achieved for each data quality criterion against the expected and planned performance. In general, this comparison will follow from the DQIs used to define each DQO. The comparison is the most critical component of the assessment process. Deviations from planned performance will be documented and evaluated to determine whether corrective action is advisable. Potential corrective actions will range from resampling and/or reanalysis of data, to qualification or exclusion of the data for use in the data interpretation. In the event that corrective action is not possible, the limitations, if any, of the data with regard to achieving the DQOs will be noted.

In conjunction with the DQI achievement review, the investigators will need to make decisions for the use of qualified values, which are a consequence of the formalized evaluation/validation process. Data qualifiers will be applied to individual data results. Data usability decisions will be made based on the assessment of the usability of each of these results for the intended purpose. Evaluation will describe the uncertainty (e.g., bias, imprecision) of the qualified results. Cumulative QC exceedances from the DQIs may require technical judgment to determine the overall effect on the usability of the data. Decisions about usability of qualified data for use in risk assessment will be based on the USEPA document mentioned, which allows for the use of estimated values. Finally, data users may choose to determine final data usability qualifiers as a result of the overall examination and decision process.

Achievement of DQOs

The third step in the data usability process concerns achievement of the DQOs. After the data set has been assessed to be of known quality, data limitations have been documented, and overall result applicability/usability for its intended purpose has been determined, the final data assessment can be initiated by considering the answers to the following questions:

- Does the data determine the likely source area and whether that source is discrete or diffuse?
- Does the data collected adequately characterize the nature and extent of COPCs?
- Is the data statistically adequate to assess COPCs on a per constituent and per media basis?
- Does the data collected allow assessment of hydrogeological factors, which may influence migration/distribution of COPCs?
- Has sufficient data been collected to determine potential risk to human health receptors associated with exposure to groundwater under current and reasonably anticipated future land use scenarios?
- Have sufficient data been collected to determine potential risk to ecological receptors identified in a *Wetland and Waters of the US Delineation Report* (Woodard & Curran, 2019) associated with potentially impacted downgradient surface water?
- Is the sample set sufficient to develop Site-specific remedial action?

The principal investigators, in conjunction with the project team, will formulate solutions if data gaps are found as a result of problems, biases, or trends in the analytical data or if conditions exist that were not anticipated in the development of the DQOs. It is particularly important that each data usability evaluation specifically address limitations on the use of the data that may result from a failure to achieve the stipulated DQO.

Identify the personnel responsible for performing the usability assessment

Data validation will be coordinated by the AECOM Project Chemist and will be conducted by the AECOM data validation staff. Data usability will be assessed by the AECOM TOM with the assistance of the entire project team.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies

The documentation generated during data validation will include a memorandum that describes the information reviewed, the results of this review, and a recommendation on data usability and limitations of specific data points. The memorandum provides information on the samples included in the review and the date they were collected, the condition of samples when received at the laboratory and discrepancies noted during the receiving process, verification of sample preparation and analysis within the method specified holding time, review of associated QC analyses including

blanks, LCSs, MSs, and field and/or laboratory duplicates. As a result of this review standard qualifiers are entered into the FUDSChem database so that data users can readily identify limitations associated with a specific data point.

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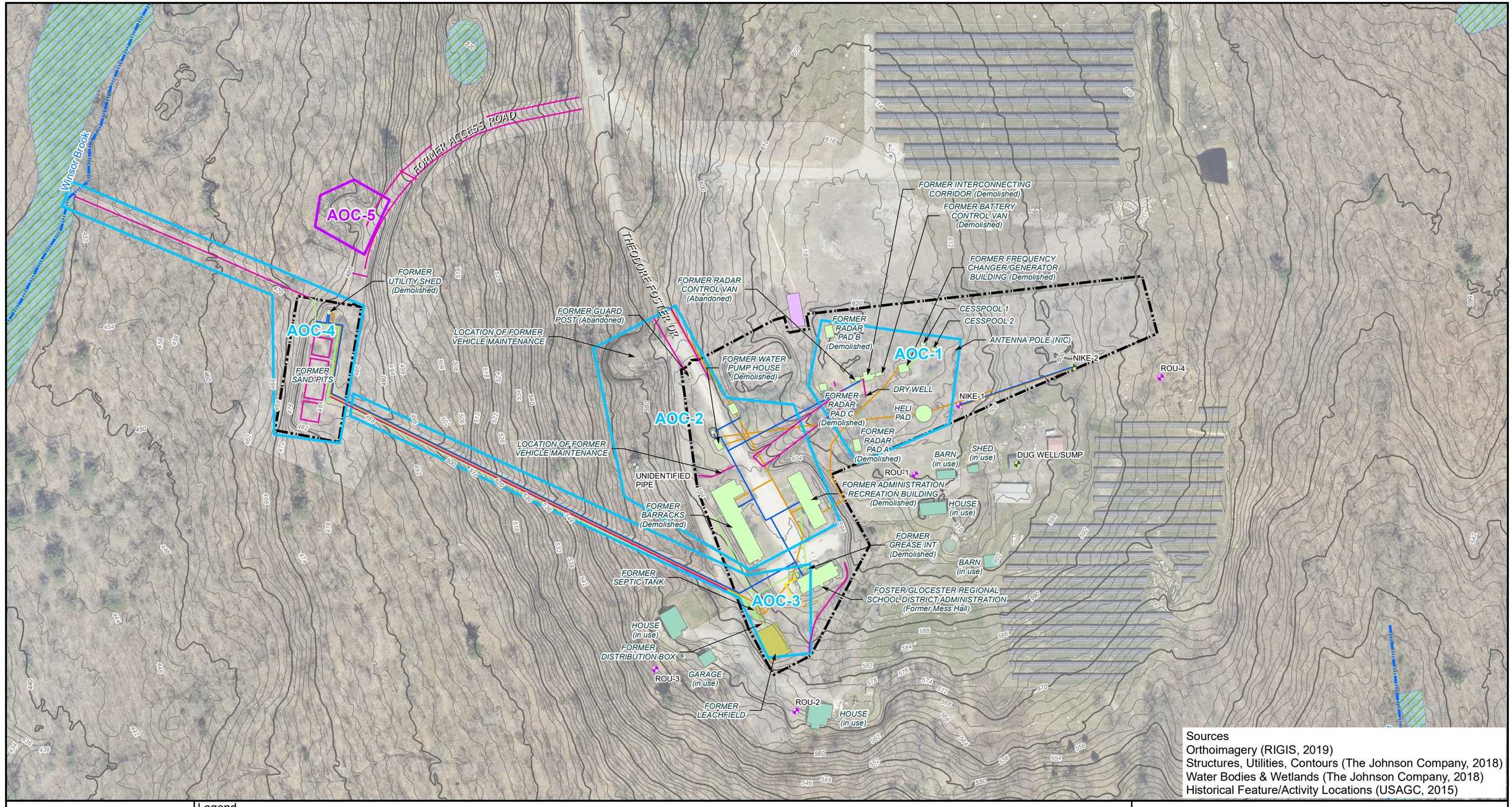
Appendix A

Figures



Sources
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Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	07/23/2020
Approved:	GH	07/23/2020
Project #:	60608703	

Legend

- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Existing Sewer Structures
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetlands
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Index Contour
- Intermediate Contour
- Existing Sewer Line
- Electrical Line
- Liquefied Petroleum Gas Line
- Sewer Line
- Water Line
- Drainage

N

0 100 200

Scale in Feet

FIGURE 10-1
SITE PLAN
DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_10_1_Site_Plan.mxd

PR79-SB-101/PR79-MW-001
PZ-011
PZ-001
PR 79 C RM 2
FORMER RADAR PAD B (Demolished)
PR 79 C 2 RM 4
FORMER 6,000 GALLON DIESEL FUEL UST (Removed)
CESSPOOL 1
CESSPOOL 2
APPROXIMATE LOCATION OF DUMPING
PR79-SB-105
FORMER BATTERY CONTROL VAN (Demolished)
ANTENNA POLE (NIC)
PZ-009
PZ-010
GPR 5
FORMER INTERCONNECTING CORRIDOR (Demolished)
SB-008
PZ-002
PR79-SB-104/PR79-MW-003
PZ-019
FORMER RADAR CONTROL VAN (Abandoned)
PR79-SB-120
SB-020
PR 79 C
FORMER FREQUENCY CHANGER/GENERATOR BUILDING (Demolished)
FORMER RADAR PAD C (Demolished)
FLOOR DRAIN
PR79-SB-103/PR79-MW-002
PR 79 C RM 1
PR 79 C 2 RM 5
PR79-BR-002
PZ-004
PR 79 C 2
PZ-003
AOC-1
PZ-007
PR79-SB-102
GPR 4
DRY WELL
NIKE-1
PZ-022
PR79-SB-107/PR79-BR-001
AS-BUILT 16-06-109 (JUNE 1957)
HELI PAD
PZ-014
GPR 3
PR 79 C RM 3
AOC-2
PZ-006
FORMER RADAR PAD A (Demolished)
PZ-005
PR79-SB-106/PR79-MW-004
SHED (in use)
GPR 2

Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	07/23/2020
Approved:	GH	07/23/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Historic Soil Boring/Piezometer (AMEC, 2013)
- Historic Soil Boring (AMEC, 2013)
- Drinking Water - Individual
- Drinking Water - Not in Use
- Geodetic Control Station (National Geodetic Survey)
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Index Contour
- Intermediate Contour
- Electrical Line
- Sewer Line
- Water Line
- Drainage
- Seismic Lines (Hager Geosciences, Inc (HGI), 2017)

N

0 20 40

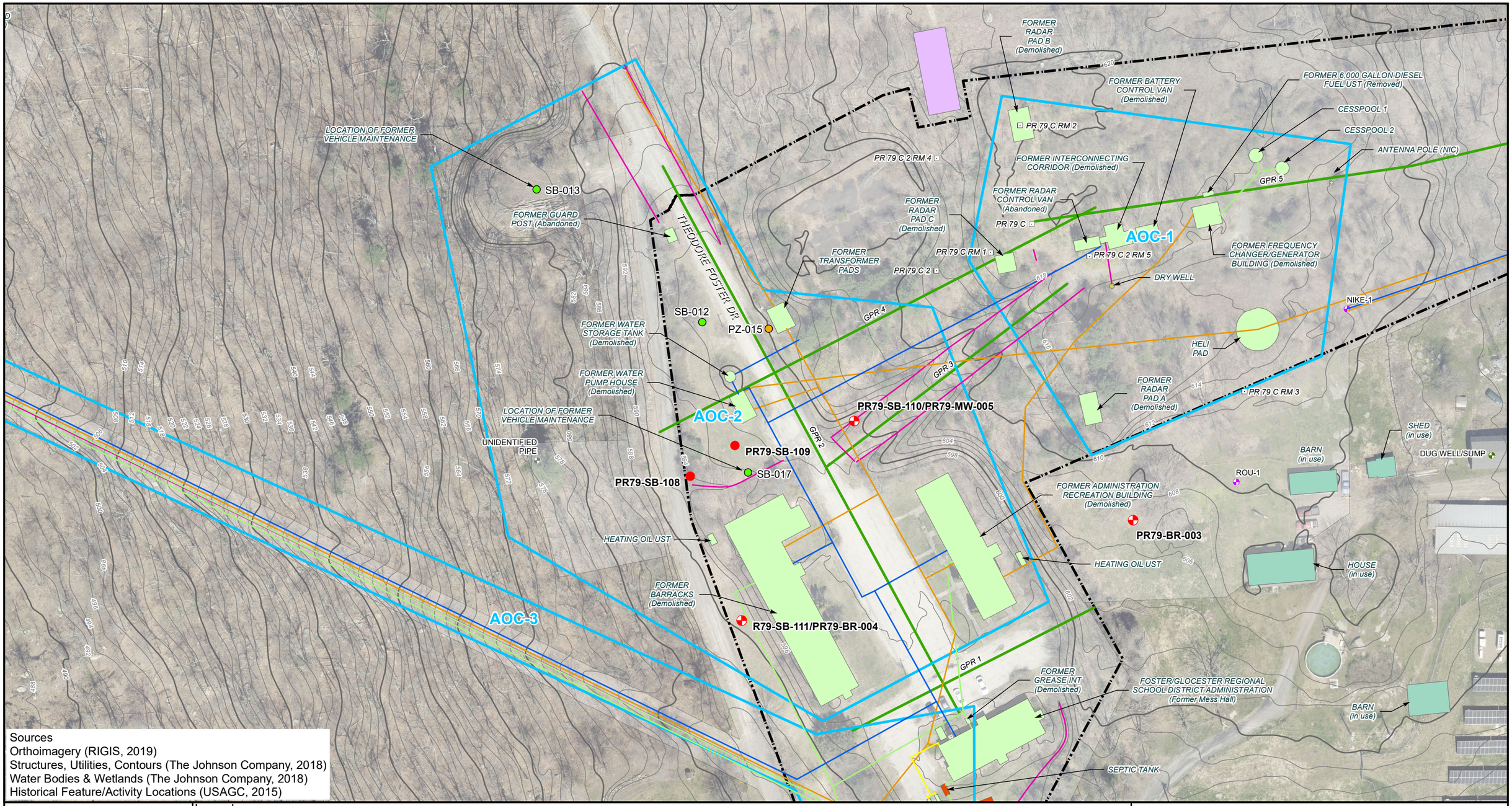
Scale in Feet

FIGURE 17-1A
AOC-1 SAMPLING PLAN

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_1A_AOC_1_Sampling_Plan.mxd



Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	07/23/2020
Approved:	GH	07/23/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Historic Soil Boring/Piezometer (AMEC, 2013)
- Historic Soil Boring (AMEC, 2013)
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Geodetic Control Station (National Geodetic Survey)
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Index Contour
- Intermediate Contour
- Utility
- Electrical Line
- Liquefied Petroleum Gas Line
- Sewer Line
- Water Line
- Drainage
- Seismic Lines (Hager Geosciences, Inc (HGI), 2017)
- Cemetery

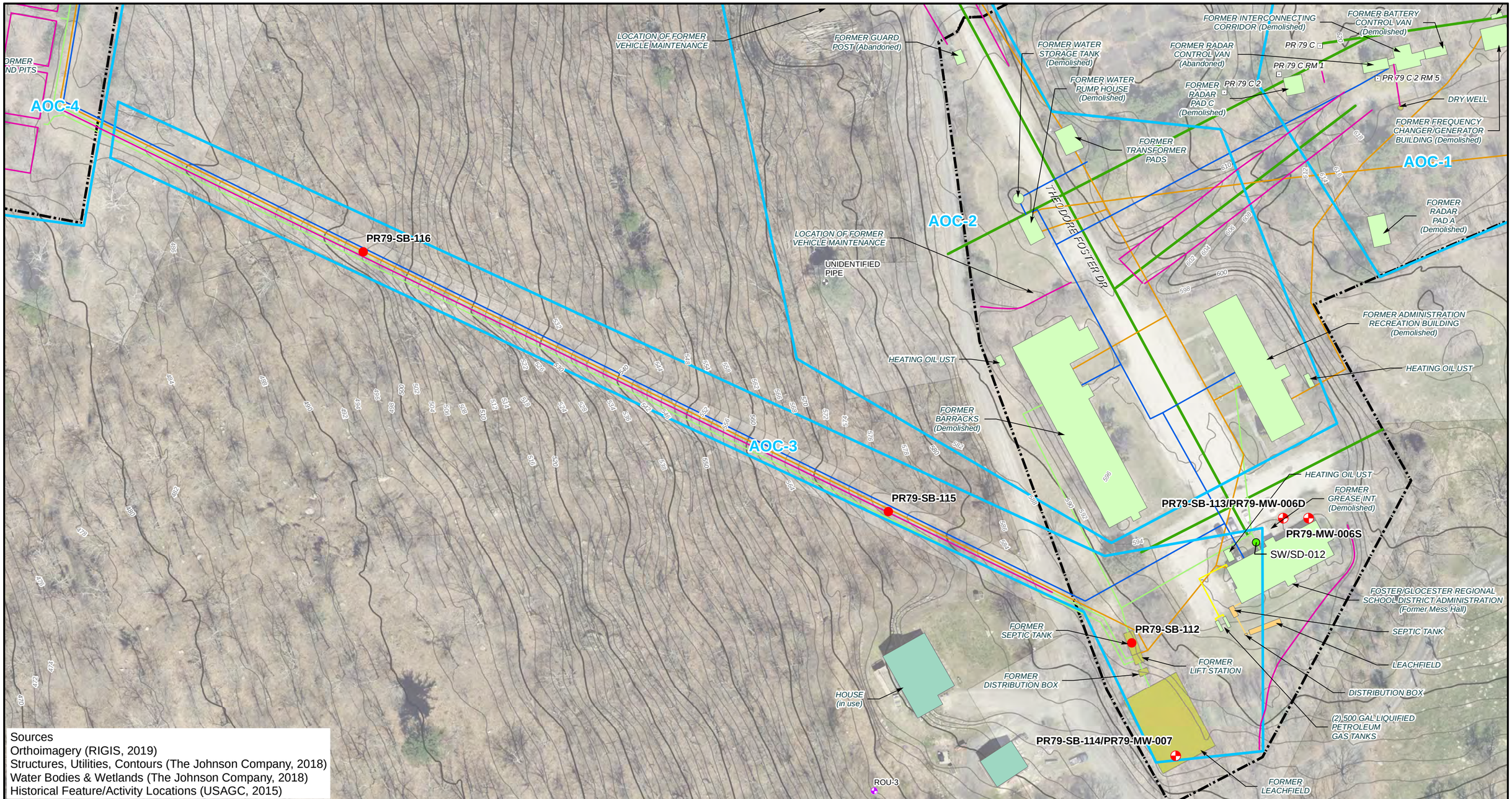
N

0 40 80
Scale in Feet

FIGURE 17-1B
AOC-2 SAMPLING PLAN
DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_1B_AOC_2_Sampling_Plan.mxd



Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	07/23/2020
Approved:	GH	07/23/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Historic Soil Boring (AMEC, 2013)
- Drinking Water - Individual
- Unidentified Pipe
- Geodetic Control Station (National Geodetic Survey)
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Former Sewer and Stormwater Structures
- Existing Sewer Structures
- Existing Sewer Line
- Index Contour
- Intermediate Contour
- Electrical Line
- Liquefied Petroleum Gas Line
- Sewer Line
- Water Line
- Drainage
- Seismic Lines (Hager Geosciences, Inc (HGI), 2017)

N

0 40 80

Scale in Feet

FIGURE 17-1C
AOC-3 SAMPLING PLAN
DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_1C_AOC_3_Sampling_Plan.mxd

AOC-5

FORMER ACCESS ROAD

CHLORINE DETENTION CHAMBER/CONCRETE WEIR

PR79-SB-119

FORMER UTILITY SHED (Demolished)

AOC-4

PR79-SB-118/PR79-MW-008

SB-023

FORMER SAND PITS

PR79-SB-117

SB-026

AOC-3

Winsor Brook

Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	07/23/2020
Approved:	GH	07/23/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Historic Soil Boring (AMEC, 2013)
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetlands
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Index Contour
- Intermediate Contour
- Electrical Line
- Sewer Line
- Water Line
- Drainage
- Flow Direction

N

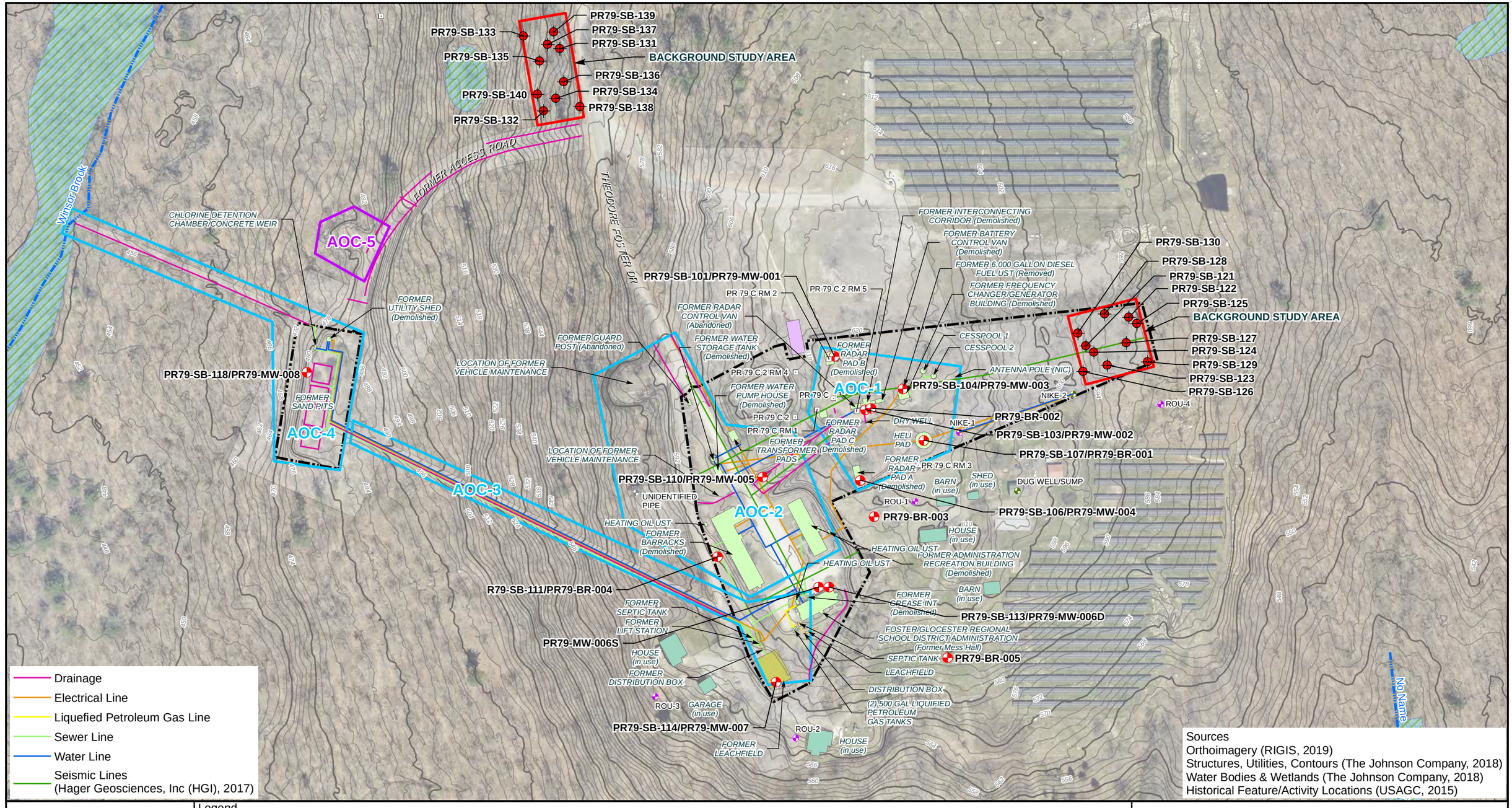
0 40 80

Scale in Feet

FIGURE 17-1D
AOC-4 SAMPLING PLAN
DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_1D_AOC_4_Sampling_Plan.mxd



AECOM

Drawn:	JB	07/23/2020
Approved:	GH	07/23/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Background Sample Location
- Proposed Background Study Area
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Geodetic Control Station (National Geodetic Survey)
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetlands
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Former Sewer and Stormwater Structures
- Existing Sewer Structures
- Existing Sewer Line
- Index Contour
- Intermediate Contour

0 100 200
Scale in Feet

N

FIGURE 17-2
PROPOSED WELL LOCATIONS
DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_2_Proposed_Well_Locations.mxd

PR79-WT-024
PR79-WT-025
PR79-WT-026
PR79-WT-027
PR79-WT-028
PR79-WT-029
PR79-WT-030
PR79-WT-031
PR79-WT-032
PR79-WT-033
PR79-WT-001
PR79-WT-002
PR79-WT-003
PR79-WT-004
PR79-WT-005
PR79-WT-006
PR79-WT-007
PR79-WT-008
PR79-WT-009
PR79-WT-010
PR79-WT-011
PR79-WT-012
PR79-WT-013
PR79-WT-014
PR79-WT-015
PR79-WT-016
PR79-WT-017
PR79-WT-018
PR79-WT-019
PR79-WT-020
PR79-WT-021
PR79-WT-022
PR79-WT-023
PR79-WL-001
PR79-STATION-001
PR79-STATION-002
AOC-1
AOC-2
AOC-3
AOC-4
AOC-5
DW-100
DW-101
DW-82
DW-79
DW-106
DW-107
DW-99
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DW-105
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DW-32
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DW-37
DW-38
DW-39
DW-40
DW-41
DW-42
DW-43
DW-44
DW-45
DW-68/ST 1
DW-69/ST 2
UNUSED
ARTESIAN WELL
BEDROCK WELL
HAND DUG WELL
DRY WELL
NIKE-1
NIKE-2
ROU-1
ROU-2
ROU-3
ROU-4
DUG WELL/SUMP
UNIDENTIFIED PIPE
LEACHFIELD
FORMER LEACHFIELD
FORMER SEPTIC TANK
FORMER 6,000 GALLON DIESEL FUEL UST (Removed)
CESSPOOL 1
CESSPOOL 2
DW-29
DW-52
DW-51
DW-23
DW-24
DW-92
DW-93
DW-83
DW-84
DW-85
DW-86
DW-88
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DW-99
DW-100
DW-101
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DW-107
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DW-41
DW-40
DW-42
DW-44
DW-45
DW-68/ST 1
DW-69/ST 2
UNUSED
ARTESIAN WELL
BEDROCK WELL
HAND DUG WELL
DRY WELL
NIKE-1
NIKE-2
ROU-1
ROU-2
ROU-3
ROU-4
DUG WELL/SUMP
UNIDENTIFIED PIPE
LEACHFIELD
FORMER LEACHFIELD
FORMER SEPTIC TANK
FORMER 6,000 GALLON DIESEL FUEL UST (Removed)
CESSPOOL 1
CESSPOOL 2
DW-29
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DW-51
DW-23
DW-24
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DW-93
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DW-20
DW-21
DW-22
DW-25
Killy Brook
Hannah Brook
No Name
Winsor Brook
Ponagansett River
Fairy Glen
THEODORE FOSTER DR
GENSAHEM RD
APPLE ROCK RD
WINSOR RD
101
Legend

- Proposed Monitoring Well
- Proposed Surface Water/Porewater/Sediment
- Gauging Station
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Parcel
- Access Needed to Parcel
- Access to Parcel
- Stream
- Perimeter Fence
- Bedrock Lineament
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetland (Woodard & Curran, 2019)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Utility
- Sewer Line
- Drainage

DRAFT

AECOM

Drawn:	JB	05/10/2020
Approved:	GH	05/10/2020
Project #:	60608703	

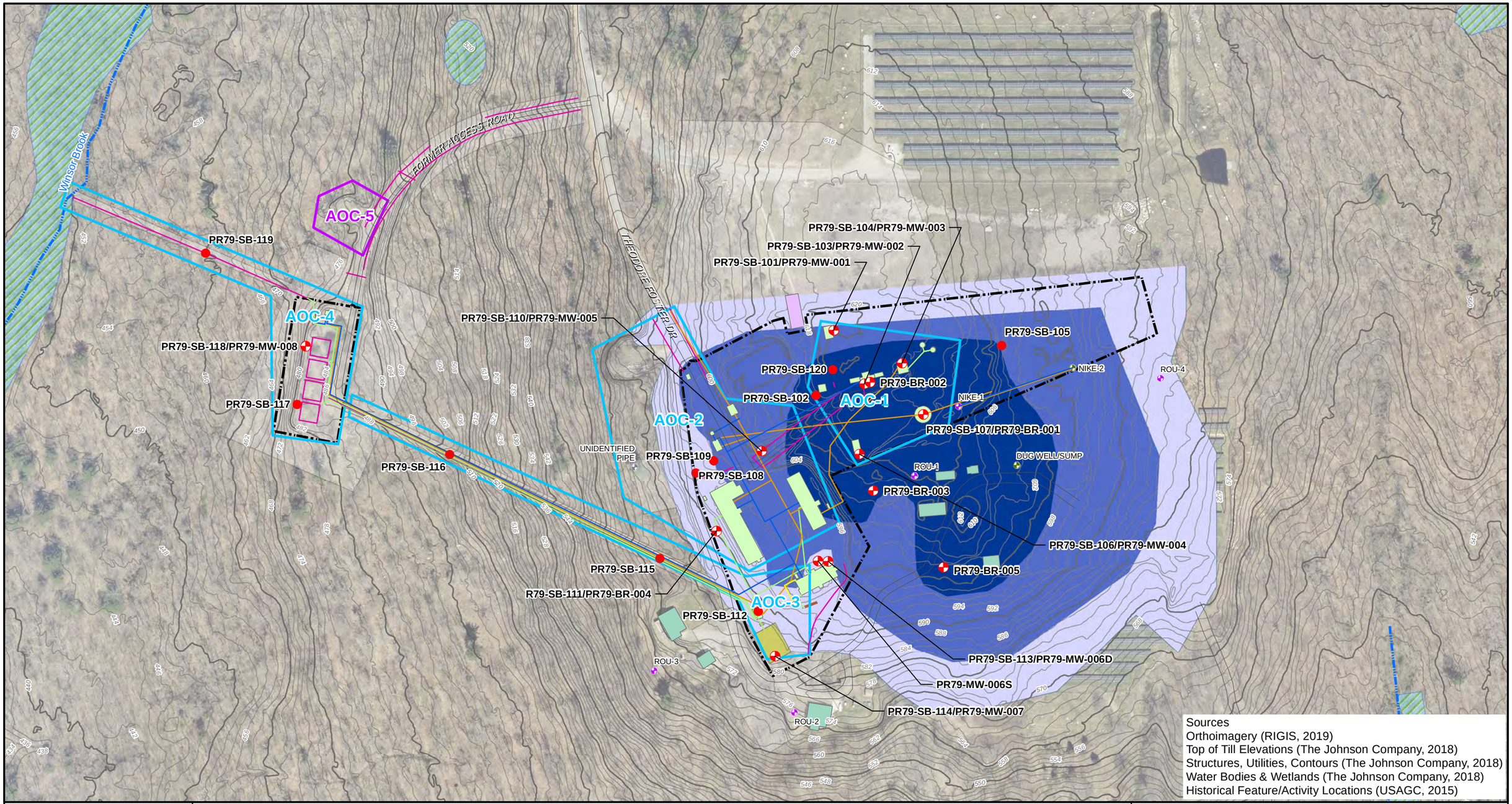
Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Surface Water Bodies & Wetlands (Woodard & Curran, 2019)
Historical Feature/Activity Locations (USAGC, 2015)
Bedrock Lineaments (The Johnson Company, 2018)

0 375 750
Scale in Feet

N

FIGURE 17-3
SITEWIDE SURFACE WATER, PORE WATER, AND SEDIMENT SAMPLING LOCATIONS, ACCESS NEEDED
FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_3_Sitewide_Surface_Water_Porewater_Sediment_Sampling_Loc.mxd



Sources
Orthoimagery (RIGIS, 2019)
Top of Till Elevations (The Johnson Company, 2018)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	05/10/2020
Approved:	GH	05/10/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetlands
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Index Contour
- Intermediate Countour
- Electrical Line
- Liquefied Petroleum Gas Line
- Sewer Line
- Water Line
- Drainage
- Utility
- Cemetery

Top of Silt Till Elevation (ft NAVD88)

- 590 - 600 ft
- 580 - 590 ft
- 570 - 580 ft

N

0 100 200

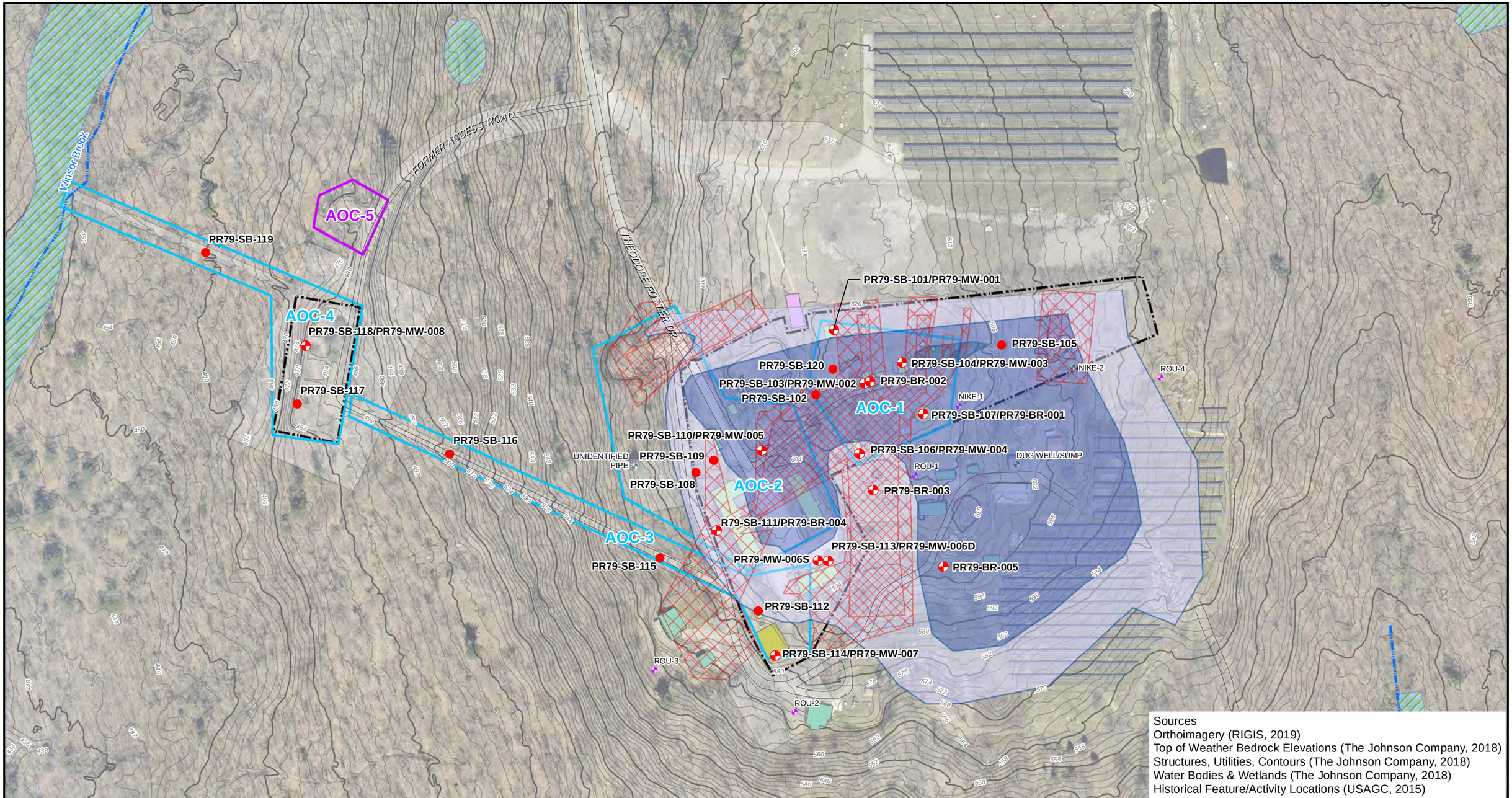
Scale in Feet

FIGURE 17-4
TOP OF TILL ELEVATIONS

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_4_Top_of_Till_Elevations.mxd



Sources
Orthoimagery (RIGIS, 2019)
Top of Weather Bedrock Elevations (The Johnson Company, 2018)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	05/10/2020
Approved:	GH	05/10/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Wetlands
- Cemetery
- Index Contour
- Intermediate Countour
- Utility

Weathered Rock Elevation (ft NAVD88)
- 590 - 600 ft
- 580 - 590 ft
- 570 - 580 ft

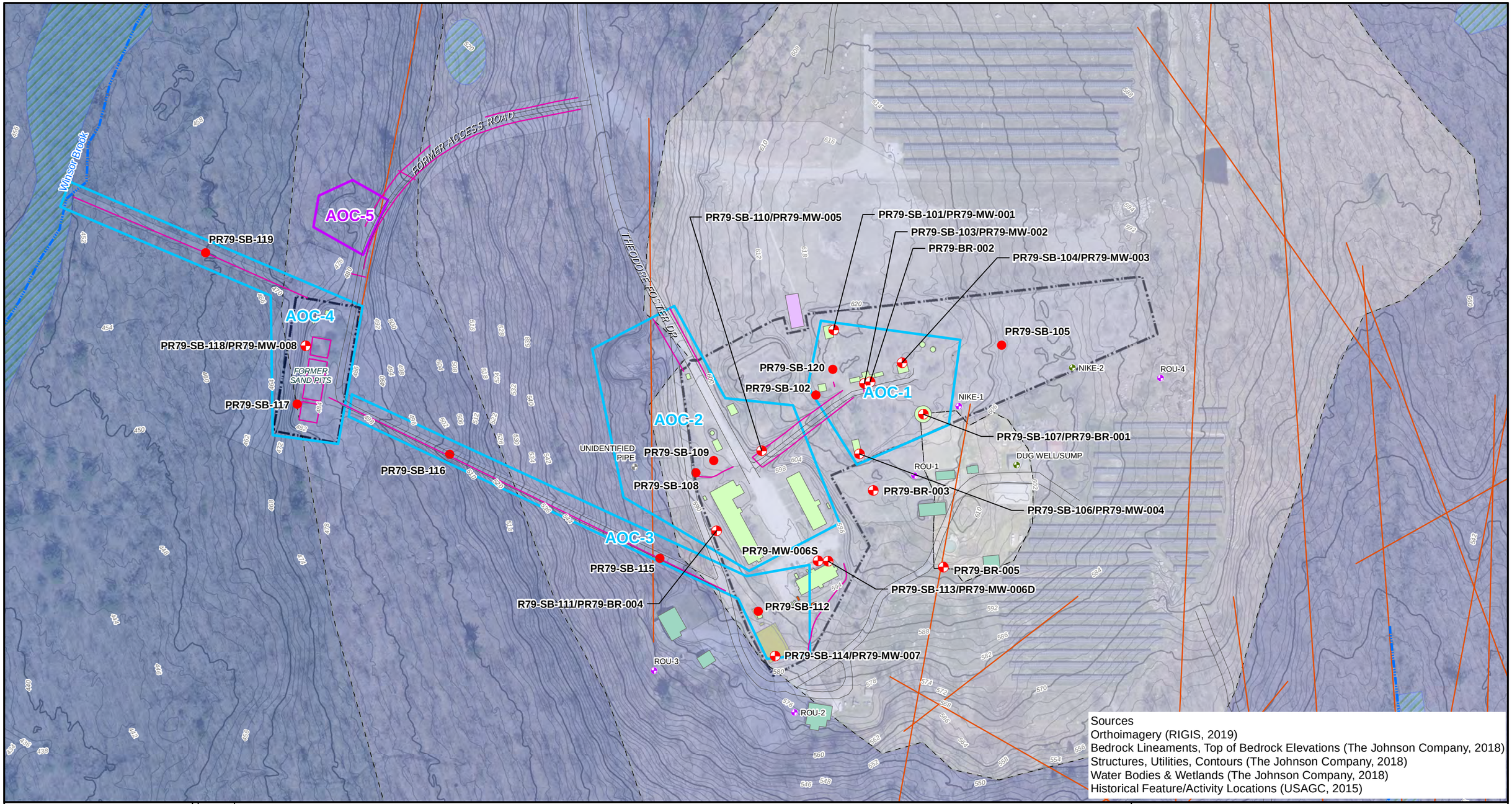
Weathered Rock Thickness from Seismic Data
- 5 ft
- 10 ft

0 100 200
Scale in Feet

N

FIGURE 17-5
TOP OF WEATHERED BEDROCK ELEVATIONS
DRAFT
FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_5_Top_of_Weathered_Bedrock_Elevations.mxd



Sources
Orthoimagery (RIGIS, 2019)
Bedrock Lineaments, Top of Bedrock Elevations (The Johnson Company, 2018)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	05/10/2020
Approved:	GH	05/10/2020
Project #:	60608703	

Legend

- Proposed Monitoring Well
- Proposed Soil Boring
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetlands
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Index Contour
- Intermediate Countour
- Utility
- Drainage
- Bedrock Lineament

Top of Rock Elevation (ft NAVD88)

- 600 - 650 ft
- 550 - 600 ft
- 500 - 550 ft
- 450 - 500 ft
- 400 - 450 ft

N

0 100 200

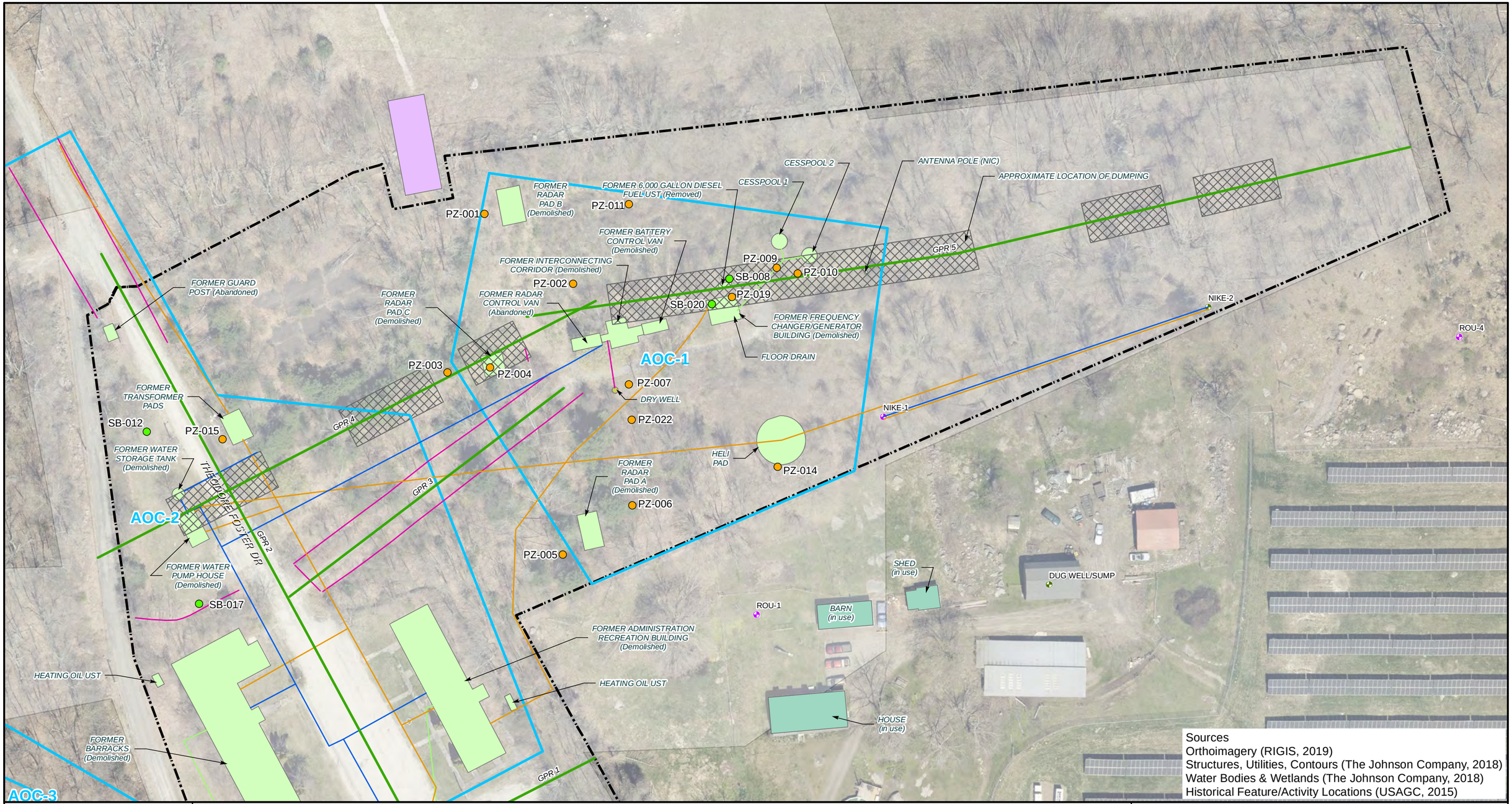
Scale in Feet

FIGURE 17-6
TOP OF BEDROCK ELEVATIONS

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_6_Top_of_Bedrock_Elevations.mxd



Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	05/10/2020
Approved:	GH	05/10/2020
Project #:	60608703	

Legend

- Historic Soil Boring/Piezometer (AMEC, 2013)
- Historic Soil Boring (AMEC, 2013)
- Drinking Water - Individual
- Drinking Water - Not in Use
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Electrical Line
- Sewer Line
- Water Line
- Drainage
- Seismic Lines (Hager Geosciences, Inc (HGI), 2017)
- Fractured Bedrock

N

0 35 70

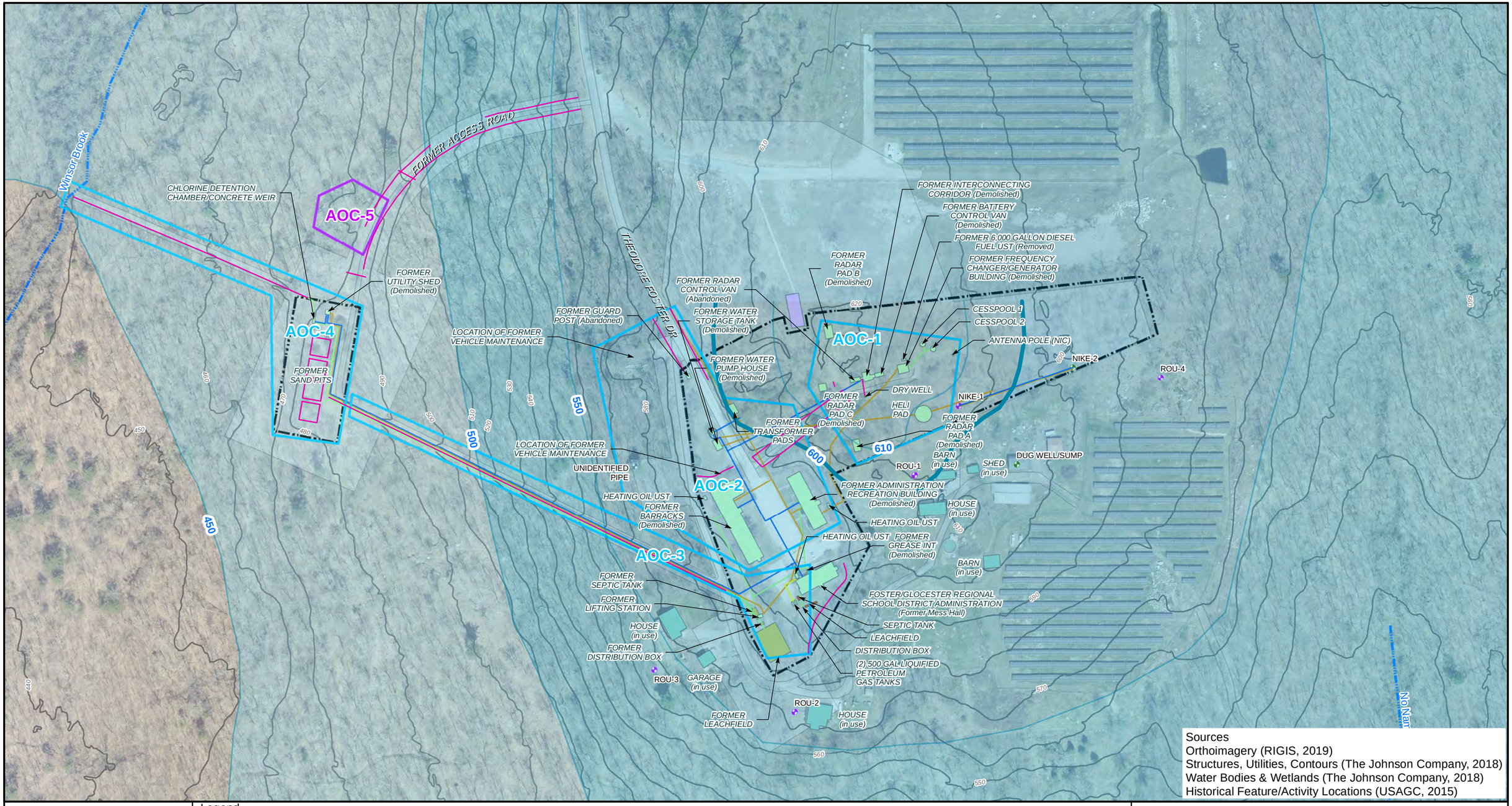
Scale in Feet

FIGURE 17-7
IDENTIFIED FRACTURED BEDROCK

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_17_7_Identified_Fractured_Bedrock.mxd



Sources
Orthoimagery (RIGIS, 2019)
Structures, Utilities, Contours (The Johnson Company, 2018)
Water Bodies & Wetlands (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	05/10/2020
Approved:	GH	05/10/2020
Project #:	60608703	

Legend

- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Index Contour (10 ft)
- Utility
- Electrical Line
- Liquefied Petroleum Gas Line
- Sewer Line
- Water Line
- Drainage

Groundwater Elevation in Overburden (Johnson Company, 2018)

- 610 - 650 ft
- 600 - 610 ft
- 550 - 600 ft
- 500 - 550 ft
- 450 - 500 ft

N

0 100 200

Scale in Feet

FIGURE 17-8
WET SEASON OVERBURDEN GROUNDWATER ELEVATION
DRAFT
FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

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Appendix B

Tables

TABLE 1
Piezometer Well Construction
Former NIKE PR-79 Control Area / Project Number D01RI0063/02
Foster, Rhode Island

Piezometer	Ground Elevation (feet msl)	Top of PVC (feet msl)	Top of Screen (feet bgs)	Top of Screen (feet msl)	Bottom of Screen (feet bgs)	Bottom of Screen (feet msl)	Refusal (feet bgs)
PZ-001	620.43	623.12	5.50	614.93	15.5	604.93	16.0
PZ-002	620.11	622.85	4.00	616.11	14.0	606.11	14.0
PZ-003	615.32	618.39	4.00	611.32	14.0	601.32	15.0
PZ-004	619.11	621.48	6.00	613.11	16.0	603.11	19.0
PZ-005	612.88	615.18	6.50	606.38	11.5	601.38	12.0
PZ-006	617.25	620.30	7.00	610.25	12.0	605.25	12.5
PZ-007	618.12	620.55	3.00	615.12	13.0	605.12	14.5
PZ-009	617.59	619.89	7.80	609.79	12.8	604.79	13.5
PZ-010	617.56	620.04	3.90	613.66	13.9	603.66	14.0
PZ-011	619.94	622.55	4.00	615.94	14.0	605.94	24.5
PZ-014	614.24	617.39	7.50	606.74	12.5	601.74	13.0
PZ-015	600.39	NM	3.00	597.39	8.0	592.39	8.0
PZ-019	618.40	621.94	4.00	614.40	14.0	604.40	20.0
PZ-022	617.18	619.92	4.00	613.18	14.0	603.18	17.5

Notes:
BGS - Below Ground Surface
MSL - Mean Sea Level
NM - Not Measured
PVC - Polyvinyl Chloride

Appendix C

AECOM Field Standard Operating Procedures

Utility Clearance

Procedure 3-01

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under contract to the Unites States Army Corp of Engineers (USACE).

1.3 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.

1.4 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.

1.5 If there are procedures, whether it be from AECOM, state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific SAP.

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Field and subcontractor personnel shall adhere to a site-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP).

3.0 Terms and Definitions

3.1 **Utility**

For the proposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

3.2 **As-Built Plans**

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

3.3 **One-Call**

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day, and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at www.call811.com.

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines, but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type or work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

3.4 **Toning**

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

4.0 Training and Qualifications

4.1 The **Project Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Site Supervisor** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Site Supervisor/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

6.0 Procedure

Proceed wtih the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.

6.1 **Prepare Preliminary Site Plan**

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific Sampling and Analysis Plan (SAP) or Work Plan. Include as many of the cultural and natural features as practical in this plan.

6.2 **Review Background Information**

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the CTO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The Project Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a project can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

6.3 **Site Visit/Locate Utilities/Toning**

- Prior to the initiation of field activities, the Site Supervisor similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Site Supervisor, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radiodetection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary

site plan. Alternatively, the Site Supervisoror designee shall document the results of the survey on the preliminary site plan.

- Report to the Site Supervisor anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed exploration or excavation areas. If this is required, the Site Supervisor or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

6.4 **Prepare Site Plan**

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the USACE and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan with the NTR to verify its accuracy prior to initiating subsurface sampling activities.

7.0 Quality Control and Assurance

7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.

8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Caryn DeJesus Senior Scientist Amanda Martin Environmental Scientist	Bob Shoemaker Senior Scientist Mark Kauffman Program Manager	Rev 0 – Initial Issue (June 2012) Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Logbooks

Procedure 3-02

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 In order to keep the logbook clean, store it in a clean location and use it only when outer gloves used for PPE have been removed.

3.0 Terms and Definitions

3.1 **Logbook**

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 **Data Form**

A data form is a predetermined format utilized for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

4.0 Training and Qualifications

4.1 The **Project Manager** or **designee** is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The **Project Manager** shall review the field logbook on at least a monthly basis. The **Project Manager** or **designee** is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.

4.2 A knowledgeable individual such as the **Site Supervisor, Project Manager**, or **Program Quality Manager** shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the dated signature of the reviewer on the last page or page immediately following the material reviewed.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Site Supervisor** is responsible for ensuring that all **field personnel** follow these procedures and that the logbook is completed properly and daily. The **Site Supervisor** is also responsible for submitting copies to the **Project Manager**, who is responsible for filing them and submitting a copy (if required by the Statement of Work).

4.5 The **logbook user** is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The **logbook user** is also responsible for safeguarding the logbook while having custody of it.

4.6 All **field personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Field logbooks shall be bound field notebooks with water-repellent pages.

5.2 Pens shall have indelible black ink.

6.0 Procedure

6.1 The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

6.2 Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

6.3 Enter field descriptions and observations into the logbook, as described in Attachment 1, using indelible black ink.

6.4 Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all on-site activities and entries made in logbooks/forms;
- Site name and description;
- Site location by longitude and latitude, if known;
- Weather conditions, including temperature and relative humidity;
- Fieldwork documentation, including site entry and exit times;
- Descriptions of, and rationale for, approved deviations from the work plan (WP) or field sampling plan;
- Field instrumentation readings;
- Names, job functions, and organizational affiliations of on-site personnel;
- Photograph references;
- Site sketches and diagrams made on site;
- Identification and description of sample morphology, collection locations, and sample numbers;
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain-of-custody (COC) information such as sample identification numbers cross-referenced to COC sample numbers;
- Sample naming convention;
- Field quality control (QC) sample information;
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations;

- Meeting information;
- Important times and dates of telephone conversations, correspondence, or deliverables;
- Field calculations;
- PPE level;
- Calibration records;
- Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number);
- Equipment decontamination procedures and effectiveness;
- Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested; and
- User signatures.

6.5 The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.

6.6 At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.

6.7 Enter logbook page numbers on each page to facilitate identification of photocopies.

6.8 If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.

6.9 At least weekly and preferably daily, the **preparer** shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

7.0 Quality Control and Assurance

7.1 Review per Section 4.2 shall be recorded.

8.0 Records, Data Analysis, Calculations

8.1 Retain the field logbook as a permanent project record. If a particular CTO requires submittal of photocopies of logbooks, perform this as required.

8.2 Deviations from this procedure shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

9.1 Attachment 1 – Description of Logbook Entries

9.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue \
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; convert to AECOM SOP for use on USACE HTRW projects (APril 2017)

Attachment 1
Description of Logbook Entries

Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

Recordkeeping, Sample Labeling, and Chain-of-Custody

Procedure 3-03

1.0 Purpose and Scope

1.1 The purpose of this standard operating procedure is to establish standard protocols for all field personnel for use in maintaining field and sampling activity records, writing sample logs, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain-of-custody/analytical request forms.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

Not applicable.

3.0 Terms and Definitions

3.1 **Logbook**

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 **Chain-of-Custody**

Chain-of-custody (COC) is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

4.0 Training and Qualifications

4.1 The **Project Manager** is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and COC forms to ensure compliance with these procedures. The **Project Manager** shall review COC forms on a monthly basis at a minimum.

4.2 The **Project Manager** and **Program Quality Manager** are responsible for evaluating project compliance with the Project Procedures Manual.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Laboratory Project Manager** or **Sample Control Department Manager** is responsible for reporting any sample documentation or COC problems to the **Project Manager** or **Laboratory Coordinator** within 24 hours of sample receipt.

4.5 The **Site Supervisor** is responsible for ensuring that all **field personnel** follow these procedures. The **Laboratory Coordinator** is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analysis plan. The **Project Manager** or **Laboratory Coordinator** is responsible for notifying the **laboratory**, **data managers**, and **data validators** in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with

the laboratory and must be made in accordance with a respective contract (e.g., remedial action contract).

4.6 All **field personnel** are responsible for following these procedures while conducting sampling activities. **Field personnel** are responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

5.0 Procedure

This procedure provides standards for documenting field activities, labeling the samples, documenting sample custody, and completing COC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

5.1 Recordkeeping

The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct each day's events. Field logs such as soil boring logs and ground-water sampling logs will also be used. These procedures are described in Procedure 3-02, *Logbooks*.

5.2 Sample Labeling

Affix a sample label with adhesive backing to each individual sample container. Place clear tape over each label (preferably prior to sampling) to prevent the labels from tearing off, falling off, being smeared, and to prevent loss of information on the label. Record the following information with a waterproof marker on each label:

- Project name or number (optional);
- COC sample number;
- Date and time of collection;
- Sampler's initials;
- Matrix (optional);
- Sample preservatives (if applicable); and
- Analysis to be performed on sample (this shall be identified by the method number or name identified in the subcontract with the laboratory).

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

5.3 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. Maintain custody of samples in accordance with the U.S. Environmental Protection Agency (EPA) COC guidelines prescribed in EPA *NEIC Policies and Procedures*, National Enforcement Investigations Center, Denver, Colorado, revised May 1986; EPA *RCRA Ground Water Monitoring Technical Enforcement Guidance Document* (TEGD); *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA OSWER Directive 9355 3-01); Appendix 2 of the *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*; and *Test Methods for Evaluating Solid Waste* (EPA SW-846)

A description of sample custody procedures is provided below.

5.3.1 Sample Collection Custody Procedures

According to the U.S. EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view;
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal);
- It is retained in a secured area with restricted access; and
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Place custody seals on sample containers immediately after sample collection and on shipping coolers if the cooler is to be removed from the sampler's custody. Place custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials; and
- Date and time that the sample/cooler was sealed.

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment 1.

Field personnel shall also log individual samples onto COC forms (carbon copy or computer generated) when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 7.4, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The **samplers** will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the **person delivering the samples for transport** will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the **sampler** and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, **both parties** shall sign and date the accompanying carbon copy COC forms, and the **individual relinquishing the samples** shall retain a copy of each form. One exception is when the samples are shipped; the **delivery service personnel** will not sign or receive a copy because they do not open the coolers. The **laboratory** shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment 2.

5.3.2 Laboratory Custody Procedures

The following custody procedures are to be followed by an **independent laboratory** receiving samples for chemical analysis; the procedures in their Naval Facilities Engineering Service Center-evaluated Laboratory Quality Assurance Plan must follow these same procedures. A **designated sample custodian** shall take custody of all samples upon their arrival at the analytical laboratory. The **custodian** shall inspect all sample labels and COC forms to ensure that the information is consistent, and that each is properly completed. The **custodian** will also measure the temperature of the temperature blank in the coolers upon arrival using either a National Institute for Standards and Technology calibrated thermometer or an infra-red temperature gun. The **custodian** shall note the condition of the samples including:

- If the samples show signs of damage or tampering;
- If the containers are broken or leaking;
- If headspace is present in sample vials;
- If proper preservation of samples has occurred (made by pH measurement, except volatile organic compounds [VOCs] and purgeable total petroleum hydrocarbons [TPH] and temperature). The pH of VOC and purgeable TPH samples will be checked by the **laboratory analyst** after the sample aliquot has been removed from the vial for analysis; and
- If any sample holding times have been exceeded.

All of the above information shall be documented on a sample receipt sheet by the **custodian**.

Discrepancies or improper preservation shall be noted by the **laboratory** as an out-of-control event and shall be documented on an out-of-control form with corrective action taken. The out-of-control form shall be signed and dated by the **sample control custodian** and **any other persons** responsible for corrective action. An example of an out-of-control form is included as Attachment 4.

The **custodian** shall then assign a unique laboratory number to each sample and distribute the samples to secured storage areas maintained at 4 degrees Celsius (soil samples for VOC analysis are to be stored in a frozen state until analysis). The unique laboratory number for each sample, COC sample number, client name, date and time received, analysis due date, and storage shall also be manually logged onto a sample receipt record and later entered into the laboratory's computerized data management system. The **custodian** shall sign the shipping bill and maintain a copy.

Laboratory personnel shall be responsible for the care and custody of samples from the time of their receipt at the laboratory through their exhaustion or disposal. Samples should be logged in and out on internal laboratory COC forms each time they are removed from storage for extraction or analysis.

5.4 Completing COC/Analytical Request Forms

COC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment 2 is an example of a generic COC/analytical request form that may be used by **field personnel**. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Attachment 3 is an example of a completed site-specific COC/analytical request form, with box numbers identified and discussed in text below.

COC forms tailored to each CTO can be drafted and printed onto multi-ply forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; TAT; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, place all VOC analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

Box 2 **Bill To:** List the name and address of the person/company to bill only if it is not in the subcontract with the laboratory.

Box 3 **Sample Disposal Instructions:** These instructions will be stated in the Master Service Agreement or each CTO statement of work with each laboratory.

Shipment Method: State the method of shipment (e.g., hand carry or air courier via FedEx or DHL).

Comments: This area shall be used by the field team to communicate observations, potential hazards, or limitations that may have occurred in the field or additional information regarding analysis (e.g., a specific metals list, samples expected to contain high analyte concentrations).

Box 4 **Cooler No.:** This will be written on the inside or outside of the cooler and shall be included on the COC. Some laboratories attach this number to the trip blank identification, which helps track samples for VOC analysis. If a number is not on the cooler, field personnel shall assign a number, write it on the cooler, and write it on the COC.

QC Level: Enter the reporting quality control (QC) requirements (e.g., Full Data Package, Summary Data Package).

Turnaround time (TAT): TAT will be determined by a sample delivery group (SDG), which may be formed over a 14-day period, not to exceed 20 samples. Once the SDG has been completed, standard TAT is 21 calendar days from receipt of the last sample in the SDG. Entering NORMAL or STANDARD in this field will be acceptable. If quicker TAT is required, it shall be in the subcontract with the laboratory and reiterated on each COC to remind the laboratory.

Box 5 **Type of Containers:** Write the type of container used (e.g., 1-liter glass amber, for a given parameter in that column).

Preservatives: Field personnel must indicate on the COC the correct preservative used for the analysis requested. Indicate the pH of the sample (if tested) in case there are buffering conditions found in the sample matrix.

Box 6 **Sample Identification (ID) Number:** This is typically a five-character alphanumeric identifier used by the contractor to identify samples. The use of this identifier is important since the laboratories are restricted to the number of characters they are able to use. Sample numbering shall be in accordance with the project-specific sampling and analysis plan.

Description (Sample ID): This name will be determined by the location and description of the sample, as described in the project-specific sampling and analysis plan. This sample identification should not be submitted to the laboratory, but should be left blank. If a computer COC version is used, the sample identification can be input, but printed with this block black. A cross-referenced list of the COC Sample Number and sample identification must be maintained separately.

Date Collected: Record the collection date in order to track the holding time of the sample. Note: For trip blanks, record the date it was placed in company with samples.

Time Collected: When collecting samples, record the time the sample is first collected. Use of the 24-hour military clock will avoid a.m. or p.m. designations (e.g., 1815 instead of 6:15 p.m.). Record local time; the laboratory is responsible for calculating holding times to local time.

Lab ID: This is for laboratory use only.

Box 7 **Matrix/QC:** Identify the matrix (e.g., water, soil, air, tissue, fresh water sediment, marine sediment, or product). If a sample is expected to contain high analyte concentrations (e.g., a tank bottom sludge or distinct product layer), notify the laboratory in the comment section. Mark an "X" for the sample(s) that have extra volume for laboratory QC matrix spike/matrix spike duplicate (MS/MSD) purposes. The sample provided for MS/MSD purposes is usually a field duplicate.

Box 8 **Analytical Parameters:** Enter the parameter by descriptor and the method number desired (e.g., BTEX 8260B, PAHs 8270C, etc.). Whenever practicable, list the parameters as they appear in the laboratory subcontract to maintain consistency and avoid confusion.

If the COC does not have a specific box for number of sample containers, use the boxes below the analytical parameter, to indicate the number of containers collected for each parameter.

Box 9 **Sampler's Signature:** The person who collected samples must sign here.

Relinquished By: The person who turned over the custody of the samples to a second party other than an express mail carrier, such as FedEx or DHL, must sign and date here.

Received By: Typically, a representative of the receiving laboratory signs and dates here. Or, a field crew member who delivered the samples in person from the field to the laboratory might sign here. A courier, such as FedEx or DHL, does not sign here because they do not open the coolers. It must also be used by the prime contracting laboratory when samples are to be sent to a subcontractor.

Relinquished By: In the case of subcontracting, the primary laboratory will sign and date the Relinquished By space and fill out an additional COC to accompany the samples being subcontracted.

Received By (Laboratory): This space is for the final destination (e.g., at a subcontracted laboratory). A representative of the final destination (e.g., subcontracted laboratory) must sign and date here.

Box 10 **Lab No. and Questions:** This box is to be filled in by the laboratory only.

Box 11 **Control Number:** This number is the "COC" followed by the first contractor identification number in that cooler, or contained on that COC. This control number must be unique (i.e., never used twice). Record the date the COC is completed. It should be the same date the samples are collected.

Box 12 **Total # of Containers:** Sum the number of containers in that row.

Box 13 **Totals:** Sum the number of containers in each column. Because COC forms contain different formats depending on who produced the form, not all of the information listed in items 1 to 13 may be recorded; however, as much of this information as possible shall be included.

6.0 Quality Control and Assurance

6.1 Recordkeeping, sample labeling, and chain-of-custody activities must incorporate quality control measures to ensure accuracy and completeness.

6.2 Deviations from this procedure or the project-specific CTO work plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

7.0 Records, Data Analysis, Calculations

7.1 The COC/analytical request form shall be faxed approximately daily to the **Laboratory Coordinator** for verification of accuracy. Following the completion of sampling activities, the sample

logbook and COC forms will be transmitted to the **Project Manager** for storage in project files. The **data validators** shall receive a copy also. The original COC/analytical request form shall be submitted by the **laboratory** along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in the records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 Attachment 1 – Chain-of-Custody Seal

8.2 Attachment 2 – Generic Chain-of-Custody/Analytical Request Form

8.3 Attachment 3 – Sample Completed Chain-of-Custody

8.4 Attachment 4 – Sample Out-of-Control Form

8.5 Environmental Protection Agency, United States (EPA). 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA.* Interim Final. EPA/540/G-89/004. Office of Emergency and Remedial Response. October.

8.6 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

8.7 EPA. 1997. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. 3rd ed., Final Update IIIA. Office of Solid Waste.

8.8 Water Resources Control Board, State of California. 1988. *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*. August.

8.9 Procedure 3-02, *Logbooks.*

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Attachment 1
Chain-of-Custody Seal

CHAIN-OF-CUSTODY SEAL

[LABORATORY]	SAMPLE NO.	DATE	SEAL BROKEN BY
	SIGNATURE		DATE
	PRINT NAME AND TITLE *(Inspector, Analyst or Technician*		

Attachment 2
Generic Chain-of-Custody / Analytical Request Form

M901376

CHAIN OF CUSTODY RECORD Page ____ of ____

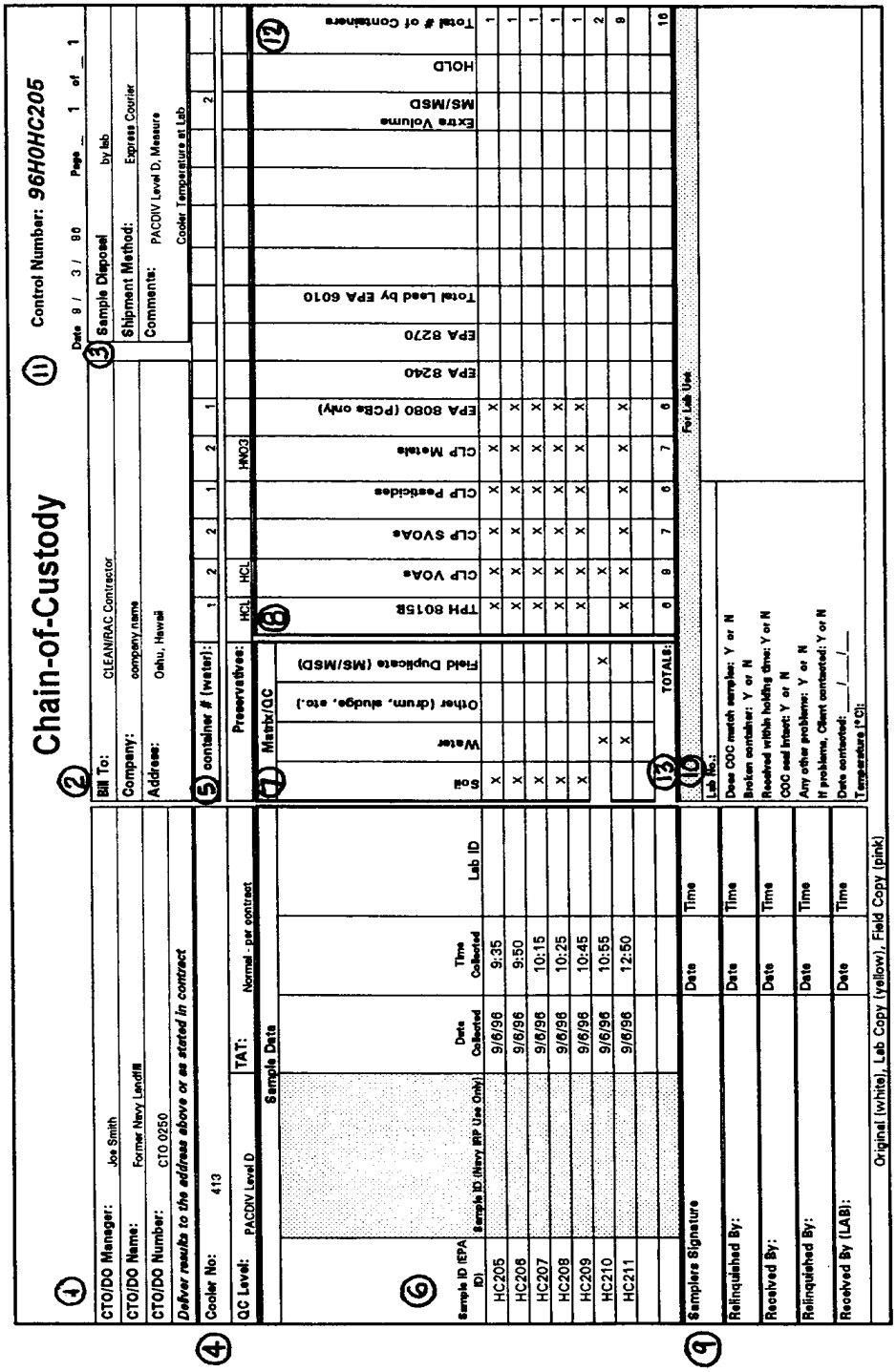
Client/Project Name:	Project Location:
Project Number:	Field Logbook No.:
Sampler: (Print Name) /Affiliation:	Chain of Custody Tape No.:
Signature:	Send Results/Report to:

Analysis Requested

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered							Lab I.D.	Remarks

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	Analytical Laboratory (Destination):
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	Serial No.

Attachment 3
Sample Completed Chain-of-Custody



Attachment 4
Sample Out-of-Control Form

OUT OF CONTROL FORM	Status	Date	Initial
	Noted OOC		
	Submit for CA*		
	Resubmit for CA*		
	Completed		

Date Recognized:	By:
Dated Occurred:	Matrix
Parameter (Test Code):	Method:
Analyst:	Supervisor:

Samples Affected (List by Accession AND Sample No.)

1. Type of Event (Check all that apply)		2. Corrective Action (CA)* (Check all that apply)	
	Calibration Corr. Coefficient <0.995		Repeat calibration
	%RSD>20%		Made new standards
	Blank >MDL		Reran analysis
	Does not meet criteria:		Sample(s) redigested and rerun
	Spike		Sample(s) reextracted and rerun
	Duplicate		Recalculated
	LCS		Cleaned system
	Calibration Verification		Ran standard additions
	Standard Additions		Notified
	MS/MSD		Other (please explain)
	BS/BSD		
	Surrogate Recovery		
	Calculations Error		
	Holding Times Missed		
	Other (Please explain	Comments:	

3. Results of Corrective Action	
	Return to Control (indicated with)
	Corrective Actions Not Successful - DATA IS TO BE FLAGGED with _____.

Analyst:	Date:
Supervisor:	Date:
QA Department:	Date:

Sample Handling, Storage, and Shipping

Procedure 3-04

1.0 Purpose and Scope

1.1 This standard operating procedure describes the actions to be used by personnel engaged in handling, storing, and transporting samples. The objective is to obtain samples of actual conditions with as little alteration as possible.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Avoid lifting heavy coolers with back muscles; instead, use leg muscles or dollies.

2.2 Wear proper gloves, such as blue nitrile and latex, as defined in the project-specific health and safety plan, when handling sample containers to avoid contacting any materials that may have spilled out of the sample containers.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Project Manager** and the **Laboratory Project Manager** are responsible for identifying instances of non-compliance with this procedure and ensuring that future sample transport activities comply with this procedure.

4.2 The **Site Supervisor** is responsible for ensuring that all samples are shipped according to this procedure.

4.3 **Field personnel** are responsible for the implementation of this procedure.

4.4 The **Program Quality Manager** is responsible for ensuring that sample handling, storage, and transport activities conducted during all CTOs comply with this procedure.

4.5 All **field personnel** are responsible for the implementation of this procedure.

5.0 Procedure

5.1 **Handling and Storage**

Immediately following collection, label all samples according to Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*. The lids of the containers shall not be sealed with duct tape, but may be covered with custody seals or placed directly into self-sealing bags. Place the sample containers in an insulated cooler with frozen gel packs (e.g., “blue ice”) or ice in double, sealed self-sealing bags. Samples should occupy the lower portion of the cooler, while the ice should occupy the upper portion. Place an absorbent material (e.g., proper absorbent cloth material) on the bottom of the cooler to contain liquids in case of spillage. Fill all empty space between sample containers with Styrofoam® “peanuts” or other appropriate material. Prior to shipping, wrap glass sample containers on the sides, tops, and bottoms with bubble wrap or other appropriate padding and/or surround them in Styrofoam to

prevent breakage during transport. Pack all glass containers for water samples in an upright position, never stacked or on their sides. Prior to shipment, replace the ice or cold packs in the coolers so that samples will be maintained as close to 4 degrees Celsius (°C) as possible from the time of collection through transport to the analytical laboratory. Ship samples within 24 hours or on a schedule allowing the laboratory to meet holding times for analyses. The procedures for maintaining sample temperatures at 4°C pertain to all field samples.

5.2 Shipping

Follow all appropriate U.S. Department of Transportation regulations (e.g., 49 Code of Federal Regulations [CFR], Parts 171-179) for shipment of air, soil, water, and other samples. Elements of these procedures are summarized below.

5.2.1 Hazardous Materials Shipment

Field personnel must state whether any sample is suspected to be a hazardous material. A sample should be assumed hazardous unless enough evidence exists to indicate it is non-hazardous. If not suspected to be hazardous, shipments may be made as described in the Section 5.2.2 for non-hazardous materials. If hazardous, follow the procedures summarized below.

Any substance or material that is capable of posing an unreasonable risk to life, health, or property when transported is classified as hazardous. Perform hazardous materials identification by checking the list of dangerous goods for that particular mode of transportation. If not on that list, materials can be classified by checking the Hazardous Materials Table (49 CFR 172.102 including Appendix A) or by determining if the material meets the definition of any hazard class or division (49 CFR Part 173), as listed in Attachment 2.

All **persons shipping hazardous materials** must be properly trained in the appropriate regulations, as required by HM-126F, Training for Safe Transportation of Hazardous Materials (49 CFR HM-126F Subpart H). The training covers loading, unloading, handling, storing, and transporting of hazardous materials, as well as emergency preparedness in the case of accidents. **Carriers**, such as commercial couriers, must also be trained. Modes of shipment include air, highway, rail, and water.

When shipping hazardous materials, including bulk chemicals or samples suspected of being hazardous, the proper shipping papers (49 CFR 172 Subpart C), package marking (49 CFR 172 Subpart D), labeling (49 CFR 172 Subpart E), placarding (49 CFR 172 Subpart F, generally for carriers), and packaging must be used. Attachment 1 shows an example of proper package markings. Refer to a copy of 49 CFR each time hazardous materials/potentially hazardous samples are shipped.

According to Section 2.7 of the International Air Transport Association Dangerous Goods Regulations publication, very small quantities of certain dangerous goods may be transported without certain marking and documentation requirements as described in 49 CFR Part 172; however, other labeling and packing requirements must still be followed. Attachment 2 shows the volume or weight for different classes of substances. A “Dangerous Goods in Excepted Quantities” label must be completed and attached to the associated shipping cooler (Attachment 3). Certain dangerous goods are not allowed on certain airlines in any quantity.

As stated in item 4 of Attachment 4, the Hazardous Materials Regulations do not apply to hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and sodium hydroxide (NaOH) added to water samples if their pH or percentage by weight criteria is met. These samples may be shipped as non-hazardous materials as discussed below.

5.2.2 Non-Hazardous Materials Shipment

If the samples are suspected to be non-hazardous based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as non-hazardous.

When a cooler is ready for shipment to the laboratory, place two copies of the chain-of-custody form inside a self-sealing bag and tape it to the inside of the insulated cooler. Then, seal the cooler with waterproof tape and label it with "Fragile," "This-End-Up" (or directional arrows pointing up), or other appropriate notices. Place chain-of-custody seals on the coolers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*.

5.2.3 Shipments from Outside the Continental United States

Shipment of sample coolers to the United States from locations outside the continental United States is controlled by the U.S. Department of Agriculture (USDA) and is subject to their inspection and regulation. A "USDA Soil Import Permit" is required to prove that the receiving analytical laboratory is certified by the USDA to receive and properly dispose of soil. In addition, all sample coolers must be inspected by a **USDA representative**, affixed with a label indicating that the coolers contain environmental samples, and accompanied by shipping forms stamped by the **USDA inspector** prior to shipment.

In addition, the U.S. Customs Service must clear samples shipped from U.S. territorial possessions or foreign countries upon entry into the United States. As long as the commercial invoice is properly completed (see below), shipments typically pass through U.S. Customs Service without the need to open coolers for inspection.

Completion and use of proper paperwork will, in most cases, minimize or eliminate the need for the USDA and U.S. Customs Service to inspect the contents. Attachment 5 shows an example of how paperwork may be placed on the outside of coolers for non-hazardous materials. For hazardous materials, refer to Section 5.2.1.

In summary, tape the paperwork listed below to the outside of the coolers to accompany sample shipments. If a shipment is made up of multiple pieces (e.g., more than one cooler), the paperwork need only be attached to one cooler, provided that the **courier** agrees. All other coolers in the shipment need only to be taped and have the address and chain-of-custody seals affixed.

1. **Courier Shipping Form & Commercial Invoice:** See Attachment 6 and Attachment 7 for examples of the information to be included on the commercial invoices for soil and water, respectively. Place the courier shipping form and commercial invoice inside a clear, plastic, adhesive-backed pouch that adheres to the package (typically supplied by the courier) and place it on the cooler lid as shown in Attachment 5.
2. **Soil Import Permit (soil only):** See Attachment 8 and Attachment 9 for examples of the soil import permit and soil samples restricted entry labels, respectively. The **laboratory** shall supply these documents prior to mobilization. The USDA often stops shipments of soil without these documents. Staple together the 2-inch × 2-inch USDA label (described below) and soil import permit, and place them inside a clear plastic pouch. The **courier** typically supplies the clear, plastic, adhesive-backed pouches that adhere to the package.

   Placing one restricted entry label as shown in Attachment 5 (covered with clear packing tape) and one stapled to the actual permit is suggested.

   The USDA does not control water samples, so the requirements for soil listed above do not apply.
3. **Chain-of-Custody Seals:** The **laboratory** should supply the seals. **CTO personnel** must sign and date these. At least two seals should be placed in such a manner that they stick to both the cooler lid and body. Placing the seals over the tape (as shown in Attachment 5), then covering it with clear packing tape is suggested. This prevents the seal from coming loose and enables detection of tampering.
4. **Address Label:** Affix a label stating the destination (laboratory address) to each cooler.
5. **Special Requirements for Hazardous Materials:** See Section 5.2.1.

Upon receipt of sample coolers at the laboratory, the **sample custodian** shall inspect the sample containers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*. The samples shall then be immediately extracted and/or analyzed, or stored in a refrigerated storage area until they are removed for extraction and/or analysis. Whenever the samples are not being extracted or analyzed, they shall be returned to refrigerated storage.

6.0 Quality Control and Assurance

6.1 Sample handling, storage, and shipping must incorporate quality control measures to ensure conformance to these and the project requirements.

7.0 Records, Data Analysis, Calculations

7.1 Maintain records as required by implementing these procedures.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or Reference

8.1 Attachment 1 – Example Hazardous Material Package Marking

8.2 Attachment 2 – Packing Groups

8.3 Attachment 3 – Label for Dangerous Goods in Excepted Quantities

8.4 Attachment 4 – SW-846 Preservative Exception

8.5 Attachment 5 – Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States

8.6 Attachment 6 – Commercial Invoice – Soil

8.7 Attachment 7 – Commercial Invoice – Water

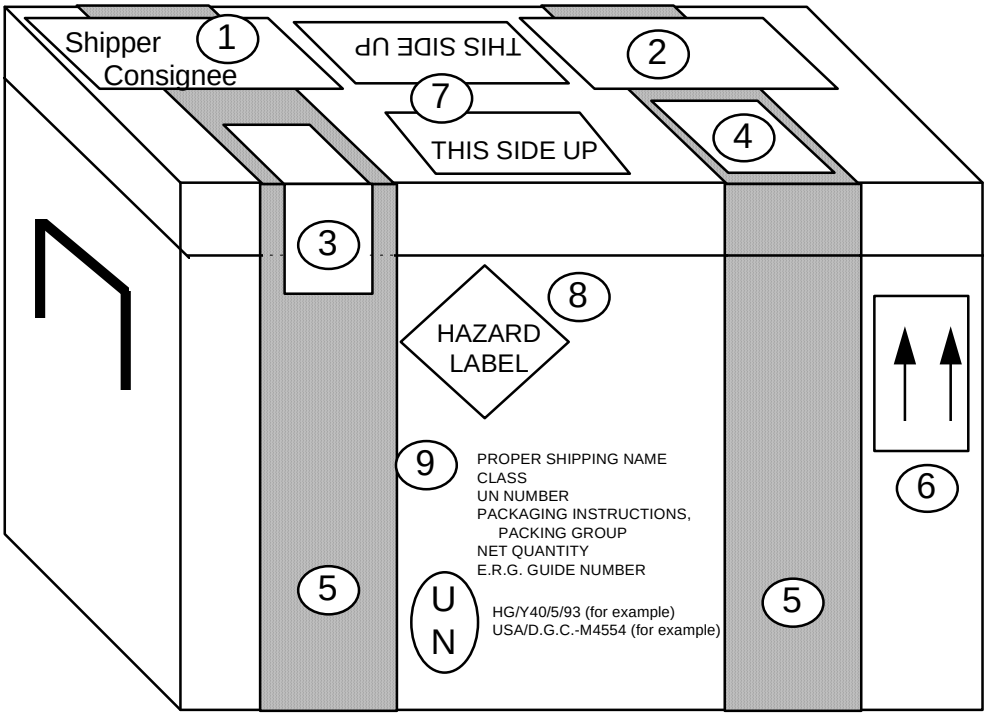
8.8 Attachment 8 – Soil Import Permit

8.9 Attachment 9 – Soil Samples Restricted Entry Labels

8.10 Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Amanda Martin Environmental Scientist	Mark Kauffman Program Manger	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW p rojects (April 2017)

Attachment 1
Example Hazardous Material Package Marking



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**
7. **THIS SIDE UP STICKERS**
8. **HAZARD LABEL**
9. **HAZARDOUS MATERIAL INFORMATION**
10. **PACKAGE SPECIFICATIONS**

Attachment 2
Packing Groups

PACKING GROUP OF THE SUBSTANCE	PACKING GROUP 1		PACKING GROUP II		PACKING GROUP III	
CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK	Packagings		Packagings		Packagings	
	Inner	Outer	Inner	Outer	Inner	Outer
1: Explosives	---------- Forbidden (Note A) ----------					
2.1: Flammable Gas	---------- Forbidden (Note B) ----------					
2.2: Non-Flammable, non-toxic gas	---------- See Notes A and B ----------					
2.3: Toxic gas	---------- Forbidden (Note A) ----------					
3. Flammable liquid	30 mL	300 mL	30 mL	500 mL	30 mL	1 L
4.1 Self-reactive substances	Forbidden		Forbidden		Forbidden	
4.1: Other flammable solids	Forbidden		30 g	500 g	30 g	1 kg
4.2: Pyrophoric substances	Forbidden		Not Applicable		Not Applicable	
4.2 Spontaneously combustible substances	Not Applicable		30 g	500 g	30 g	1 kg
4.3: Water reactive substances	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.1: Oxidizers	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.2: Organic peroxides (Note C)	See Note A		30 g or 30 mL	500 g or 250 mL	Not Applicable	
6.1: Poisons - Inhalation toxicity	Forbidden		1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - oral toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - dermal toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.2: Infectious substances	---------- Forbidden (Note A) ----------					
7: Radioactive material (Note D)	---------- Forbidden (Note A) ----------					
8: Corrosive materials	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
9: Magnetized materials	---------- Forbidden (Note A) ----------					
9: Other miscellaneous materials (Note E)	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L

Note A: *Packing groups are not used for this class or division.*
Note B: *For inner packagings, the quantity contained in receptacle with a water capacity of 30 mL. For outer packagings, the sum of the water capacities of all the inner packagings contained must not exceed 1 L.*
Note C: *Applies only to Organic Peroxides when contained in a chemical kit, first aid kit or polyester resin kit.*
Note D: *See 6.1.4.1, 6.1.4.2, and 6.2.1.1 through 6.2.1.7, radioactive material in excepted packages.*
Note E: *For substances in Class 9 for which no packing group is indicated in the List of Dangerous Goods, Packing Group II quantities must be used.*

Attachment 3
Dangerous Goods in Excepted Quantities

DANGEROUS GOODS IN EXCEPTED QUANTITIES

This package contains dangerous goods in excepted small quantities and is in all respects in compliance with the applicable international and national government regulations and the IATA Dangerous Goods Regulations.

_____

Signature of Shipper

_____ _____

Title Date

_____

_____

Name and address of Shipper

This package contains substance(s) in Class(es)
(check applicable box(es))

Class:	2	3	4	5	6	8	9
	☐	☐	☐	☐	☐	☐	☐

and the applicable UN Numbers are:

Attachment 4
SW-846 Preservative Exception

Measurement	Vol. Req. (mL)	Container[2]	Preservative[3,4]	Holding Time[5]
MBAS	250	P, G	Cool, 4ºC	48 Hours
NTA	50	P, G	Cool, 4ºC	24 Hours

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.

2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

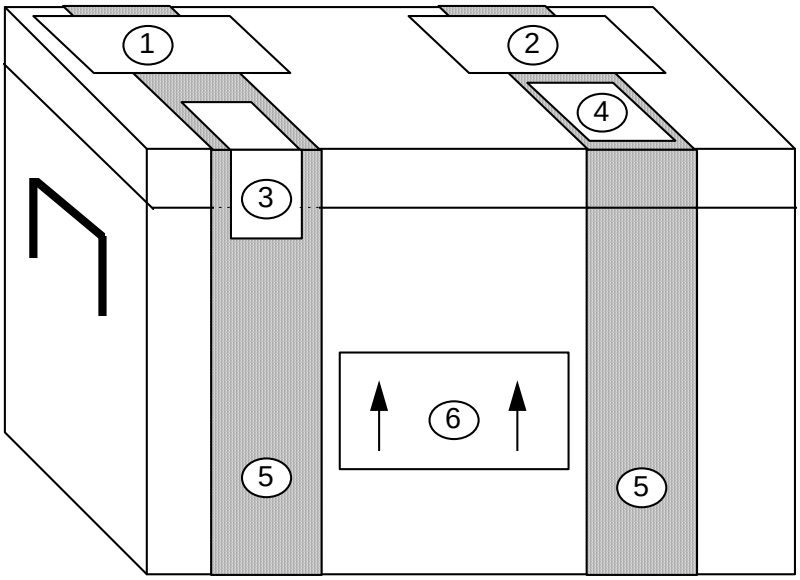
3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.

4. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. for the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentration of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

6. Should only be used in the presence of residual chlorine.

Attachment 5
Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**

Attachment 6
Commercial Invoice – Soil

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <CTO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	
INTERNATIONAL AIR WAYBILL NO.	(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Soil samples for laboratory analysis only				$1.00	$3.00

TOTAL NO. OF PKGS.	TOTAL WEIGHT	TOTAL INVOICE VALUE
3		$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

Attachment 7
Commercial Invoice – Water

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <COC #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO. _____ (NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Water samples for laboratory analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

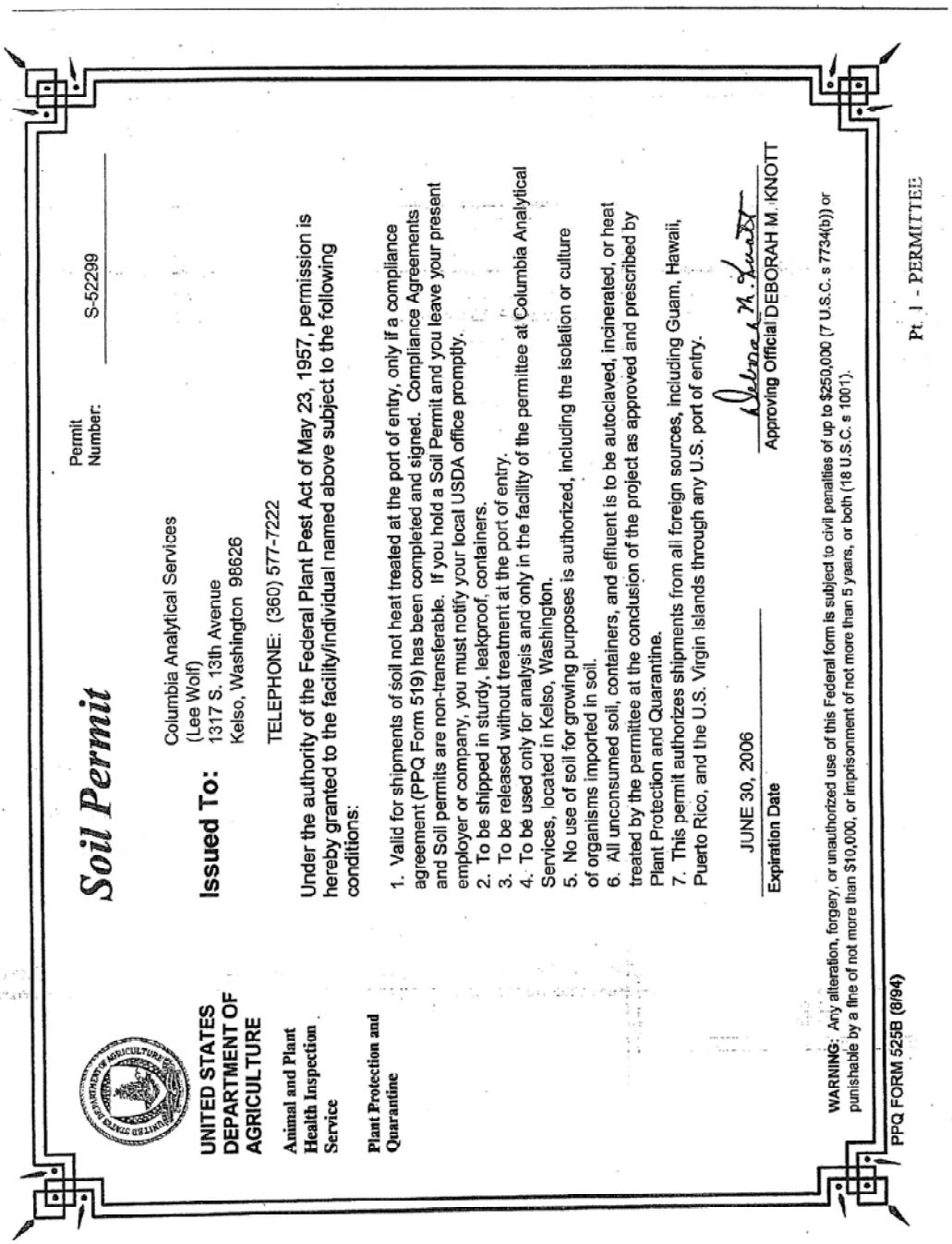
DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden _____ Joe Smith _____ 1/1/94 _____

Attachment 8
Soil Import Permit



UNITED STATES
DEPARTMENT OF
AGRICULTURE

Animal and Plant
Health Inspection
Service

Plant Protection and
Quarantine

Soil Permit

Permit
Number: S-52299

Issued To: Columbia Analytical Services
(Lee Wolf)
1317 S. 13th Avenue
Kelso, Washington 98626

TELEPHONE: (360) 577-7222

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a compliance agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil permits are non-transferable. If you hold a Soil Permit and you leave your present employer or company, you must notify your local USDA office promptly.
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry.
4. To be used only for analysis and only in the facility of the permittee at Columbia Analytical Services, located in Kelso, Washington.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the project as approved and prescribed by Plant Protection and Quarantine.
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.

JUNE 30, 2006
Expiration Date

Approving Official DEBORAH M. KNOTT

WARNING: Any alteration, forgery, or unauthorized use of this Federal form is subject to civil penalties of up to $250,000 (7 U.S.C. s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C. s 1001).

PPQ FORM 525B (8/94)

Pt. 1 - PERMITTEE

Attachment 9
Soil Samples Restricted Entry Labels

U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

SOIL SAMPLES

RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 *Edition of 12/77 may be used*

(JAN 83)

Investigation-Derived Waste Management

Procedure 3-05

1.0 Purpose and Scope

This standard operating procedure (SOP) describes activities and responsibilities of the United States Army Corp of Engineers (USACE), New England District, with regard to management of investigation-derived waste (IDW).

The purpose of this procedure is to provide guidance for the minimization, handling, labelling, temporary storage, inventory, classification, and disposal of IDW generated under the ER Program. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to IDW then those procedures may be added as an appendix to the project specific SAP.

This procedure shall serve as management-approved professional guidance and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Project Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

This procedure was developed to serve as management-approved professional guidance for the management of IDW generated. It focuses on the requirements for minimizing, segregating, handling, labeling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

2.0 Safety

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP). In the absence of a APP, work will be conducted according to the WP and/or direction from the **Site Safety and Health Officer (SSHO)**.

All **Field Personnel** responsible for IDW management must adhere to the APP and must wear the PPE specified in the site-specific APP. Generally, this includes, at a minimum, steel-toed boots or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). If safe alternatives are not achievable, discontinue site activities immediately.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Project Manager** is responsible for ensuring that IDW management activities comply with this procedure. The **Project Manager** is responsible for ensuring that all personnel involved in IDW management shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Site Supervisor** is responsible for ensuring that all IDW is managed according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

The equipment and supplies required for implementation of this SOP include the following:

- Containers for waste (e.g., [U.S. Department of Transportation] DOT approved 55-gallon open and closed top drums) and material to cover waste to protect from weather (e.g., plastic covering);
- Hazardous /non-hazardous waste drum labels (weatherproof);
- Permanent marking pens;
- Inventory forms for project file;
- Plastic garbage bags, zip lock storage bags, roll of plastic sheeting; and
- Steel-toed boots, chemical resistant gloves, coveralls, safety glasses, and any other PPE required in the HASP.

6.0 Procedure

The following procedures are used to handle the IDW.

6.1 Drum Handling

6.1.1 IDW shall be containerized using DOT approved drums. The drums shall be made of steel or plastic, have a 55-gallon capacity, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Typically 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. New steel drums are preferred over recycled drums.

6.1.2 Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.

6.1.3 For long-term IDW storage at other project locations, the DOT approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids.

6.1.4 If the ring is only partially attached to the drum lid, or if a portion of the ring is missing, select another drum lid with a sealing ring that is in sound condition.

6.1.5 To prepare IDW drums for labeling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labeling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and

disposable sampling equipment. Label all IDW drums and place them on pallets prior to storage.

6.2 Labeling

6.2.1 Containers used to store IDW must be properly labelled. Two general conditions exist: 1) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or 2) waste characteristics are unknown until additional data are obtained.

6.2.2 For situations where the waste characteristics are known, the waste containers should be packaged and labelled in accordance with state regulations and any federal regulations that may govern the labelling of waste.

6.2.3 The following information shall be placed on all non-hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.4 The following information shall be placed on all hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Generator information (i.e., name, address, contact telephone number);
- EPA identification number (supplied by on-site client representative);
- Date when the waste was first accumulated.

6.2.5 When the final characterization of a waste is unknown, a notification label should be placed on the drum with the words “waste characterization pending analysis” and the following information included on the label:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.6 Once the waste has been characterized, the label should be changed as appropriate for a nonhazardous or hazardous waste.

6.2.7 Waste labels should be constructed of a weatherproof material and filled out with a permanent marker to prevent being washed off or becoming faded by sunlight. It is recommended that waste labels be placed on the side of the container, since the top is more subject to weathering. However, when multiple containers are accumulated together, it also may be helpful to include labels on the top of the containers to facilitate organization and disposal.

6.2.8 Each container of waste generated shall be recorded in the field notebook used by the person responsible for labelling the waste. After the waste is disposed of, either by transportation off-site or disposal on-site in an approved disposal area, an appropriate record shall be made in the same field notebook to document proper disposition of IDW.

6.3 Types of Site Investigation Waste

Several types of waste are generated during site investigations that may require special handling. These include solid, liquid, and used PPE, as discussed further below.

Solid Waste

Soil cuttings from boreholes will typically be placed in containers unless site specific requirements allow for soil cuttings to be placed back into the borehole after drilling is complete. Drilling mud generated during investigation activities shall be collected in containers. Covers should be included on the containers and must be secured at all times and only open during filling activities. The containers shall be labelled in accordance with this SOP. An inventory containing the source, volume, and description of material put in the containers shall be logged on prescribed forms and kept in the project file.

Non-hazardous solid waste can be disposed on-site in the designated site landfill or in a designated evaporation pond if it is liquefied. Hazardous wastes must be disposed off-site at an approved hazardous waste landfill.

Liquid Waste

Groundwater generated during monitoring well development, purging, and sampling can be collected in truck-mounted containers and/or other transportable containers (i.e., 55-gallon drums). Lids or bungs on drums must be secured at all times and only open during filling or pumping activities. The containers shall be labelled in accordance with this SOP. Non-hazardous liquid waste can be disposed of in one of the designated lined evaporation ponds on-site. Hazardous wastes must be handled separately and disposed off-site at an approved hazardous waste facility.

Personal Protective Equipment

PPE that is generated throughout investigation activities shall be placed in plastic garbage bags. If the solid or liquid waste that was being handled is characterized as hazardous waste, then the corresponding PPE should also be disposed as hazardous waste. If not, all PPE should be disposed as non-hazardous waste in the designated on-site landfill. Trash that is generated as part of field activities may be disposed of in the landfill as long as the trash was not exposed to hazardous media.

6.4 Waste Accumulation On-Site

6.4.1 Solid, liquid, or PPE waste generated during investigation activities that are classified as nonhazardous or “characterization pending analysis” should be disposed of as soon as possible. Until disposal, such containers should be inventoried, stored as securely as possible, and inspected regularly, as a general good practice.

6.4.2 Solid, liquid, or PPE waste generated during investigation activities that are classified as hazardous shall not be accumulated on-site longer than 90 days. All hazardous waste containers shall be stored in a secured storage area. The following requirements for the hazardous waste storage area must be implemented:

- Proper hazardous waste signs shall be posted as required by any state or federal statutes that may govern the labelling of waste;
- Secondary containment to contain spills;
- Spill containment equipment must be available;
- Fire extinguisher;
- Adequate aisle space for unobstructed movement of personnel.

6.4.3 Weekly storage area inspections shall be performed and documented to ensure compliance with these requirements. Throughout the project, an inventory shall be maintained to itemize the type and quantity of the waste generated.

6.5 **Waste Disposal**

6.5.1 Solid, liquid, and PPE waste will be characterized for disposal through the use of client knowledge, laboratory analytical data created from soil or groundwater samples gathered during the field activities, and/or composite samples from individual containers.

6.5.2 All waste generated during field activities will be stored, transported, and disposed of according to applicable state, federal, and local regulations. All wastes classified as hazardous will be disposed of at a licensed treatment storage and disposal facility or managed in other approved manners.

6.5.3 In general, waste disposal should be carefully coordinated with the facility receiving the waste. Facilities receiving waste have specific requirements that vary even for non-hazardous waste, so characterization should be conducted to support both applicable regulations and facility requirements.

6.6 **Regulatory Requirements**

The following federal and state regulations shall be used as resources for determining waste characteristics and requirements for waste storage, transportation, and disposal:

- Code of Federal Regulations (CFR), Title 40, Part 261;
- CFR, Title 49, Parts 172, 173, 178, and 179.

6.7 **Waste Transport**

A state-certified hazardous waste hauler shall transport all wastes classified as hazardous. Typically, the facility receiving any waste can coordinate a hauler to transport the waste. Shipped hazardous waste shall be disposed of in accordance with all RCRA/USEPA requirements. All waste manifests or bills of lading will be signed either by the client or the client's designee.

7.0 Quality Control and Assurance

7.1 Management of IDW must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 Maintain records as required by implanting the procedures in this SOP.

8.2 Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/-fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of Energy, United States (DOE). 1994. *The Off-Site Rule*. EH-231-020/0194. Office of Environmental Guidance. March.

1999. *Management of Remediation Waste under the Resource Conservation and Recovery Act (RCRA)*. Office of Environmental Policy and Assistance. 20 December.

Environmental Protection Agency, United States (EPA). 1991. *Management of Investigative-Derived Wastes During Site Inspections*. Office of Emergency and Remedial Response. EPA/540/G-91/009. May.

1992a. *Guidance for Performing Site Inspections under CERCLA*. EPA/540/R-92/021. Office of Emergency and Remedial Response. September.

1992b. *Guide to Management of Investigative-Derived Wastes*. Quick reference fact sheet. OSWER Dir. 9345.3-03FS. Office of Solid Waste and Emergency Response. January.

1997a. *Sending Wastes Off Site? OSC and RPM Responsibilities under the Off-Site Rule*. EPA/540-F-97-006, Office of Solid Waste and Emergency Response. September.

1997b. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IIIA. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

1998. *Management of Remediation Waste under RCRA*. EPA/530-F-98-026. Office of Solid Waste and Emergency Response. October.

(No Date). *Compliance with the Off-Site Rule During Removal Actions.* Office of Regional Counsel (Region 3). Hendershot, Michael.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Equipment Decontamination

Procedure 3-06

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes methods of equipment decontamination, to be used for activities where samples for chemical analysis are collected or where equipment will need to be cleaned before leaving the site or before use in subsequent activities.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

It is the responsibility of the **Site Safety and Health Officer (SSHO)** to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. Typically, for equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing cleaning solvents as described in this procedure or the Work Plan (WP) and distilled water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

All **Field Personnel** responsible for equipment decontamination must adhere to the site-specific accident prevention plan (APP) and Site Safety and Health Plan (SSHP) and must wear the personal protective equipment (PPE) specified in the site-specific APP/SSHP. Generally this includes, at a minimum, Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). Air monitoring by the **SSHO** may result in an upgrade to the use of respirators and cartridges in the decontamination area; therefore, this equipment must be available on site. If safe alternatives are not achievable, discontinue site activities immediately.

In addition to the aforementioned precautions, the following sections describe safe work practices that will be employed.

2.1 **Chemical Hazards associated with Equipment Decontamination**

- Avoid skin contact with and/or incidental ingestion of decontamination solutions and water.
- Utilize PPE as specified in the site-specific HSP to maximize splash protection.
- Refer to material safety data sheets, safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE including skin and respiratory).
- Take the necessary precautions when handling detergents and reagents.

2.2 **Physical Hazards associated with Equipment Decontamination**

- To avoid possible back strain, it is recommended to raise the decontamination area 1 to 2 feet above ground level.

- To avoid heat stress, over exertion, and exhaustion, it is recommended to rotate equipment decontamination among all site personnel.
- Take necessary precautions when handling field sampling equipment.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Project Manager** is responsible for ensuring that decontamination activities comply with this procedure. The **Project Manager** is responsible for ensuring that all personnel involved in equipment decontamination shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Site Supervisor (SS)** is responsible for ensuring that all field equipment is decontaminated according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Procedure

Decontamination of equipment used in soil/sediment sampling, groundwater monitoring, well drilling and well development, as well as equipment used to sample groundwater, surface water, sediment, waste, wipe, asbestos, and unsaturated zone, is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- Location where the decontamination procedures will be conducted
- Types of equipment requiring decontamination
- Frequency of equipment decontamination
- Cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- Method for containing the residual contaminants and wash water from the decontamination process
- Use of a quality control measure to determine the effectiveness of the decontamination procedure

The following subsections describe standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

5.1 **Decontamination Area**

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

5.2 **Types of Equipment**

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters.

Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and caps, wipe sampling templates, composite liquid waste samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 Frequency of Equipment Decontamination

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 Cleaning Solutions and Techniques

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment and rinse it with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent (Alconox®, Liquinox®, or other suitable detergent) and potable water solution; (2) rinse with potable water; (3) spray with laboratory-grade isopropyl alcohol; (4) rinse with deionized or distilled water; and (5) spray with deionized or distilled water. If possible, disassemble equipment prior to cleaning. Add a second wash at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and potable water; (2) potable water rinse; (3) potable water rinse; and (4) deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent; however, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of distilled or deionized water. Steam-distilled water shall not be used in the decontamination process as this type of water usually contains elevated concentrations of metals. Decontamination solvents to be used during field activities will be specified in the CTO WP.

Rinse equipment used for measuring field parameters, such as pH (indicates the hydrogen ion concentration – acidity or basicity), temperature, specific conductivity, and turbidity with deionized or distilled water after each measurement. Also wash new, unused soil sample liners and caps with a fresh

detergent solution and rinse them with potable water followed by distilled or deionized water to remove any dirt or cutting oils that might be on them prior to use.

5.5 **Containment of Residual Contaminants and Cleaning Solutions**

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure 3-05, *IDW Management*.

6.0 Quality Control and Assurance

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

7.0 Records, Data Analysis, Calculations

Any project where sampling and analysis is performed shall be executed in accordance with an approved sampling and analysis plan. This procedure may be incorporated by reference or may be incorporated with modifications described in the plan.

Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

8.2 NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

8.3 Procedure 3-05, *IDW Management*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Land Surveying

Procedure 3-07

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for acquiring land surveying data to facilitate the location and mapping of geologic, hydrologic, geotechnical data, and analytical sampling points and to establish topographic control over project sites.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

1.5 If there are procedures, whether it be from AECOM, state and/or federal, that are not addressed in this SOP and are applicable to land surveying then those procedures may be added as an appendix to the project specific Sampling and Analysis Plan (SAP).

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to conducting fieldwork. All **field sampling personnel** must review the project-specific Accident Prevention Plan (APP) with Site-Safety and Health Plan (SSHP), paying particular attention to the control measures planned for the specific field tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSHO.

2.4 The health and safety considerations for the work associated with land surveying include:

- Slip, trips and falls associated with work in the field;

- Biological hazards associated with work in the field; and,
- Potential hazards associated with contaminants of concern (COC) that may be located in the survey area,

3.0 Terms and Definitions

3.1 Boundary Survey

Boundary surveys are conducted by Certified Land Surveyors in order to delineate a legal property line for a site or section of a site.

3.2 Global Positioning System (GPS)

A system of satellites, computers, and receivers that is able to determine the latitude and longitude of a receiver on Earth by calculating the time difference for signals from different satellites to reach the receiver.

4.0 Interferences

4.1 Commercially available GPS units typically have a level of precision of (±) 3 to 5 meters. Field corrections can be made as described in Section 8.3 below.

5.0 Training and Qualifications

5.1 Qualifications and Training

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Project Manager** is responsible for ensuring that land surveying activities comply with this procedure. The Project Manager is responsible for ensuring that all field sampling personnel involved in land surveying shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all field personnel follow these procedures. In virtually all cases, subcontractors will conduct these procedures. The SS or designee is responsible for overseeing the activities of the subcontractor and ensuring that sampling points and topographic features are properly surveyed.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the APP;
- Commercially available GPS unit; and,
- Field Logbook.

7.0 Calibration or Standardization

7.1 An authorized manufacturer's representative shall inspect and calibrate survey instruments in accordance with the manufacturer's specifications regarding procedures and frequencies. At a minimum, instruments shall be calibrated no more than six months prior to the start of the survey work.

7.2 Standards for all survey work shall be in accordance with National Oceanic and Atmospheric Administration standards and, at a minimum, with accuracy standards set forth below. The horizontal accuracy for the location of all grid intersection and planimetric features shall be (±) 0.1 feet. The horizontal accuracy for boundary surveys shall be 1 in 10,000 feet (1:10,000). The vertical accuracy for ground surface elevations shall be (±) 0.1 feet. Benchmark elevation accuracy and elevation of other permanent features, including monitoring wellheads, shall be (±) 0.01 feet.

8.0 Procedure

8.1 Theodolite/Electronic Distance Measurement (EDM)

Follow the procedures listed below during theodolite/EDM land surveying:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems and base all elevations and benchmarks established on U.S. Geological Survey datum, 1929 general adjustment.
- Reference surveyed points to Mean Sea Level (Lower Low Water Level).
- Jointly determine appropriate horizontal and vertical control points prior to the start of survey activities. If discrepancies in the survey (e.g., anomalous water level elevations) are observed, the surveyor may be required to verify the survey by comparison to a known survey mark. If necessary, a verification survey may be conducted by a qualified third party.
- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified by the project Work Plan or SAP.
- Begin and end all surveys at the designated horizontal and vertical control points to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly colored weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing or well riser that is surveyed by filing grooves into the casing/riser on either side of the surveyed point, or by marking the riser with a permanent ink marker.

8.2 Global Positioning System (GPS) to Conduct Land Survey

Follow the procedures listed below during land surveying using GPS:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems and base all elevations and benchmarks established on U.S. Geological Survey datum, 1929 general adjustment.

- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified in the project Work Plan or SAP.
- Begin and end all surveys at the designated horizontal and vertical control points (as applicable) to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly colored weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing that is surveyed by filing grooves into the casing on either side of the surveyed point.

8.3 Global Positioning System (GPS) to Position Sample Locations or Locate Site Features

Experienced field personnel may use a GPS system unit to position sample locations (e.g. grid positioned samples, soil boring locations) at a site. The decision to use field personnel or a licensed land surveyor will depend on the objectives of the survey (e.g. vertical elevation is not required) and the levels of precision required. Typically when a level of precision greater than (±) 3 to 5 meters is required, a licensed surveyor will be required. When a level of precision of (±) 3 to 5 meters is sufficient to meet project requirements (i.e. when laying sampling grids, identifying significant site features, or locating features identified in GIS figures) experienced field personnel may use commercially available, consumer-grade GPS units. Follow the procedures listed below to locate samples or site features using GPS:

- A commercially available GPS unit with Wide Angle Averaging System (WAAS), topographic map display, and waypoint storage capabilities should be used.
- If waypoints are to be imported into a GIS database, the same grid projection system should be used.
- If a permanent reference point near the site is available, it is recommended that a waypoint at this location be taken every day waypoints are stored.
- When laying out a sampling grid from a GIS map, upload the coordinates from GIS to the GPS unit, including coordinates for an easily identified, permanent, nearby feature (i.e. building corner, roadway intersection, or USGS benchmark).
- If during the initial site walk, the permanent feature identified does not overlay within (±) 5 meters as identified in the GPS unit, field corrections of the waypoints should be made.
- Field corrections can be made by adding/subtracting the difference in x,y coordinates between the field measurement of the permanent site feature and the anticipated x,y coordinates. This correction should then be applied to the x,y coordinates for each sampling location to be marked. Corrected x,y coordinates can then be uploaded into the GPS unit.
- Sampling points and site features can then be located in the field using the GPS units “Go To” function. When the distance to the sampling point or feature remains close to zero, the location can be marked.
- If no field corrections to the sampling location need to be made, or if sampling locations are to be surveyed by a licensed surveyor at a later date, no additional waypoints need to be taken. If significant changes to the sampling location are made, GPS coordinates at the corrected location shall be stored and labeled.

- It is recommended that GPS coordinates be uploaded to a storage device such as PC at the end of each day.
- Field logs shall indicate manufacturer and model number for GPS unit used, map datum and projection used, and any field corrections made. If the GPS unit cannot lock onto a WAAS system at the site, this should also be noted.

9.0 Quality Control and Assurance

None.

10.0 Data and Records Management

The surveyor shall record field notes daily using generally accepted practices. The data shall be neat, legible, in indelible ink, and easily reproducible. Copies of the surveyor's field notes and calculation forms generated during the work shall be obtained and placed in the project files.

Surveyor's field notes shall, at a minimum, clearly indicate:

- The date of the survey;
- General weather conditions;
- The name of the surveying firm;
- The names and job titles of personnel performing the survey work;
- Equipment used, including serial numbers; and,
- Field book designations, including page numbers.

A land surveyor registered in the state or territory in which the work was done shall sign, seal, and certify the drawings and calculations submitted by the surveyor.

Dated records of land surveying equipment calibration shall be provided by the surveyor and placed in the project files. Equipment serial numbers shall be provided in the calibration records.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Amanda Martin Environmetnal Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Surface Water and Liquid Sampling

Procedure 3-10

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for surface water and liquid sampling (for example, liquid characterization sampling). This SOP describes the equipment, field procedures, materials, and documentation procedures necessary to surface water samples from shallow and deep water using a variety of samplers. Specific information regarding coring locations can be found in the associated Sampling and Analysis Plan (SAP).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under contract to the United States Army Corp of Engineers (USACE).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first surface water sampling location. All **field sampling personnel** responsible for sampling activities must review the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP/ SSHP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSHO.

2.4 The health and safety considerations for the work associated with surface water sampling include:

- Proper selection of personal protective equipment for work around water bodies (e.g., personal flotation devices [PFDs]), as specified in the project-specific APP.
- Appropriate health and safety protocols for working in a boat (if applicable), as specified in the project-specific APP.
- Proper lifting techniques when retrieving surface water samplers, large muscles of the legs should be used, not the back.
- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- To avoid slip/trip/fall hazards as a result of working on wet surfaces, wear work boots/work boot covers with textured soles.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended), and in cases of extreme cold, wear fitted insulated clothing

3.0 Terms and Definitions

None.

4.0 Interferences

None.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The **Project Manager** is responsible for ensuring that surface water sampling activities comply with this procedure. The Project Manager or designee shall review all surface water sampling forms on a minimum monthly basis. The Project Manager is responsible for ensuring that all field sampling personnel involved in surface water sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure. Minimum qualifications for field sampling personnel require that one individual on the field team shall have a minimum of 6 months of experience with surface water sampling.

5.2.5 The **field sampler and/or task manager** is responsible for directly supervising the surface water sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the Program Quality Manager and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Work Plan
- Maps/Plot plan
- Tape measure
- Survey stakes, flags, or buoys
- Camera and film
- Stainless steel, plastic, or other appropriate composition (e.g., Teflon) bucket
- Laboratory supplied sampling containers
- Ziploc plastic bags for samples, and sample jars
- Logbook
- Labels
- Chain of Custody (COC) forms
- Site description forms
- Cooler(s)
- Ice
- Equipment/Apparatus
- Decontamination supplies/equipment
- Spade or shovel
- Spatula
- Scoop
- Trowel
- Task specific surface water sampling equipment

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Selection of Sampling Techniques

Proper selection of sampling points and collection methodology are essential to meeting the objectives of a surface water sampling program. Sampling points should be selected for collection of surface water samples on the basis of characteristics of the body of surface water body to be monitored, the location of the body of surface water, and its hydrologic boundaries with respect to the site. Other considerations include the contaminants of concern, logistical considerations, such as access to the surface water body, the direction of flow, and determination of a background location.

Methods of collecting surface water samples vary from hand sampling procedures at a single point to sophisticated, multipoint sampling techniques. The number and type of samples to be collected depends on the characteristics of the body of water, the amount of suspended sediment that a moving body carries, the size of the discharge area at the site, and other factors. Multipoint sampling techniques apply to larger bodies of water; the samples are composited to provide a more representative sample.

Whenever possible, the sampling device, either disposable or constructed of a nonreactive material, should hold at least 500 milliliters to minimize the number of times the liquid must be disturbed, thus reducing agitation of any sediment layers. A 1-liter polypropylene or stainless steel beaker with a pour spout and handle works well. Any sampling device might contribute contaminants to a sample. The correct sampling device will not compromise the integrity of the sample and will give the desired analytical results.

8.1.1 Shallow Water Body Surface Water Sample Collection

A dip or grab sample is appropriate for a small body of water, or for collecting near-surface samples in a larger surface water body. The sampling method involves filling a sample container by submerging it either just below the surface, or by lowering the container to a desired depth by using a weighted holder. For shallow bodies of surface water, hold the sample container carefully just beneath the water surface to avoid disturbing the streambed and stirring the sediment. Position the container's mouth so that it faces upstream, while the sampling personnel are standing downstream. Any preservative added to the sample should be added after sample collection to avoid loss of preservative. Alternatively, a transfer device may be dipped into the water, and then the contents transferred to the appropriate container containing the preservative. For near-surface sample collection in a large surface water body, a pond sampler may be used if an extended reach is required to collect a representative sample. A pond sampler consists of a single use sample container attached to a telescoping, heavy-duty, aluminium pole via an adjustable clamp attached to the end. The collection technique for shallow surface water samples can be used for near-surface samples in a large surface water body.

8.1.2 Deep Surface Water Sample Collection

For deeper surface water bodies, either sample containers or transfer devices may be used to collect a sample. A weighted holder that allows either a sample transfer device or a sample container to be lowered, opened for filling, closed, and returned to the surface is suggested for sampling deeper surface water bodies. This is because concentrations of constituents near the surface of a deeper body of surface water might differ from the total concentration distributed throughout the water column cross section and thus a surface sample would not be representative of the water body. An open container that is lowered and raised to the surface at a uniform rate so that the bottle is just filled on reaching the surface is appropriate for deeper stagnant water bodies, however this method does not collect a truly representative sample in deeper flowing surface water bodies.

Kemmerer Samplers. Collect samples near the shore unless sampling from a boat is feasible and permitted. If a boat is used, the body of water should be cross-sectioned and samples should be collected at various depths across the water in accordance with the project specific SAP. The Kemmerer Sampler consists of a glass, plastic, or Teflon bottle, a weighted sinker, a bottle stopper, and a line that is used to open the bottle and to lower and raise the sampler during sampling. The general procedure for using the sampler is as follows (or refer to manufacturer's instructions):

1. Obtain the sampler and check the knot at the bottom of the sampler for tightness and size. The knot should be sufficiently large so that it will not pull through the central tube of the sampler.
2. Assemble the weighted bottle sampler for making the cast by pulling the trip head into the trip plate. This can be done by holding the top and bottom stoppers and giving a short, hard pull to the bottom stopper.
3. Measure and mark the desired depth on the sampling line. Tie the free end of the line to the railing of the vessel to prevent accidental dropping of the sampler.

4. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely.
5. Pull out the stopper with a sharp jerk of the sampler line or by lowering a messenger down the line to trip the stoppers.
6. Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
7. Raise the sampler and cap the bottle. Untie the line from the railing and carry the sampler to your sampling station.
8. Transfer water into appropriate sample containers. Preserve the sample, if necessary, following guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.
9. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
10. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
11. Immediately place the properly labeled sample bottle(s) in a cooler with ice.
12. Wipe the sample clean and decontaminate if necessary for the collection of additional samples. Decontaminate according to the procedures in SOP 3-06 Equipment Decontamination.
13. Always store the sampler in the open position (stoppers not in the tube).

Teflon Bailers. Teflon bailers can also been used to collect samples in deep bodies of water. When the use of Teflon bailers is deemed appropriate for sampling water from a specific depth, the bailers shall be equipped with a check valve that closes during sample retrieval.

1. Attach a line that is premeasured to the appropriate sampling depth to the dedicated Teflon bailer and lower to the desired depth.
2. Ensure that the check valve is engaged tugging on the line with a sharp jerk.
3. Raise the bailer and transfer the water to sample containers. Preserve the sample, if necessary, following guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.
4. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
5. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
6. Immediately place the properly labeled sample bottle(s) in a cooler with ice.
7. A new dedicated bailer and new line should be used for each sampling location.

Peristaltic Pump. Another method of extending the reach of sampling efforts is to use a small peristaltic pump. In this method, the sample is drawn through heavy-wall Teflon tubing and pumped directly into the sample container. This system allows the operator to reach into the liquid body, sample from depth, or sweep the width of narrow streams.

If medical-grade silicon tubing is used in the peristaltic pump, the system is suitable for sampling almost any analyte, including most organics. Some volatile stripping may occur; due to the relatively high flow rate of the pump. Therefore, avoid pumping methods for sampling volatile organics. Battery-operated peristaltic pumps are available and can be easily carried by hand or with a shoulder sling, as needed. It is necessary in most situations to change both the Teflon suction line and the silicon pump tubing between sampling locations to avoid cross contamination. This action requires maintaining a sufficiently large stock of material to avoid having to clean the tubing in the field.

Peristaltic pumps work especially well for sampling large bodies of water when a near-surface sample will not sufficiently characterize the body as a whole. When sampling a liquid stream that exhibits a considerable flow rate, it may be necessary to weight the bottom of the suction line.

Use the following procedures for collecting samples using peristaltic pumps:

1. Install clean, silicone tubing in the pump head, per the manufacturer's instructions. Pharmaceutical-grade silicone tubing (e.g., PharMed tubing) may be required for some projects depending on the analyses required. Refer to the project specific SAP for specific tubing requirements. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into sample bottles but only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing. (Some types of thinner Teflon tubing may be used.).
2. Select the length of suction intake tubing necessary to reach the required sample depth and attach it to the tubing on the intake side of the pump. If necessary, a small weight composed of inert material (e.g., stainless steel) which will not react with chemicals of concern may be used to weight the intake tubing. Heavy-wall Teflon of a diameter equal to the required pump tubing will suit most applications. (A heavier wall will allow for a slightly greater lateral reach.)
3. A purge volume that is at a minimum equal to the tubing volume should be passed through the system prior to sample collection. Collect this purge volume in a bucket. Once the sample has been collected, the purged water volume can be returned to the water body.
4. Fill necessary sample bottles by allowing pump discharge to flow gently down the side of bottle with smooth laminar flow and minimal entry turbulence. Cap each bottle as it is filled.
5. Preserve the sample, if necessary, following guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.
6. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
7. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
8. Immediately place the properly labeled sample bottle in a cooler with ice.
9. Allow the system to drain thoroughly, and then disassemble.

8.2 Transfer Devices

Samples from various locations and depths can be composited if project quality objectives indicate that it is appropriate; otherwise, collect separate samples. Identify approximate sampling points on a sketch of the water body. Use the following procedures for collecting samples using transfer devices:

1. Submerge a stainless steel dipper or other suitable device, causing minimal disturbance to the surface of the water and the sediment at the floor of the surface water body. Note the approximate depth and location of the sample source (e.g., 1 foot up from bottom or just below the surface).
2. Allow the device to fill slowly and continuously.
3. Retrieve the dipper or device from the surface water with minimal disturbance.
4. Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the dipper or device edge.
5. Empty the dipper or device slowly, allowing the sample stream to flow gently down the side of the bottle with smooth laminar flow and minimal entry turbulence.
6. Continue delivery of the sample until the bottle is filled.
7. If necessary, preserve the sample according to guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.

8. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
9. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
10. Dismantle the sampler and decontaminate according to the procedures in SOP 3-06 Equipment Decontamination.

Multipoint sampling techniques that represent both dissolved and suspended constituents and both vertical and horizontal distributions are applicable to larger bodies of water. Subsequent to sample collection, multipoint sampling techniques may require a compositing and sub-sampling process to homogenize all the individual samples into the number of subsamples required to perform the analyses of interest. Homogenizing samples is discouraged for samples collected for volatile organic analysis, because aeration causes a loss of volatile compounds. If collection of composite samples is required, then include the procedure for compositing in the project-specific work plan.

The sampling devices selected must not compromise sample integrity. Collect samples with either disposable devices, or devices constructed of a nonreactive material, such as glass, stainless steel, or Teflon. The device must have adequate capacity to minimize the number of times the liquid must be disturbed, reducing agitation of any sediment layers. Further, the device must be able to transfer the water sample into the sample container without loss of volatile compounds. A single- or double-check valve or stainless steel bailer made of Teflon equipped with a bottom discharging device may be utilized.

All equipment used for sample collection must be decontaminated before and after use in accordance with Procedure 3-06 – Equipment Decontamination.

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality Control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation, holding times, container types, as well as various QC samples such as trip blanks, field blanks, equipment blanks, and field duplicates.

10.0 Data and Records Management

10.1 Field notes will be kept during sampling activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. During the completion of sampling activities, fill out the sample logbook and transmit forms to the CTO Manager for storage in project files.

10.2 Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. EPA/540/P-87/001, EPA, Office of Emergency and Remedial Response, Washington, D.C.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Monitoring Well Installation

Procedure 3-12

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the methods to be used during the installation of groundwater monitoring wells. It describes the components of monitoring well design and installation and sets forth the rationale for use of various well installation techniques in specific situations.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). In the absence of a APP/SSHP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Before well installation commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated well locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

2.3 Physical Hazards Associated with Well Installation

- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- When using an approved retractable-blade knife, always cut away from one self and make sure there are no other people in the cutting path or the retractable-blade knife.
- To avoid slip/trip/fall conditions during drilling activities, keep the area clear of excess soil cuttings and groundwater. Use textured boots/boot cover bottoms in muddy areas.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and personal protective equipment (PPE), drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- To avoid hazards associated with subsurface utilities, ensure all sampling locations have been properly surveyed as described in SOP 3-01, Utility Clearance.
- Be aware of restricted mobility caused by PPE.

3.0 Terms and Definitions

3.1 **Annulus:** The annulus is the down-hole space between the borehole wall and the well casing and screen.

3.2 **Bridge:** A bridge is an obstruction in the drill hole or annulus. A bridge is usually formed by caving of the wall of the well bore, by the intrusion of a large boulder, or by the placement of filter pack materials during well completion. Bridging can also occur in the formation during well development.

3.3 **Filter Pack:** Filter pack is sand or gravel that is smooth, uniform, clean, well-rounded, and siliceous. It is placed in the annulus of the well between the borehole wall and the well screen to prevent formation materials from entering the well and to stabilize the adjacent formation.

3.4 **Grout:** Grout is a fluid mixture of cement and water that can be forced through a tremie pipe and emplaced in the annular space between the borehole and casing to form an impermeable seal. Various additives, such as sand, bentonite, and polymers, may be included in the mixture to meet certain requirements.

3.5 **Heaving (Running) Sands:** Loose sands in a confined water-bearing zone or aquifer which tend to rise up into the drill stem when the confining unit is breached by the drill bit. Heaving sands occur when the water in the aquifer has a pressure head great enough to cause upward flow into the drill stem with enough velocity to overcome the weight of the sand.

3.6 **Sieve Analysis:** Sieve analysis is the evaluation of the particle-size distribution of a soil, sediment, or rock by measuring the percentage of the particles that will pass through standard sieves of various sizes.

4.0 Interferences

4.1 Heaving sands may be problematic in unconsolidated sands encountered below the water table.

4.2 Rotary drilling methods requiring bentonite-based drilling fluids should be used with caution to drill boreholes that will be used for monitoring well installation. The bentonite mud builds up on the borehole walls as a filter cake and permeates the adjacent formation, potentially reducing the permeability of the material adjacent to the well screen.

4.3 If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water that was introduced, either by flushing the borehole prior to well installation and/or by overpumping the well during development.

4.4 Track-mounted drill rigs are suitable for travelling on many types of landscapes that truck-mounted units cannot access, but may have limitations on extremely uneven or soft terrain.

4.5 Care should be taken to prevent cross-contamination between well locations. All drilling equipment coming in contact with potentially contaminated soil and/or groundwater will be decontaminated by the drilling subcontractor prior to initial drilling activities and between drilling locations in accordance with SOP 3-06, Equipment Decontamination.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 **Project Managers** are responsible for issuing sampling and analysis plans (SAPs) that reflect the procedures and specifications presented in this procedure. Individual municipalities, county agencies, and possibly state regulatory agencies enforce regulations that may include well construction and installation requirements. The **Project Manager** shall be familiar with current local and state regulations, and ensure that these regulations are followed. The **Project Manager** is responsible for ensuring that all personnel involved in monitoring well installation shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for direct supervision of the installation of monitoring wells and ensuring that procedures and specifications are implemented in the field in accordance with the approved SAP and well installation permits. The qualifications for the **SS** must be in accordance with local jurisdictions with authority over the operations conducted.

5.2.4 All field personnel are responsible for the implementation of this procedure.

5.2.5 The on-site hydrogeologist/engineer is expected to obtain a description of the lithologic samples obtained during the excavation and construction of a monitoring well. These data are often required to provide guidance regarding the installation of specific components of the monitoring well. Guidance for lithologic sample collection and sample description is contained within SOP 3-16, Soil and Rock Classification.

6.0 Equipment and Supplies

6.1 Materials provided by the drilling contractor may include:

- Drill rig, drill rods, hollow stem augers, etc.
- Decontamination equipment (e.g., steam cleaner, high-pressure washer, brushes, etc.)
- Decontamination pad materials
- Well screen/riser pipe with flush-threaded couplings including riser and bottom caps
- Clean, filter sand
- Bentonite chips or pellets
- Cement grout and tremie pipe
- Portland cement for well pad completion
- Steel protective riser covers and locking caps
- Weighted calibrated tape
- Split-spoon samplers
- 55-gallon drums or containers for drill cuttings, decontamination fluids, etc.

6.2 In addition to those materials provided by the drilling contractor, equipment and materials required by the project geologist/engineer may include, but is not limited to, the following:

- Photoionization Detector (PID)
- Spill kit, including at a minimum sorbent pads and shovel (if not provided by subcontractor)

- Plastic sheeting
- Teaspoon or spatula
- Resealable plastic bags
- Boring Log Records
- Decontamination materials (per SOP No. 3-06 - Equipment Decontamination)
- Weighted measuring tape for depth measurement
- Soil logging materials (e.g. USCS classification field card, millimeter rule, hand lens, etc.)
- Survey lathes or pin flags
- Digital camera
- PPE as required by the APP/SSHP
- Planning documents including the site-specific APP/SSHP and SAP
- Large indelible ink or paint pen
- Field logbook/field forms/site maps (water proof)

7.0 Procedure

7.1 General Procedures

- Specific drilling, sampling, and installation equipment and methodology will be dictated by the type of well to be installed (e.g., single case (Type II), double case (Type III), bedrock, etc.), geologic characteristics of the site, the type of contaminants being monitored, and local and state regulations.
- For access to locations when travelling over difficult terrain, an appropriate line should be chosen before mobilizing the drill rig or other support vehicles. If clearing of trees or ground cover is required, perform these activities in advance to avoid down time. Avoid wet or soft areas where possible or use ground mats and/or timbers to aid in supporting the rig as it travels. If drilling on soft material, place geomatting and ground mats under the rig tracks or stabilizers prior to drilling.
- A utility locate must be conducted to identify all underground utilities at the site prior to drilling (refer to SOP 3-01, Utility Clearance). Proper clearance procedures for aboveground/overhead utilities must also be followed as specified in the APP/SSHP.
- Although new well materials (well screen and riser pipe) generally arrive at the site boxed and sealed within plastic bags, it is sometimes necessary to decontaminate the materials prior to their use. Well materials should be inspected by the project geologist/engineer upon delivery to check for cleanliness. If the well materials appear dirty, or if local or regional regulatory guidance requires decontamination, then well material decontamination should be performed by the drilling subcontractor in accordance with SOP 3-06, Equipment Decontamination.
- The diameter of the borehole must be a minimum of 2 inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs, bentonite seals, and grout seals, and allow the passage of tremie pipe where grouting at depth is required. Bedrock wells may require reaming after coring in order to provide a large enough borehole diameter for well installation.
- When soil sampling is required (refer to the SAP), soil samples will be collected for visual logging by advancing split-spoon samplers through the augers. The soil will be visually logged by a field geologist and include lithologic characteristics (i.e., soil type, color, density, moisture content, etc.) using the the

methods described in SOP 3-16, Soil and Rock Classification. This information will be recorded on a boring/well log form, along with well construction details.

7.2 Drilling Techniques

Drilling of monitoring well boreholes may be accomplished by a variety of methods as described below. Preferred methods include those that temporarily case the borehole during drilling (i.e., hollow stem auger and sonic methods) using an override system. Other methods can be used where specific subsurface conditions or well design criteria dictate.

- Hollow stem auger (HSA) – Borings are advanced by rotating steel hollow stem augers with an attached cutting head. Soil cuttings are displaced by the cutting head and transported to the surface via continuous spiral flights attached to each auger stem. This method is widely used for unconsolidated soils that have a tendency to collapse within the boring. A bottom plug can be placed in the bottom auger to prevent soils from entering and clogging the auger, especially in the case of heaving sands. However, a bottom plug cannot be used when soil samples are to be collected through the augers. Soil plugs that accumulate in the bottom of the auger must be removed or knocked out prior to sampling or well installation.
- Solid stem auger – This type of drilling method is similar to HSA drilling using a solid stem or sealed hollow stem auger flights to advance the boring. Solid stem, continuous flight auger use is limited to semi-consolidated sediments or to cohesive or semi-cohesive unconsolidated sediments that don't have a tendency to collapse when disturbed.
- Sonic methods – Sonic drilling consists of advancing concentric hollow drill casings (inner and outer) using rotation in conjunction with axial vibration of the drill casing. Once the casings are advanced to the appropriate depth, the inner string is removed with a core of drill cuttings while the outer casing remains in place to keep the borehole open. Cuttings are removed from the inner casing relatively intact for logging or sampling purposes. This drilling method is used for a variety of soil types, from heaving sands to consolidated or indurated formations. Smearing of the formation along the borehole walls is minimal since moderate vibration and rotation techniques are used to advance the casings. Since the total borehole diameter in sonic drilling is only incrementally larger than the inner casing diameter, care should be taken during installation of the monitoring well to ensure the well is centered and adequate space is available for annular materials.
- Rotary methods (water or mud) – Rotary drilling methods consist of drill rods coupled to a drill bit that rotates and cuts through the soils to advance the borehole. Water or drilling fluid ("mud") is forced through the hollow drill rods and drill bit as the rods are rotated. The soil cuttings are forced up the borehole with the drilling fluids to the surface and the fluids recirculated. The drilling fluid provides a hydrostatic pressure that reduces or prevents the borehole from collapsing. Clean, potable water must be used for water-rotary drilling to prevent introducing trace contaminants. A sample of the potable water should be collected during the course of well installation for analysis of the same parameters defined for the groundwater samples. If mud-rotary is used to advance boreholes, potable water and bentonite drilling mud should only be used. No chemical additives shall be mixed in the drilling fluid to alter viscosity or lubricating properties. Adequate well development is essential for removal of drilling mud and fluids from the formation materials and ensure collection of representative groundwater samples.
- Rotary methods (Air) – Air rotary methods are similar to water rotary but use high air velocities in place of drilling fluids to rotate the drill bit and carry the soil cuttings up the borehole to the surface. Care must be taken to ensure that contaminants are not introduced into the air stream from compressor oils, etc. Most compressor systems are compatible with a coalescing filter system. Cuttings exiting the borehole under pressure must be controlled, especially when drilling in a zone of potential contamination. This can be accomplished by using an air diverter with hose or pipe to carry the cuttings to a waste container. Letting the cuttings blow uncontrolled from the borehole is not acceptable.

7.3 Well Construction and Installation

- If rotary drilling techniques are used, the borehole should be flushed or blown free of material prior to well installation. If hollow stem augers are used, the soil or bottom plug should be removed and the augers raised approximately six inches above the bottom of the borehole, while slowly rotating the augers to remove cuttings from the bottom of the boring. The depth of the borehole should be confirmed with a weighted, calibrated tape.
- The riser pipe and screen should be connected with flush-threaded joints and assembled wearing clean, disposable gloves. No solvent or anti-seize compound should be used on the connections. The full length of the slotted portion of the well screen and unslotted riser pipe should be measured and these measurements recorded on a well construction form (Attachment 1).
- If placed in an open borehole, the assembled well should be carefully lowered and centered in the borehole so that the well is true, straight, and vertical throughout. Centering can also be accomplished with the use of centralizers, if necessary. However, centralizers should be placed so that they do not inhibit the installation of filter sand, bentonite seal, and annular grout. Wells less than 50 deep generally do not require centralizers.
- If hollow stem augers are used, the well should be lowered through the augers and each auger flight removed incrementally as the filter sand, bentonite seal, and grout are tremmied or poured into the annular space of the well. The well should be temporarily capped before filter sand and other annular materials are installed.
- Clean, silica sand should be placed around the well screen to at least 1 foot above the top of the screen. The filter sand should be appropriately graded and compatible with the selected screen size and surrounding formation materials. In general, the filter pack should not extend more than 3 feet above the top of the screen to limit the thickness of the monitoring zone. As the filter pack is placed, a weighted tape should be lowered in the annular space to verify the depth to the top of the layer. This measurement will be recorded on the well construction form (Attachment 1). If necessary, to eliminate possible bridging or creation of voids, placement of the sand pack may require the use of a tremie pipe. Tremie pipe sandpack installations are generally suggested for deeper wells and for wells which are screened some distance beneath the water table.
- A minimum 2-foot thick layer of bentonite pellets or slurry seal will be installed immediately above the filter sand to prevent vertical flow within the boring from affecting the screened interval. Bentonite chips/pellets must be hydrated if place above the water table prior to grouting. If bridging is of concern as in the case of deep wells, powdered bentonite may be mixed with water into a very thick slurry and a tremie pipe used to place the seal to the desired depth. Placement of the bentonite seal in the borehole will be recorded on the well construction form (Attachment 1).
- The remaining annular space around the well will be grouted from the top of the bentonite seal to the surface with a grout composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.
- Each well riser will be secured with an expandable, locking cap (vented if possible). Optionally, a hole can be drilled in the upper portion of the riser to allow venting of the well.
- The well will be completed within a concrete well pad consisting of a Portland cement/sand mixture. Well pads are generally 3 feet by 3 feet square but may be larger or smaller depending on site conditions and state-specific well construction standards. Round concrete well pads are also acceptable. A minimum of 1 inch of the finished pad should be below grade to prevent washing and undermining by soil erosion.
- If completed as a flush-mount well, the well riser will be cut off approximately 4 to 6 inches below ground surface and an expandable, locking cap placed on the well riser. The area around the riser is dug out and a steel well vault or manhole cover placed over the riser and set almost flush to the ground

to protect the well. The manhole cover should be water-tight and secured with bolts to prevent casual access. The well pad will then be constructed around the well vault and slightly mounded at the center and sloping away to prevent surface water from accumulating in the well vault.

- If completed as a stick-up well, the well riser is cut approximately 2.5 to 3 feet above the ground surface and an expandable, locking cap placed on the well riser. A steel guard pipe with hinged, locking cap is placed over the well riser as a protective casing. The bottom of the guard pipe will be set approximately 2 feet below ground surface and sealed by pouring concrete from the top of the annular grout around the pipe to grade. The concrete well pad should be completed at the same time. Weep holes will be drilled in the base of the guard pipe to facilitate draining of rainwater or purge water from inside the guard pipe.
- Bumper posts or bollards may be necessary for additional well protection, especially in high traffic areas. The bumper posts should be placed around the well pad in a configuration that provides maximum protection to the well and extend a minimum of 3 feet above the ground.

7.4 Double Cased Wells

Under certain site conditions, the use of a double-cased or telescoping (Type III) well may be necessary. Installation of double-cased wells may be required to prevent the interconnection of two separate aquifers, seal off a perched aquifer without creating a vertical hydraulic conduit, prevent cross-contamination during construction of wells in deeper aquifers hydro-stratigraphically below impacted aquifers, or case off highly impacted soils present above the aquifer to prevent potential “dragging down” of contaminants.

Similar to conventional wells, construction of double-cased wells can be accomplished using a varety of drilling methods. Well construction is initiated by “keying” a large diameter, outer casing into a stratigraphic zone of low permeability (clay layer or bedrock). The size of the outer casing should be a minimum of 2 inches greater than the outside diameter of the inner casing to allow installation of annular seal materials during well completion. A pilot borehole should be drilled through the overburden soil and/or contaminated zone into a clay confining layer or bedrock. The borehole for the outer casing should be of sufficient size to contain the outer casing with a minimum of 2 inches around the outside diameter to allow sufficient annular space for tremie or pressure grouting. The boring should extend a minimum of 2 feet into a clay layer and a minimum of 1 foot into bedrock, if possible, to ensure an adequate seal. The boring should never breach a confining layer or keyed zone under any circumstances.

Once the boring is completed, the outer casing can be set in the borehole and sealed with grout. The outer casing can be set two ways, with or without a bottom cap. If no bottom cap is applied, the casing is usually driven approximately 6 inches into the clay confining unit. A grout plug is generally placed in the bottom of the casing and once set, standing water in the casing is evacuated prior to drilling below the casing. As an alternative, a cap can be placed on the bottom of the casing and if set below the water table, the casing can be filled with clean, potable water to hold down the casing in the boring. Grouting should be conducted using tremie-grouting or pressure-grouting methods by pumping grout into the annular space between the outer casing and the borehole wall from the bottom of the casing to the ground surface. Grout around the casing should be allowed to cure at least 24 hours before attempting to drill through the bottom.

Once the grout is cured, a smaller diameter drill pipe/bit is used to bore through the grout plug or bottom cap to the desired well depth. The well is then constructed as described in Section 7.3 above.

7.5 Post Installation Procedures

- Wells should be permanently labelled or marked for identification. Well tags can be used to record the site name, well number, total depth, installation date, etc. At a minimum, the well number will be written in indelible marker or paint on both the outside of the protective casing and inside beneath the casing lid, as well as on the riser pipe.

- A measuring point will be marked on the top of the riser pipe for taking water level measurements. The measuring point can be notched using a knife or saw or can be marked with a waterproof marker or paint. The measuring point will also be the point which will be surveyed for vertical elevation data.
- Upon completion, the following measurements will be taken by the field geologist/engineer and recorded on the well construction diagram.
    - Depth to static water level
    - Depth of non-aqueous phase liquid (NAPL), if present
    - Total depth of well measured from top of casing (TOC)
    - Height of well casing above ground surface
    - Height of protective casing above ground surface
- All monitoring wells will be surveyed for horizontal and vertical control by a licensed surveyor.
- Investigation-derived waste (IDW) including drill cuttings, spent materials (e.g., PPE), and decontamination water should be properly managed in accordance with SOP 3-05, IDW Management.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the SAP. Certain quality control (QC) measures should be taken to ensure proper well installation and construction in accordance with this SOP, project specific SAP, and applicable well standards.

8.2 The borehole will be checked for total open depth, and extended by further drilling or shortened by backfilling, as required before installation of the well materials.

8.3 Water level and NAPL presence will be checked during well installation to ensure that the positions of well screen, filter sand, and seals relative to water level conform to project requirements

8.4 The depth to top of each layer of annular materials (i.e., filter sand, bentonite, grout) will be verified and adjusted as necessary for proper placement.

9.0 Records, Data Analysis, Calculations

All field information will be recorded in the field logbook and/or standardized field forms by field personnel. Field data recorded will include drilling contractor information, drilling methods, well material and construction information provided on the boring logs and well construction forms, observations or problems encountered during drilling, fluid level data, and any deviations from the procedures in this SOP and other project plans. Well Construction Forms (Attachment 1) will provide visual and descriptive information the monitoring well and are often the most critical form of documentation generated during the installation of a monitoring well. The field logbook is kept as a general log of activities and should not be used in place of the boring log.

10.0 Attachments or References

10.1 Attachment 1 – Monitoring Well Construction Form

10.2 Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. Office of Solid Waste and Emergency Response. EPA/540/P-87/001.

10.3 EPA. 1990. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*. EPA/600/4-89/034. Office of Research and Development, Washington. March.

10.4 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

10.5 EPA, 2008. SESD Operating Procedure SESDGUID-101-R0: *Design and Installation of Monitoring Wells*. USEPA, Science and Ecosystem Support Division (SESD), Athens, Georgia. Effective Date February 18, 2008.

10.6 U.S. Army Corps of Engineers. 2008. Manual No. EM 385-1-1. *Safety and Health Requirements*. 15 November 2008. http://140.194.76.129/publications/eng-manuals/em385-1-1/2008_English/toc.html.

10.7 SOP 3-01, *Utility Clearance*.

10.8 SOP 3-05, *IDW Management*

10.9 SOP 3-06, *Equipment Decontamination*.

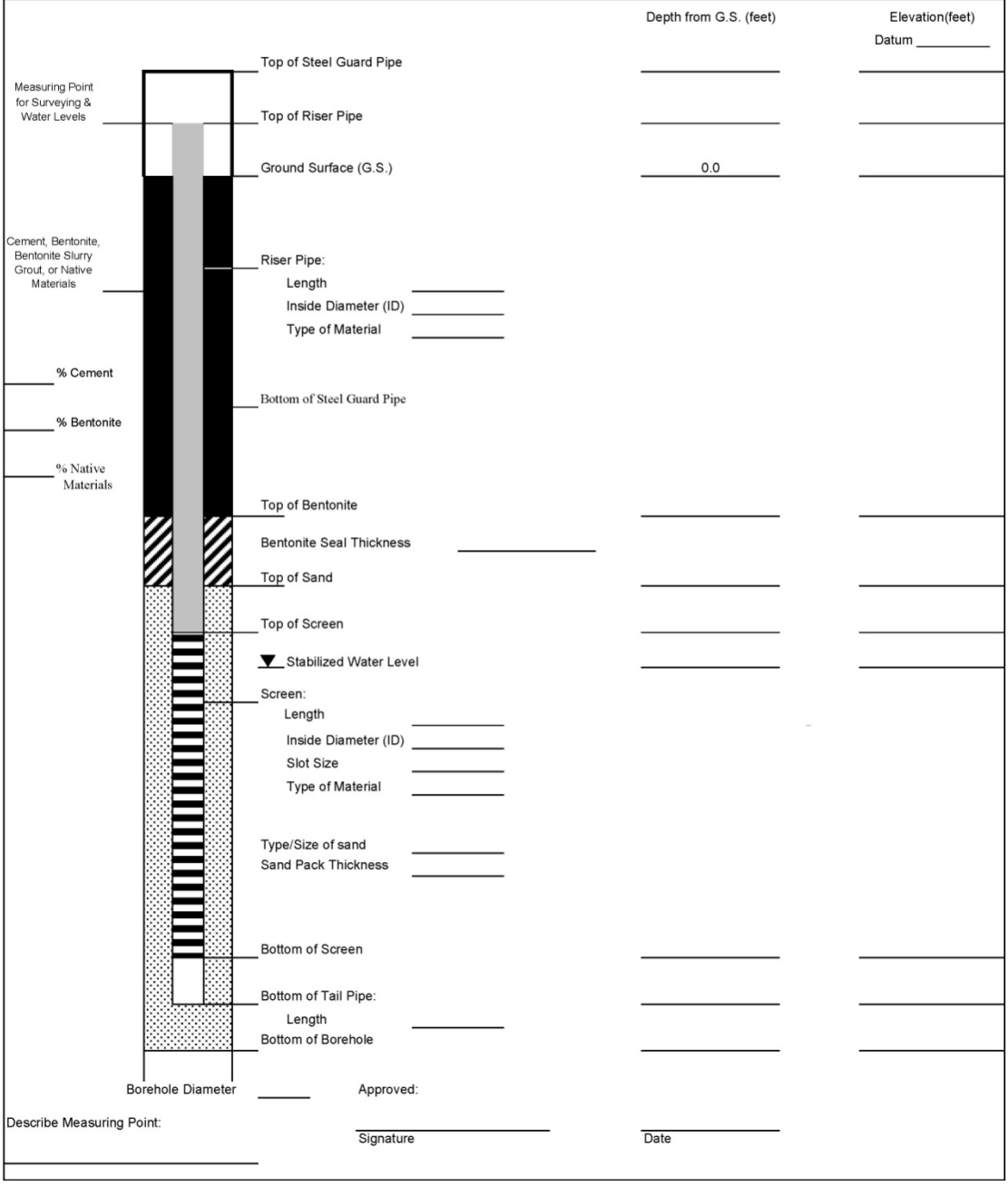
10.10 SOP 3-16, *Soil and Rock Classification.*

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Attachment 1
Monitoring Well Construction Form

	Client:	***WELL ID:***
	Project Number:	
	Site Location:	*Date installed:*
	Well Location: *Coords:*	*Inspector:*
	Method:	*Contractor:*

MONITORING WELL CONSTRUCTION DETAIL



AECOM

Boring Log

Project Name: Camp Hero Remedial Investigation	Site:	**Hole ID:**
Project Number: 60443903.4.1	*Northing:*	Total Depth (feet):
Drilling Contractor: New England Geotech	*Easting:*	Date / Time Started:
Driller:	Elevation (feet MSL): *Ground:*	Date / Time Finished:
Drilling Equipment:	▼ Water Depth During Drilling (feet bgs):	Date / Time Completed:
Drilling Method:	Logged By:	Checked By:
Borehole Diameter (inches):	Weather/Comments:	

Depth (feet)	USCS Description	Log: Graphic	Log: USCS or Rock Type	Samples: Attempted Recovered	Samples: Method	Samples: Run Number	Samples: PID/FID (ppm)	Samples: Time	Well Diagram	Remarks (list sample numbers here)
5										**Soil Sample Info** SB S#: SD: ST: Analyses: SB S#: SD: ST: Analyses:
10										**Groundwater Sample Info** Well-head PID (ppm): DTW (ft bgs): GW S#: Screen interval (ft bgs): ST: Analyses: Temp (C): pH: SC (mS/cm): ORP (mV): DO (mg/L): Turbidity (NTU):
15										

Tracking Codes: 10/19/12, 12:21

USCS Name, **Consistency/Density** (predominantly fine: very soft {n=0-1}, soft {n=2-4}, medium stiff {n=5-8}, stiff {n=9-15}, very stiff {n=16-30}, hard {n=31+}/predominently coarse: very loose {n=0-4}, loose {n=5-10}, medium dense {n=11-30}, dense {n=31-50}, very dense {n=51+}). **Moisture**, (dry, moist, wet). **Color**. **Gradation** (relative percentages of soil components). **Plasticity/Cohesiveness** (predominently fine: nonplastic, slightly plastic, low plasticity, medium plasticity, high plasticity)/predominently coarse: cohesionless, slightly cohesive, cohesive). **Stratification/Structure** (blocky, massive, lensed, etc) (contacts: sharp, gradational) (bedding: horizontal, inclined). **Cementation** (none, weak, moderate, strong). **Other descriptive elements; Geologic Origin**
S# = Sample Number, **SD** = Sample Depth, **ST** = Sample Time, **A** = Analysis.
BZ = Breathing Zone, **BG** = Background, **BH** = Borehole, **CB** = Cuttings Bin

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Boring Log (Continued)

Project Name: Camp Hero Remedial Investigation	Site:	Hole ID:

Depth (feet)	USCS Description	Log: Graphic	Log: USCS or Rock Type	Samples: Attempted Recovered	Samples: Method	Samples: Run Number	Samples: PID/FID (ppm)	Samples: Time	Well Diagram	Remarks (list sample numbers here)
20										
25										
30										

Tracking Codes: 10/19/12, 12:20

Form: 00-BLANK LOG

Monitoring Well Development

Procedure 3-13

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures used for developing newly installed monitoring wells and/or redeveloping existing wells.

1.2 The purpose of well development is to remove interferences from a well to provide better connection between the well and the formation, to improve pumping performance of the well, and to be able to collect more representative information from the well (e.g., samples, test results, etc.). Proper well development will:

- Remove drilling residuals (e.g., water, mud) from the borehole and surrounding formations;
- Improve or restore hydraulic conductivity of the surrounding formations which may have been disturbed during the drilling process;
- Remove residual fines from the well screen and sand pack (filter pack) materials, thus reducing turbidity of groundwater and permitting the collection of more representative groundwater samples.

1.3 There may be circumstances where well development is not desirable, for example, in the presence of non-aqueous phase liquids (NAPL) or other significant contamination if development could worsen the contaminant impact. If NAPL begins to intrude during development, the development process will be halted. This situation will be considered a cause for sample modification requiring approval by the Project Manager and other stakeholders, as applicable.

1.4 The applicable well development procedures for a particular site may be subject to State or local regulatory requirements. In all cases, the project team should consult their local regulatory requirements and document the selected well development procedure in the project-specific Sampling and Analysis Plan (SAP). For project-specific information refer to the Work Plan (WP) and Sampling and Analysis Plan (SAP), which takes precedence over these procedures.

1.5 This procedure is the professional guidance for work performed by AECOM under contract to the United States Army Corp of Engineers (USACE).

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project-specific Accident Precention Plan (APP) and Site Safety and Health Plan (SSHP). In the absence of a APP/SSHP, work will be conducted according to the project-specific WP, SAP, and/or direction from the Site Safety and Health Officer (SSHO).

2.2 Monitoring well development may involve chemical hazards associated with potential contaminants in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Equipment/materials used for development may react with the groundwater during development. Appropriate development equipment has been selected for the anticipated condition of the groundwater.

4.2 Appropriate development methods such as using a surge-block to flush suspended fines in the groundwater in and out of the well screen can improve the yield of wells and improve their potential to be developed successfully. However, the effectiveness of development can be significantly reduced in wells that do not yield sufficient water to allow this flushing to take place.

4.3 For formations with a significant content of fine-grained materials (silts and clays), or wells with improperly sized screens, it may not be possible to reduce turbidity to commonly acceptable levels. Possible solutions may include collecting a sample even if excessively turbid, or installing a replacement well.

4.4 Development itself disturbs the surrounding formation and disrupts equilibrium conditions within the well. Groundwater samples will not be collected until a minimum of 24 hours after a well is developed to allow conditions to stabilize. For sites with fine-grained formations (silts and clays) and highly sorptive contamination, a longer time period between development and sampling should be considered.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Project Manager** is responsible for ensuring that well development activities comply with this procedure. The **Project Manager** is responsible for ensuring that all personnel involved in well development shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor** is responsible for ensuring that all well development activities are conducted according to the either this procedure or the applicable procedure presented in the project-specific SAP.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the well development procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 This equipment list was developed to aid in field organization and should be used in planning and preparation. Depending on the site-specific requirements and the development method selected, additional or alternative material and equipment may be necessary. In addition, for sites where groundwater is expected to be contaminated, the materials to be placed down the well and in contact with groundwater should be evaluated so that they are compatible with the chemical conditions expected in the well.

6.2 Equipment and materials used for well development may include, but is not limited to:

Well development equipment

- Surge block

- Disposable Teflon bailers, appropriate to the diameter of the well(s): 1-inch to 1.5-inch for 2-inch inside diameter (ID) monitoring wells.
- Watterra® footvalve
- Electric submersible pump
- 12-volt power source for electric pump
- High density polyethylene (HDPE) tubing appropriately sized for Watterra® footvalve and/or electric submersible pump
- Drums or containers for storage of purge water
- Nephelometer to measure turbidity
- Multi-parameter water quality meter(s) to measure temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP)
- Instrument calibration solutions
- Water level meter
- Oil/water interface probe

General equipment

- Project-specific plans including the site-specific HASP and SAP
- Field notebook/field forms/site maps
- Indelible markers/pens
- 5-gallon buckets

Equipment decontamination supplies (refer to SOP 3-06, Equipment Decontamination)

- Health and safety supplies, including personal protective equipment (PPE)
- Appropriate hand tools
- Keys or combinations to access monitoring wells
- Distilled/deionized water supply
- Disposable bailer string (polypropylene)
- Plastic trash bags

7.0 Procedure

Development generally consists of removing water and entrained sediment from the well until the water is clear (to the extent feasible) and the turbidity is reduced, which indicates the well is in good hydraulic connection with the surrounding formation. In addition to simply removing water, development can be improved when flushing through the well screen and gravel pack takes place in both directions, that is, both into the well and into the formation. This action breaks down sediment bridges that can occur in the formation or sand pack, which reduce the connection between the well and the formation

7.1 General Preparation

- All down-well equipment should be decontaminated prior to use and between well locations in accordance with SOP 3-06, Equipment Decontamination
- Although equipment is decontaminated between well locations, if wells are known or suspected to be contaminated based on observations during well installation, it is recommended that well development be conducted in order from the least contaminated to the most contaminated well to minimize the chances of cross-contamination.
- Management of investigation-derived waste (IDW), including development purge water and miscellaneous expendable materials generated during the development process, will be conducted in accordance with SOP 3-05, IDW Management.

- Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. Nothing from the ground surface should be allowed to enter the well.
- The depth to water and total well depth should be measured with a water level meter and recorded in the field logbook or on a Well Development Record (Attachment 1). This information will be used to calculate the volume of standing water (i.e., the well volume) within the well, and plan the specific details of the well development. If wells are suspected to contain NAPL, an oil/water interface probe should be used to measure liquid levels and depth to bottom of the well.
- Permanent monitoring wells will be developed no sooner than 24 hours after well installation is completed in order to allow well completion materials to set properly.

7.2 Monitoring Well Development Procedures

Generally, development will begin by gently surging the well with a surge block or bailer as described in Sections 7.2.1 and 7.2.2, respectively. Surging can become more vigorous as development progresses but initially the well must be gently surged to allow material blocking the screen to become suspended without damaging the well. Next, a bailer can be used to remove the sediment settled at the base of the well. A bailer, Watterra® pump, or electric submersible pump will then be used to purge the well, per Sections 7.2.2, 7.2.3, or 7.2.4, respectively. The well will be purged until the removed water becomes less turbid or per the requirements of the project-specific SAP, or State or local requirements. At this point the well will be surged again with a surge block or bailer. The well can be surged more vigorously at this point. After surging, the well will be purged again until the turbidity once again decreases. The surge/purge cycle should be completed at least three times during the development process. After the last surge, the well will be purged until the development completion criteria outlined in 7.3.2 or per the project-specific SAP are met.

7.2.1 Surge Block

The default method of well development is the use of a surge block in conjunction with pumping or bailing to remove sediment-laden water.

- The construction of the surge block must be appropriate for the diameter of the well. The surge block must be mounted on rods or other stiff materials to extend it to the appropriate depths and to allow for the surge block to be moved up and down in the well.
- Insert the surge block into the well and lower it slowly to the screened or open interval below the static water level. Start the surge action by slowly and gently moving the surge block up and down in the well. A slow initial surging, using plunger strokes of approximately 1 meter or 3 feet, will allow material which is blocking the screen to separate and become suspended.
- After 5 to 10 plunger strokes, remove water from the well using a separate bailer (Section 7.2.2) or pumping techniques (Sections 7.2.3 or 7.2.4). The returned water should be heavily laden with suspended fines. The water will be discharged to 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- In some cases, the bailer or Watterra® foot valve can act as a surge block, flushing water in and out of the well screen as groundwater is removed.
- Repeat the process of surging and pumping/bailing. As development continues, slowly increase the depth of surging to the bottom of the well screen. Surging within the riser portion of the well is neither necessary nor effective.

7.2.2 Bailer

- Tie a string or other cable securely to the bailer. Lower it to the screened or open interval of the monitoring well below the static water level.
- The bailer may be raised and lowered repeatedly within the screened interval to attempt to simulate the action of a surge block by pulling fines through the well screen, and pushing water out into the formation to break down bridging.

- With the bailer full of water, remove it from the well and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The Watterra® system (Section 7.2.3) or electric submersible pump (Section 7.2.4) may be used as a complementary development method to the bailer, especially when removal of additional water at a faster rate is beneficial.
- Continue alternately surging and bailing, monitoring the purge water periodically (Section 7.3.1) until development completion criteria are met (Section 7.3.2).

7.2.3 Watterra® system

- Attach high-density polyethylene (HDPE) tubing to the decontaminated Watterra® pump foot valve
- Lower the foot valve and tubing assembly near the bottom of the well.
- Lift and lower the tubing to allow water to enter the Watterra® foot valve and travel up the tubing and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The lifting and lowering action of the Watterra® sysem will cause some surging action to aid in breaking up fine material in the surrounding formation.
- A bailer (Section 7.2.2) may be used as a complementary development method to the Watterra® system, especially during the initial stages of development when a high volume of sediment may be required to be removed.
- An electric submersible pump (Section 7.2.4) may also be used as a complementary development method to the Watterra® system, especially when more volume of water is desired to be pumped or the turbidity criteria cannot be met due to the surging action of the Watterra® system.
- Continue alternately surging and pumping, monitoring the purge water periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.2.4 Electric Submersible Pump

- Attach HDPE tubing to the decontaminated electric submersible pump.
- Lower the pump and tubing assembly near the bottom of the well, at least a few inches above the well total depth.
- Begin pumping, discharging the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- Continue alternately surging and pumping, monitoring the purge water discharge periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.3 Discharge Monitoring

7.3.1 Monitoring the Progress of Development

The progress of the development is evaluated through visual observation of the suspended sediment load and measurement of the turbidity and other parameters in the purged diischarge water. As development progresses, the water should become clearer, measured turbidity should decrease, and specific capacity (pumping rate divided by drawdown) should stabilize. Water quality parameters, including DO, conductivity, ORP, pH, temperature, and turbidity may be measured and recorded periodically to determine the progress of development using the criteria outlined in Section 7.3.2 or per the project-specific SAP. Water quality parameters should be measured on each well volume removed.

7.3.2 Completion of Development

The well will be considered developed when the following criteria are met or per the criteria set forth in the project-specific SAP:

- A minimum of three times the standing water volume in a well (to include the well screen and casing plus saturated annulus, assuming 30 percent porosity) is removed.

- Groundwater parameters for three consecutive standing water volumes are within the following:
    - pH – within ± 0.2 units
    - Specific conductivity – within ± 3%
    - ORP – within ± 10 mV
    - Temperature – within ±1 degree Celsius
    - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU.
- The sediment thickness remaining within the well is less than 1 percent of the screen length or less than 30 millimeters (0.1 ft) for screens equal to or less than 10 feet long.

Dissolved oxygen (DO) readings may be recorded but DO readings will not be used as development completion criteria because DO may not stabilize.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry three times in succession and the turbidity has decreased, or per the requirements set forth in the project-specific SAP. Water quality parameters may be recorded if feasible using the flow-through cell.

If any water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development, or per the requirements set forth in the project-specific SAP.

7.4 Development of Wells with Low Yield

Water is the primary mechanism to remove fines and flush water through the gravel pack for effective development. Therefore, development can be a challenge in wells that do not yield sufficient water to recharge when water is removed. However, often these wells are the most in need of development to improve their performance as they are typically installed in low permeability formations with a high content of fines. Development of these wells can improve their yield.

The surging portion of the development can be successfully performed in a well with standing water regardless of its yield. It is the subsequent removal of fine materials that is hindered when insufficient water is recharged to the well. When wells go dry or drawdown significantly during development, development can be performed intermittently, allowing sufficient water to recharge prior conducting the next stage of surging. These intermittent procedures can take place hours or even days apart, depending on project-specific time constraints.

7.5 Wells containing NAPL

Additional care should be taken when planning development of wells that contain NAPL. If the NAPL is flammable, there are health and safety as well as handling issues to consider. If NAPL in excess of a persistent sheen is noted, the recharge rate will be evaluated through hand bailing. In most cases, it is generally preferable to remove NAPL by bailing to the extent practical prior to performing development. Groundwater parameters, excluding turbidity, will not be collected during well development if NAPL or excessive sheen is noticed in the purged water during development to ensure the meter probes are not fouled or destroyed. Well development will be halted.

Development by surging or pumping the well dry can result in the spreading of NAPL vertically in the soil column around the well. These methods can be used, if information exists describing the vertical thickness of the NAPL smear zone around the well, and if the methods do not result in mounding or drawdown that exceeds this thickness. Alternate methods such as bailing may also be used, but any method should not allow the well to be pumped dry or result in significant drawdown that would spread the NAPL vertically.

7.6 Temporary Well Points

For certain projects, temporary well points (TWPs) may be installed to collect groundwater samples at a site. Since no sand pack, bentonite chips, or bentonite grout are generally used in the construction of the TWPs, development can proceed as soon as sufficient water has entered the well to static conditions. Due to the small diameter of these wells, generally ¾-inch to 1-inch ID, development will be performed using either a small diameter (0.5-inch) bailer and/or a peristaltic pump with dedicated tubing. The TWPs will have minimal water column and may purge dry during development. However, attempts will be made to remove fines from the well prior to sampling. Purging and sampling may occur as soon as approximately 80% of the static water has re-entered the TWP, or per the requirements set forth in the project-specific SAP.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP.

8.2 Quality control (QC) requirements are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for equipment decontamination (frequency and materials) and IDW handling.

9.0 Records, Data Analysis, Calculations

9.1 All data and information (e.g., development method used) must be documented on field data sheets (Attachment 1) or within site logbooks with permanent ink. Data recorded may include the following:

- Well Location
- Weather conditions
- Date and Time
- Purge Method
- Reading/measurements obtained

10.0 Attachments or References

Attachment 1 – Well Development Record

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change o nly; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Attachment 1
Well Development Record

Well/Piezometer Development Record

Well ID:

Client: _____

Project No: _____ Date: _____ Developer: _____

Site Location: _____

Well/Piezometer Data

Well ☐ Piezometer ☐ Diameter _____ Material _____

Measuring Point Description _____ Geology at Screen Interval (if known) _____

Depth to Top of Screen (ft.) _____

Depth to Bottom of Screen (ft.) _____ Time of Water Level Measurement _____

Total Well Depth (ft.) _____ Calculate Purge Volume (gal.) _____

Depth to Static Water Level (ft.) _____ Disposal Method _____

Headspace _____

Original Well Development ☐ Redevelopment ☐ Date of Original Development _____

DEVELOPMENT METHOD _____

PURGE METHOD _____

Time	Total Volume Purged (gal.)	Flow Rate (gpm)	Turbidity (NTU)	Color	pH	Temp	Other

ACCEPTANCE CRITERIA (from workplan)

Minimum Purge Volume Required _____ gallons
Maximum Turbidity Allowed _____ NTUs
Stabilization of parameters _____%

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Has parameters stabilized	☐	☐	☐

If no or N/A explain below:

_____

_____

_____

Signature _____ Date: _____

Monitoring Well Sampling

Procedure 3-14

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the actions to be used during monitoring well sampling activities and establishes the method for sampling groundwater monitoring wells for water-borne contaminants and general groundwater chemistry. The objective is to obtain groundwater samples that are representative of aquifer conditions with as little alteration to water chemistry as possible.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under contract to the United States Army Corp of Engineers (USACE).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. All field sampling personnel responsible for sampling activities must review the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP), paying particular attention to the control measures planned for the well sampling tasks. Conduct preliminary area monitoring of sampling wells to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and liquid matrix through the use of of appropriate personal protective equipment (PPE).

2.2 Observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves and rubberized steel-toed boots. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations. Refer to the project-specific APP/SSHP for the required PPE.

2.3 Physical Hazards associated with Well Sampling

- To avoid lifting injuries associated with pump and bailers retrieval, use the large muscles of the legs, not the back.
- Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
- When using tools for cutting purposes, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid slip/trip/fall conditions as a result of pump discharge, use textured boots/boot cover bottoms.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- Be aware of restricted mobility due to PPE.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross-contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The **Project Manager** is responsible for ensuring that monitoring well sampling activities comply with this procedure. The **Project Manager** is responsible for ensuring that all field sampling personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the groundwater sampling procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 Purging and Sampling Equipment

- Pump (Peristaltic, Portable Bladder, Submersible)
- Polyethylene or Teflon bladders (for portable bladder pumps)
- Bladder pump controller (for portable bladder pumps)
- Air compressor (for portable bladder pumps)
- Nitrogen cylinders (for portable bladder pumps)
- 12-volt power source
- Polyethylene inlet and discharge tubing (except for VOC analysis which requires Teflon tubing)
- Silicone tubing appropriate for peristaltic pump head
- Teflon bailer appropriately sized for well

- Disposable bailer string (polypropylene)
- Individual or multi-parameter water quality meter(s) with flow-through cell to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity
- Turbidity meter
- Water level meter
- Oil/water interface probe

6.2 General Equipment

- Sample kit (i.e., bottles, labels, preservatives, custody records and tape, cooler, ice)
- Sample Chain-of-Custody (COC) forms
- Sample Collection Records
- Sample packaging and shipping supplies
- Waterproof marker or paint
- Distilled/deionized water supply
- Water dispenser bottles
- Flow measurement cup or bucket
- 5-gallon buckets
- Instrument calibration solutions
- Stopwatch or watch
- Disposable Nitrile gloves
- Paper towels
- Trash bags
- Zipper-lock bags
- Equipment decontamination supplies
- Health and safety supplies (as required by the APP/SSHP)
- Approved plans such as: project-specific APP/SSHP and SAP
- Well keys or combinations
- Monitoring well location map(s)
- Field project logbook/pen

7.0 Calibration or Standardization

7.1 Field instruments will be calibrated daily according to the requirements of the SAP and manufacturer's specifications for each piece of equipment. Equipment will be checked daily with the calibration solutions at the end of use of the equipment. Calibration records shall be recorded in the field logbook or appropriate field form.

7.2 If readings are suspected to be inaccurate, the equipment shall be checked with the calibration solutions and/or re-calibrated.

8.0 Procedure

8.1 Preparation

8.1.1 Site Background Information

Establish a thorough understanding of the purposes of the sampling event prior to field activities. Conduct a review of all available data obtained from the site and pertinent to the water sampling. Review well history data including, but not limited to, well locations, sampling history, purging rates, turbidity problems, previously used purging methods, well installation methods, well completion records, well development methods, previous analytical results, presence of an immiscible phase, historical water levels, and general hydrogeologic conditions.

Previous groundwater development and sampling logs give a good indication of well purging rates and the types of problems that might be encountered during sampling, such as excessive turbidity and low well yield. They may also indicate where dedicated pumps are placed in the water column. To help minimize the potential for cross-contamination, well purging and sampling and water level measurement collection shall proceed from the least contaminated to the most contaminated well as indicated by previous analytical results. This order may be changed in the field if conditions warrant it, particularly if dedicated sampling equipment is used. A review of prior sampling procedures and results may also identify which purging and sampling techniques are appropriate for the parameters to be tested under a given set of field conditions.

8.1.2 Groundwater Analysis Selection

Establish the requisite field and laboratory analyses prior to water sampling. Decide on the types and numbers of quality assurance/quality control (QA/QC) samples to be collected (refer to the project-specific SAP), as well as the type and volume of sample preservatives, the type and number of sample containers, the number of coolers required, and the quantity of ice or other chilling materials. The field sampling personnel shall ensure that the appropriate number and size sample containers are brought to the site, including extras in case of breakage or unexpected field conditions. Refer to the project-specific SAP for the project analytical requirements.

8.2 Groundwater Sampling Procedures

Groundwater sampling procedures at a site shall include:

1) An evaluation of the well security and condition prior to sampling;
2) Decontamination of equipment;
3) Measurement of well depth to groundwater;
4) Assessment of the presence or absence of an immiscible phase;
5) Assessment of purge parameter stabilization;
6) Purging of static water within the well and well bore; and
7) Obtaining a groundwater sample.

Each step is discussed in sequence below. Depending upon specific field conditions, additional steps may be necessary. As a rule, at least 24 hours should separate well development and well sampling events. In all cases, consult the State and local regulations for the site, which may require more stringent time separation between well development and sampling.

8.2.1 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information may be noted on a Groundwater Sample Collection Record (Attachment 1) or in the field logbook:

- Condition of the well's identification marker.
- Condition of the well lock and associated locking cap.
- Integrity of the well – well pad condition, protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing.
- Condition of the general area surrounding the well.

8.2.2 Decontamination of Equipment

Where possible, dedicated supplies should be used at each well location to minimize the potential for cross-contamination and minimize the amount of investigation derived waste (IDW) fluids resulting from the decontamination process. If decontamination is necessary, establish a decontamination station before beginning sampling. The station shall consist of an area of at least 4 feet by 2 feet covered with plastic sheeting and be located upwind of the well being sampled. The station shall be large enough to fit the appropriate number of wash and rinse buckets, and have sufficient room to place equipment after decontamination. One central cleaning area may be used throughout the entire sampling event. The area around the well being sampled shall also be covered with plastic sheeting to prevent spillage. Further details are presented in SOP 3-06, Equipment Decontamination.

Decontaminate each piece of equipment prior to entering the well. Also, conduct decontamination prior to sampling at a site, even if the equipment has been decontaminated subsequent to its last usage. Additionally, decontaminate each piece of equipment used at the site prior to leaving the site. It is only necessary to decontaminate dedicated sampling equipment prior to installation within the well. Do not place clean sampling equipment directly on the ground or other contaminated surfaces prior to insertion into the well. Dedicated sampling equipment that has been certified by the manufacturer as being decontaminated can be placed in the well without on-site decontamination.

8.2.3 Measurement of Static Water Level Elevation

Before purging the well, measure water levels in all of the wells within the zone of influence of the well being purged. The best practice, if possible, is to measure all site wells (or wells within the monitoring well network) prior to sampling. If the well cap is not vented, remove the cap several minutes before measurement to allow water levels to equilibrate to atmospheric pressure.

Measure the depth to standing water and the total depth of the well to the nearest 0.01 foot to provide baseline hydrologic data, to calculate the volume of water in the well, and to provide information on the integrity of the well (e.g., identification of siltation problems). If not already present, mark an easily identified reference point for water level measurements which will become the measuring point for all water level measurements. This location and elevation must be surveyed.

The device used to measure the water level surface and depth of the well shall be sufficiently sensitive and accurate in order to obtain a measurement to the nearest 0.01 foot reliably. An electronic water level meter will usually be appropriate for this measurement; however, when the groundwater within a particular well is highly contaminated, an inexpensive weighted tape measure can be used to determine well depth to prevent adsorption of contaminants onto the meter tape. The presence of light, non-aqueous phase liquids (LNAPLs) and/or dense, non-aqueous phase liquids (DNAPLs) in a well requires measurement of the elevation of the top and the bottom of the product, generally using an interface probe. Water levels in such wells must then be corrected for density effects to accurately determine the elevation of the water table.

At each location, measure water levels several times in quick succession to ensure that the well has equilibrated to atmospheric conditions prior to recording the measurement. As stated above, measure all site wells (or wells within the monitoring well network) prior to sampling whenever possible. This will provide a water level database that describes water levels across the site at one time (a synoptic sampling). Prior to sampling, measure the water level in each well immediately prior to purging the well to ascertain that static conditions have been achieved prior to sampling.

8.2.4 Detection of Immiscible Phase Layers

Complete the following steps for detecting the presence of LNAPL and DNAPL before the well is purged for conventional sampling. These procedures may not be required for all wells. Consult the project-specific SAP to determine if assessing the presence of LNAPL and/or DNAPL is necessary.

1) Sample the headspace in the wellhead immediately after the well is opened for organic vapors using either a PID or an organic vapor analyzer, and record the measurements.

2) Lower an interface probe into the well to determine the existence of any immiscible layer(s), LNAPL and/or DNAPL, and record the measurements.

3) Confirm the presence or absence of an immiscible phase by slowly lowering a clear bailer to the appropriate depth, then visually observing the results after sample recovery.

4) In rare instances, such as when very viscous product is present, it may be necessary to utilize hydrocarbon- and water-sensitive pastes for measurement of LNAPL thickness. This is accomplished by smearing adjacent, thin layers of both hydrocarbon- and water-sensitive pastes along a steel measuring tape and inserting the tape into the well. An engineering tape showing tenths and hundredths of feet is required. Record depth to water, as shown by the mark on the water-sensitive paste, and depth to product, as shown by the mark on the product-sensitive paste. In wells where the approximate depth to water and product thickness are not known, it is best to apply both pastes to the tape over a fairly long interval (5 feet or more). Under these conditions, measurements are obtained by trial and error and may require several insertions and retrievals of the tape before the paste-covered interval of the tape encounters product and water. In wells where approximate depths of air-product and product-water interfaces are known, pastes may be applied over shorter intervals. Water depth measurements should not be used in preparation of water table contour maps until they are corrected for depression by the product.

5) If the well contains an immiscible phase, it may be desirable to sample this phase separately. Section 8.2.6 presents immiscible phase sampling procedures. It may not be meaningful to conduct water sample analysis of water obtained from a well containing LNAPLs or DNAPLs. Consult the **Project Manager** and **Program Quality Manager** if this situation is encountered.

8.2.5 Purging Equipment and Use

General Requirements

The water present in a well prior to sampling may not be representative of in situ groundwater quality and shall be removed prior to sampling. Handle all groundwater removed from potentially contaminated wells in accordance with the IDW handling procedures in SOP 3-05, IDW Management. Purging shall be accomplished by methods as indicated in the project-specific SAP or by those required by State requirements. For the purposes of this SOP, purging methods will be described by removing groundwater from the well using low-flow techniques.

According to the U.S. Environmental Protection Agency (EPA) (EPA, 1996), the rate at which groundwater is removed from the well during purging ideally should be less than 0.2 to 0.3 liters/minute. EPA further states that wells should be purged at rates below those used to develop the well to prevent further development of the well, to prevent damage to the well, and to avoid disturbing accumulated

corrosion or reaction products in the well. EPA also indicates that wells should be purged at or below their recovery rate so that migration of water in the formation above the well screen does not occur.

Realistically, the purge rate should be low enough that substantial drawdown in the well does not occur during purging. In addition, a low purge rate will reduce the possibility of stripping volatile organic compounds (VOCs) from the water, and will reduce the likelihood of increasing the turbidity of the sample due to mobilizing colloids in the subsurface that are immobile under natural flow conditions.

The field sampler shall ensure that purging does not cause formation water to cascade down the sides of the well screen. Wells should not be purged to dryness if recharge causes the formation water to cascade down the sides of the screen, as this will cause an accelerated loss of volatiles. This problem should be anticipated based on the results of either the well development task or historical sampling events. In general, place the intake of the purge pump in the middle of the saturated screened interval within the well to allow purging and at the same time minimize disturbance/overdevelopment of the screened interval in the well. Water shall be purged from the well at a rate that does not cause recharge water to be excessively agitated unless an extremely slow recharging well is encountered where complete evacuation is unavoidable. During the well purging procedure, collect water level and/or product level measurements to assess the hydraulic effects of purging. Sample the well when it recovers sufficiently to provide enough water for the analytical parameters specified. If the well is purged dry, allow the well to recover sufficiently to provide enough water for the specified analytical parameters, and then sample it.

Evaluate water samples on a regular basis during well purging and analyze them in the field preferably using in-line devices (i.e., flow through cell) for temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation-reduction (redox) potential. Turbidity should be measured separately (outside of the flow-through cell) with a nephelometer or similar device.

Readings should be taken every 2 to 5 minutes during the purging process. These parameters are measured to demonstrate that the natural character of the formation waters has been restored.

Purging shall be considered complete per the requirements set forth in the project-specific SAP, State requirements, or when three consecutive field parameter measurements of temperature, pH, specific conductivity, DO and ORP stabilize within approximately 10 percent and the turbidity is at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU. This criterion may not be applicable to temperature if a submersible pump is used during purging due to the heating of the water by the pump motor. Enter all information obtained during the purging and sampling process into a groundwater sampling log. Attachment 1 shows an example of a groundwater sampling log and the information typically included in the form. Whatever form is used, all blanks need to be completed on the field log during field sampling.

Groundwater removed during purging shall be stored according to the project-specific SAP or per SOP 3-05, IDW Management.

Purging Equipment and Methods

Submersible Pump

A stainless steel submersible pump may be utilized for purging both shallow and deep wells prior to sampling the groundwater for semivolatile and non-volatile constituents, but are generally not preferred for VOCs unless there are no other options (e.g., well over 200 feet deep). For wells over 200 feet deep, the submersible pump is one of the few technologies available to feasibly accomplish purging under any yield conditions. For shallow wells with low yields, submersible pumps are generally inappropriate due to overpumpage of the wells (<1 gallon per minute), which causes increased aeration of the water within the well.

Steam clean or otherwise decontaminate the pump and discharge tubing prior to placing the pump in the well. The submersible pump shall be equipped with an anti-backflow check valve to limit the amount of

water that will flow back down the drop pipe into the well. Place the pump in the middle of the saturated screened interval within the well and maintain it in that position during purging.

Bladder Pump

A stainless steel bladder pump can be utilized for purging and sampling wells up to 200 feet in depth for volatile, semivolatile, and non-volatile constituents. Use of the bladder pump is most effective in low to moderate yield wells and are often the preferred method for low-flow sampling. When sampling for VOCs and/or SVOCs, Teflon bladders should be used. Polyethylene bladders may be used when sampling for inorganics.

Either compressed dry nitrogen or compressed dry air, depending upon availability, can operate the bladder pump. The driving gas utilized must be dry to avoid damage to the bladder pump control box. Decontaminate the bladder pump prior to use.

Centrifugal, Peristaltic, or Diaphragm Pump

A centrifugal, peristaltic, or diaphragm pump may be utilized to purge a well if the water level is within 20 feet of ground surface. New or dedicated tubing is inserted into the midpoint of the saturated screened interval of the well. Water should be purged at a rate that satisfies low-flow requirements (i.e., does not cause drawdown). Centrifugal, peristaltic, or diaphragm pump are generally discouraged for VOCs sampling; however, follow methods allowed per the project-specific SAP or State requirements.

Air Lift Pump

Airlift pumps are not appropriate for purging or sampling.

Bailer

Avoid using a bailer to purge a well because it can result in overdevelopment of the well and create excessive purge rates. If a bailer must be used, the bailer should either be dedicated or disposable. Teflon-coated cable mounted on a reel is recommended for lowering the bailer in and out of the well.

Lower the bailer below the water level of the well with as little disturbance of the water as possible to minimize aeration of the water in the well. One way to gauge the depth of water on the reel is to mark the depth to water on the bailer wire with a stainless steel clip. In this manner, less time is spent trying to identify the water level in the well.

8.2.6 Monitoring Well Sampling Methodologies

Sampling Light, Non-Aqueous Phase Liquids (LNAPL)

Collect LNAPL, if present, prior to any purging activities. The sampling device shall generally consist of a dedicated or disposable bailer equipped with a bottom-discharging device. Lower the bailer slowly until contact is made with the surface of the LNAPL, and to a depth less than that of the immiscible fluid/water interface depth as determined by measurement with the interface probe. Allow the bailer to fill with LNAPL and retrieve it.

When sampling LNAPLs, never drop bailers into a well and always remove them from the well in a manner that causes as little agitation of the sample as possible. For example, the bailer should not be removed in a jerky fashion or be allowed to continually bang against the well casing as it is raised. Teflon bailers should always be used when sampling LNAPL. The cable used to raise and lower the bailer shall be composed of an inert material (e.g., stainless steel) or coated with an inert material (e.g., Teflon).

Sampling Dense, Non-Aqueous Phase Liquids (DNAPL)

Collect DNAPL prior to any purging activities. The best method for collecting DNAPL is to use a double-check valve, stainless steel bailer, or a Kemmerer (discrete interval) sampler. The sample shall be collected by slow, controlled lowering of the bailer to the bottom of the well, activation of the closing device, and retrieval.

Groundwater Sampling Methodology

The well shall be sampled when groundwater within it is representative of aquifer conditions per the methods described in Section 8.2.5. Prior to sampling the flow-through cell shall be removed and the samples collected directly from the purge tubing. Flow rates shall not be adjusted once aquifer conditions are met. Additionally, a period of no more than 2 hours shall elapse between purging and sampling to prevent groundwater interaction with the casing and atmosphere. This may not be possible with a slowly recharging well. Measure and record the water level prior to sampling in order to monitor drawdown when using low-flow techniques and gauge well volumes removed and recharged when using non-low-flow techniques.

Sampling equipment (e.g., especially bailers) shall never be dropped into the well, as this could cause aeration of the water upon impact. Additionally, the sampling methodology utilized shall allow for the collection of a groundwater sample in as undisturbed a condition as possible, minimizing the potential for volatilization or aeration. This includes minimizing agitation and aeration during transfer to sample containers, minimizing exposure to sunlight, and immediately placing the sample on ice once collected.

Sampling equipment shall be constructed of inert material. Equipment with neoprene fittings, polyvinyl chloride (PVC) bailers, Tygon® tubing, silicon rubber bladders, neoprene impellers, polyethylene, and Viton® are not acceptable when sampling for organics. If bailers are used, an inert cable/chain (e.g., fluorocarbon resin-coated wire or stainless steel wire or cable) shall be used to raise and lower the bailer. Dedicated equipment is highly recommended for all sampling programs.

Submersible Pumps

The submersible pump must be specifically designed for groundwater sampling (i.e., pump composed of stainless steel and Teflon, sample discharge lines composed of Teflon) and must have a controller mechanism allowing the required low-flow rate. Adjust the pump rate so that flow is continuous and does not pulsate to avoid aeration and agitation within the sample discharge lines. Run the pump for several minutes at the low-flow rate used for sampling to ensure that the groundwater in the lines was obtained at the low-flow rate.

Bladder Pumps

A gas-operated stainless steel bladder pump with adjustable flow control and equipped with a Teflon bladder and Teflon-lined tubing can be effectively utilized to collect a groundwater sample and is considered to be the best overall device for sampling inorganic and organic constituents. If only inorganics are being sampled, polyvinyl bladders and tubing may be used. Operate positive gas displacement bladder pumps in a continuous manner so that they minimize discharge pulsation that can aerate samples in the return tube or upon discharge.

When using a compressor, take several precautions. If the compressor is being powered by a gasoline generator, position the generator downwind of the well. Ground fault circuit interrupters (GFCIs) should always be used when using electric powered equipment. Do not connect the compression hose from the compressor to the pump controller until after the engine has been started.

When all precautions are completed and the compressor has been started, connect the compression hose to the pump controller. Slowly adjust the control knobs to discharge water in the shortest amount of time while maintaining a near constant flow. This does not mean that the compressor must be set to discharge the water as hard as possible. The optimal setting is one that produces the largest volume of purge water per minute (not per purge cycle) while maintaining a near constant flow rate.

Prior to sampling, adjust the flow rate (purge rate) to yield 100 to 300 mL/minute. Avoid settings that produce pulsating streams of water instead of a steady stream if possible. Operate the pump at this low flow rate for several minutes to ensure that drawdown is not occurring. At no time shall the sample flow rate exceed the flow rate used while purging.

For those samples requiring filtration, it is recommended to use an in-line high capacity filter after all non-filtered samples have been collected.

***Peristaltic Pumps*:**

A peristaltic pump is a type of positive displacement pump that moves water via the process of peristalsis. The pump uses a flexible hose fitted inside a circular pump casing. A rotor with cams compresses the flexible tube as the rotor turns, which forces the water to be pumped to move through the tube. In peristaltic pumps, no moving parts of the pump are in contact with the water being pumped. Displacement is determined by tube size, so delivery rate can only be changed during operation by varying pump speed. Peristaltic pumps are simple and quite inexpensive for the flow rates they provide.

There are several methods available for transferring the sample into the laboratory containers. The selected method may vary based on State requirements and should be documented in the project-specific SAP. Samples typically can be collected directly from the discharge end of the Teflon tubing, after it has been disconnected from the flow through cell. For volatile analyses, the sampler should make sure that the pump is set such that a smooth laminar flow is achieved. In all cases, the project team should consult their local regulatory requirements and document the selected sample collection procedure in the project-specific SAP.

Bailers

A single- or double-check valve Teflon or stainless steel bailer equipped with a bottom discharging device can be utilized to collect groundwater samples. Bailers have a number of disadvantages, however, including a tendency to alter the chemistry of groundwater samples due to degassing, volatilization, and aeration; the possibility of creating high groundwater entrance velocities; differences in operator techniques resulting in variable samples; and difficulty in determining where in the water column the sample was collected. Therefore, use bailers for groundwater sampling only when other types of sampling devices cannot be utilized for technical, regulatory, or logistical reasons.

Dedicated or disposable bailers should always be used in order to eliminate the need for decontamination and to limit the potential of cross-contamination. Each time the bailer is lowered to the water table, lower it in such a way as to minimize disturbance and aeration of the water column within the well.

8.2.7 Sample Handling and Preservation

Many of the chemical constituents and physiochemical parameters to be measured or evaluated during groundwater monitoring programs are chemically unstable and require preservation. The U.S. EPA document entitled, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods (SW-846)* (EPA 1997), includes a discussion of appropriate sample preservation procedures. In addition, SW-846 provides guidance on the types of sample containers to use for each constituent or common set of parameters. In general, check with specific laboratory or State requirements prior to obtaining field samples. In many cases, the laboratory will supply the necessary sample bottles and required preservatives. In some cases, the field sampling personnel may add preservatives in the field.

Improper sample handling may alter the analytical results of the sample. Therefore, transfer samples in the field from the sampling equipment directly into the container that has been prepared specifically for that analysis or set of compatible parameters as described in the project-specific SAP. It is not an acceptable practice for samples to be composited in a common container in the field and then split in the laboratory, or poured first into a wide mouth container and then transferred into smaller containers.

Collect groundwater samples and place them in their proper containers in the order of decreasing volatility and increasing stability. A preferred collection order for some common groundwater parameters is:

1. VOCs and total organic halogens (TOX)

2. Dissolved gases, total organic carbon (TOC), total fuel hydrocarbons
3. Semivolatile organics, pesticides
4. Total metals, general minerals (unfiltered)
5. Dissolved metals, general minerals (filtered)
6. Phenols
7. Cyanide
8. Sulfate and chloride
9. Nitrate and ammonia
10. Radionuclides

When sampling for VOCs, collect water samples in vials or containers specifically designed to prevent loss of VOCs from the sample. The analytical laboratory performing the analysis shall provide these vials. Collect groundwater from the sampling device in vials by allowing the groundwater to slowly flow along the sides of the vial. Sampling equipment shall not touch the interior of the vial. Fill the vial above the top of the vial to form a positive meniscus with no overflow. No headspace shall be present in the sample container once the container has been capped. This can be checked by inverting the bottle once the sample is collected and tapping the side of the vial to dislodge air bubbles. Sometimes it is not possible to collect a sample without air bubbles, particularly water that has high concentrations of dissolved gasses. In these cases, the field sampling personnel shall document the occurrence in the field logbook and/or sampling worksheet at the time the sample was collected. Likewise, the analytical laboratory shall note in the laboratory analysis reports any headspace in the sample container(s) at the time of receipt by the laboratory.

Special Handling Considerations

In general, samples for organic analyses should not be filtered. However, high turbidity samples for PCB analysis may require filtering. Consult the project-specific SAP for details on filtering requirements. Samples shall not be transferred from one container to another because this could cause aeration or a loss of organic material onto the walls of the container. TOX and TOC samples should be handled in the same manner as VOC samples.

When collecting total and dissolved metals samples, the samples should be collected sequentially. The total metals sample is collected from the pump unfiltered. The dissolved metals sample is collected after filtering with a 0.45-micron membrane in-line filter. Allow at least 500 mL of effluent to flow through the filter prior to sampling to ensure that the filter is thoroughly wetted and seated in the filter capsule. If required by the project-specific SAP, include a filter blank for each lot of filters used and always record the lot number of the filters.

Field Sampling Preservation

Preserve samples immediately upon collection. Ideally, sampling containers will be pre-preserved with a known concentration and volume of preservative. Certain matrices that have alkaline pH (greater than 7) may require more preservative than is typically required. An early assessment of preservation techniques, such as the use of pH strips after initial preservation, may therefore be appropriate. Guidance for the preservation of environmental samples can be found in the U.S. EPA *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA 1982). Additional guidance can be found in other U.S. EPA documents (EPA 1992, 1996).

Field Sampling Log

A groundwater sampling log provided as Attachment 1 shall document the following:

- Identification of well

- Well depth
- Static water level depth and measurement technique
- Presence of immiscible layers and detection method
- Well yield
- Purge volume and pumping rate
- Time that the well was purged
- Sample identification numbers
- Well evacuation procedure/equipment
- Sample withdrawal procedure/equipment
- Date and time of collection
- Types of sample containers used
- Preservative(s) used
- Parameters requested for analysis
- Field analysis data
- Field observations on sampling event
- Name of sampler
- Weather conditions

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

10.0 Data and records management

10.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Sample Collection Records;
- Field logbook;
- Chain-of-custody forms; and
- Shipping labels.

10.2 Sample collection records (Attachment 1) will provide descriptive information for the purging process and the samples collected at each monitoring well.

10.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.

10.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

10.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

11.0 Attachments or References

Attachment 1 – Groundwater Sampling Collection Record

ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

Environmental Protection Agency, United States (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater.* EPA-600/4-82-029. Cincinnati: EPA Office of Research and Development, Environmental Monitoring and Support Laboratory.

EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

EPA. 1996. *Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. Office of Solid Waste and Emergency Response. April.

EPA. 1997. *Test Methods for Evaluating Solid Waste, Physical/Chemical Method (SW-846)*. 3rd ed., Final Update IIIA. Office of Solid Waste. Online updates at: http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

SOP 3-03, *Recordkeeping, Sample Labelling, and Chain-of-Custody*.

SOP 3-05, *IDW Management.*

SOP 3-06, *Equipment Decontamination*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Attachment 1
Groundwater Sample Collection Record

Well ID:

Groundwater Sample Collection Record

Client: _____ Date: _____ Time: Start _____ am/pm
Project No: _____ Finish _____ am/pm
Site Location: _____
Weather Conds: _____ Collector(s): _____

1. **WATER LEVEL DATA: (measured from Top of Casing)**
   a. Total Well Length _____ c. Length of Water Column _____ (a-b)
   b. Water Table Depth _____ d. Calculated Well Volume (see back) _____
   Casing Diameter/Material _____

2. **WELL PURGEABLE DATA**
   a. Purge Method: _____
   b. Acceptance Criteria defined (see SAP or Work Plan)
   - Minimum Required Purge Volume (@ _____ well volumes) _____
   - Maximum Allowable Turbidity _____ NTUs
   - Stabilization of parameters _____ %
   c. Field Testing Equipment used: Make Model Serial Number

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

(continued on back)

   d. Acceptance criteria pass/fail

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

   If no or N/A - Explain below.
   _____

3. **SAMPLE COLLECTION:** Method: _____

Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

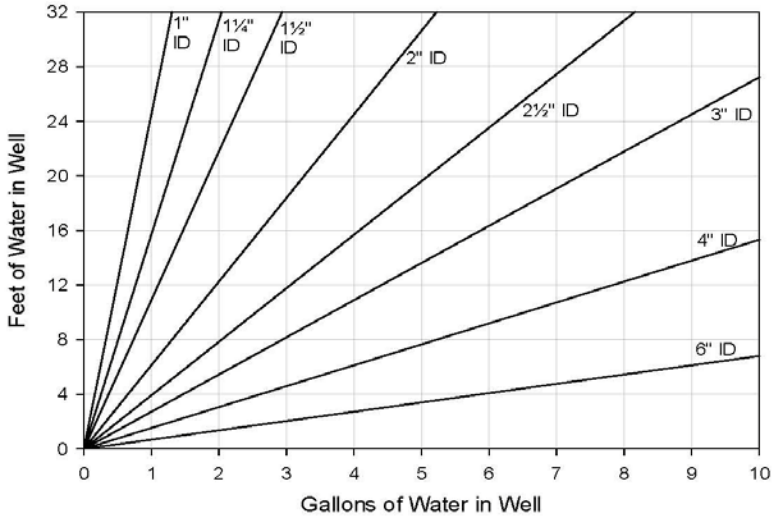
Comments _____

Signature _____ Date _____

Page 1 of 2

Purge Volume Computation

Well ID:



Volume / Linear Ft. of Pipe		
ID (in)	**Gallon**	**Liter**
¼	0.0025	0.0097
3/8	0.0057	0.0217
½	0.0102	0.0386
¾	0.0229	0.0869
1	0.0408	0.1544
1¼	0.0637	0.2413
1½	0.0918	0.3475
2	0.1632	0.6178
2½	0.2550	0.9653
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600

(continued from front)

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

Signature _____ Date _____

Monitoring Well and Borehole Abandonment

Procedure 3-15

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the methods used for the abandonment of groundwater monitoring wells, piezometers, and direct-push boreholes.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the United States Army Corp of Engineers (USACE).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). In the absence of a APP/SSHP, work will be conducted according to the Work Plan (WP) and/or direction from the Site Safety Health Officer (SSHO).

2.2 Physical hazards associated with well installation include:

- To avoid lifting injuries associated with well abandonment practices, use the large muscles of the legs, not the back. The drilling contractor should use the drill rig wenching cables and appropriate heavy equipment to minimize manual lifting.
- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- When using an approved retractable-blade knife, cut away from one self.
- To avoid slip/trip/fall conditions during site activities, keep the area clear of excess soil cuttings and formation groundwater and use textured boots/boot cover bottoms in muddy areas.
- To avoid heat/cold stress because of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 - 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- Be aware of restricted mobility caused by PPE.

3.0 Terms and Definitions

3.1 **Annulus:** The annulus is the down-hole space between the borehole wall and the well casing and screen.

3.2 **Bridge:** A bridge is an obstruction in the drill hole or annulus. A bridge is usually formed by caving of the wall of the well bore, by the intrusion of a large boulder, or by the placement of filter pack materials during well completion. Bridging can also occur in the formation during well development.

3.3 **Filter Pack:** Filter pack is sand or gravel that is smooth, uniform, clean, well-rounded, and siliceous. It is placed in the annulus of the well between the borehole wall and the well screen to prevent formation materials from entering the well and to stabilize the adjacent formation.

3.4 **Grout:** Grout is a fluid mixture of cement and water that can be forced through a tremie pipe and emplaced in the annular space between the borehole and casing to form an impermeable seal. Various

additives, such as sand, bentonite, and polymers, may be included in the mixture to meet certain requirements.

4.0 Interferences

4.1 The total depth of the monitoring well will be measured and the measurement will be compared to the original well completion log prior to abandonment.

4.2 A map with the location of the well to be abandoned and the surrounding wells, if any, will be utilized in the field to confirm the location of the well to be abandoned.

4.3 Information from the well identification tags/markings will be noted and the information compared to both the well completion log and the total depth measurement obtained in the field to confirm the identity of the well being abandoned.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Project Manager** is responsible for ensuring that well abandonment activities comply with this procedure. The **Project Manager** is responsible for ensuring that all personnel involved in well abandonment shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor** is responsible for ensuring that all well abandonment activities are conducted according to the either this procedure or the applicable procedure presented in the project-specific SAP.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the well abandonment procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 Equipment and materials used during monitoring well and piezometer abandonment include the following:

- Drill rig or trailer-mounted mixer and grout pump
- Filter pack material
- Pure sodium bentonite with no additives
- Bentonite pellets/chips
- Bentonite grout
- Portland Type II cement
- Water from an approved source
- Weighted tape measure

- Flexible hose
- Tremie pipe (small-diameter, rigid polyvinyl chloride [PVC] pipe)
- Weatherproof bound field logbook with numbered pages
- Appropriate health and safety equipment

7.0 Procedure

7.1 General Procedures

The following procedure applies to the abandonment of wells aborted prior to completion, existing wells determined to be ineffective or otherwise in need of closure, temporary wells, or boreholes (with the exception that boreholes will not have casing and pipe in place). Prior to abandoning any developed well, you may need to acquire a permit from the State or local governing body in which you are working. The permit application may require a detailed design of the well abandonment. In addition, prior to abandonment, all obstructions (e.g., pumps, lost equipment) must be removed from the well. Some States are strict in requiring the removal of all lost equipment prior to abandonment and will not allow the closure of a well with lost equipment in it. The State may require the removal of all objects to allow a proper seal during abandonment. Great lengths must be taken to reclaim lost items, such as the use of downhole video cameras to inspect and aid in the recovery of items. Prior to abandonment, confirm that the well selected for abandonment is properly located and identified to avoid abandoning the wrong well.

At locations where a well log is not available, the following procedure shall be implemented:

- The casing should be pulled, drilled out, or thoroughly pierced.
- With the use of a tremie pipe, grout should be placed from the bottom of the hole to within 3 feet of the ground surface.
- The material should be allowed to settle for 24 hours.
- The remainder of the hole should be filled with concrete.
- All historical sample data and abandonment procedures should be included in the records of work.

At locations where a well completion log is available, the following procedure shall be implemented:

- With the use of a tremie pipe, grout should be placed from the bottom of the hole to within 3 feet of the ground surface.
- The material should be allowed to settle for 24 hours.
- The remainder of the hole should be filled with concrete.
- All boring logs, historical sample data, completion records, and abandonment procedures should be included in the records of work.

Depending on the regulatory body under which you are working, the procedures listed above may differ. All work shall be performed by a licensed well driller in the State work is being performed. The licensed well driller is responsible for documenting the abandonment of the monitoring well with the appropriate State agency.

7.2 Replacement Wells

Replacement wells (if any) should normally be offset at least 15 feet from any abandoned well in an upgradient or crossgradient groundwater flow direction. Site-specific conditions may necessitate variation of this placement requiring the replacement well to be located either closer or further in proximity to the original well. To avoid potential issues related to grout migration into a well filter pack and/or screen section, replacement wells should be installed after the original/adjacent well is properly abandoned.

7.3 Grout

Bentonite grout is preferred for the abandonment of monitoring wells. Cement grout, if used for abandonment, should be composed of the following by weight:

- 20 parts cement (Portland cement, Type II or V)
- 0.4 to 1 part (maximum) (2 to 5 percent) bentonite
- 8 gallons (maximum) approved water per 94-pound bag of cement

Neither additives nor borehole cuttings should be mixed with the grout. Bentonite should be added after the required amount of cement has been mixed with the water. All grout material should be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout should be recirculated through the grout pump prior to placement. The mixture can be combined and recirculated through a drill rig equipped for mud rotary drilling or through a mixer and grout pump mounted on a trailer.

Grout should be placed with the use of a commercially available grout pump and a rigid tremie pipe. Casing and grouting should be removed in stages, aquifer by aquifer, sealing the boring from the bottom to ground surface. This should be accomplished by placing a tremie pipe to the bottom and pumping grout through the pipe until undiluted grout reaches the bottom of the next higher section of casing or, for the topmost section, until grout flows from the boring at the ground surface.

After 24 hours, the abandoned drilling site should be checked for grout settlement. Any settlement depression should be filled with grout and rechecked 24 hours later. This process should be repeated until firm grout remains at the ground surface.

Be aware that when the drillers are finished, they will need a large supply of water to rinse out their equipment. This wash water must be containerized as IDW in accordance with SOP 3-05, *IDW Management*. Also, any materials (such as the removed protective casing, manhole covers, and concrete collars) shall be disposed of properly, or per the requirements of the project-specific SAP.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP.

8.2 Quality Control (QC) measures should be taken to ensure proper well abandonment in accordance with this SOP, project-specific SAP, and applicable well standards.

9.0 Records, Data Analysis, Calculations

9.1 All field information must be documented in the field logbook and/or on field data sheets with permanent ink. Data recorded may include the following:

- Date/time
- Well/piezometer location
- Personnel/subcontractor on site
- Abandonment method
- Depth of well/piezometer
- Materials used to seal each stratum
- Detailed description of procedure
- Date/time of return visit(s)
- Activities performed on return visit(s)
- Observations or problems encountered during abandonment

10.0 Attachments or References

Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. Office of Solid Waste and Emergency Response. EPA/540/P-87/001.

SOP 3-05, *IDW Management*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Soil and Rock Classification

Procedure 3-16

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) to thoroughly describe the physical characteristics of the sample and classify it according to the Unified Soil Classification System (USCS).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under United States Army Corp of Engineers (USACE) contracts.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling. All **field sampling personnel** responsible for sampling activities must review the project-specific Accident Prevention Plan (APP), with a Site-Safety and Health Plan (SSHP) attachment, paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP/SSHP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSHO.

2.4 The health and safety considerations for the work associated with soil classification include:

- At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.
- Stay clear of all moving equipment and be aware of pinch points on machinery. Avoid wearing loose fitting clothing.
- When using cutting tools, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid heat/cold stress as a results of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear insulating clothing.

3.0 Terms and Definitions

None.

4.0 Interference

None.

5.0 Training and Qualifications

5.1 The **Project Manager** is responsible for ensuring that the soil and rock classification procedures comply with this procedure. The **Project Manager** is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.3 The **Site Supervisor** is responsible for ensuring that all project **field personnel** follow these procedures.

5.4 Field personnel are responsible for the implementation of this procedure. Minimum qualifications for **field sampling personnel** require that one individual on the field team shall have a minimum of 6 months of experience with soil and rock classification.

5.5 The **project geologist** and/or **task manager** is responsible for directly supervising the soil and rock classification procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the **Program Quality Manager** and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the APP/SSHP
- Field log book and pen with indelible ink
- Boring log

- Munsell Soil Color Chart
- Scoopula, spatula, and/or other small hand tools
- California Sampler
- Hand-held penetrometer

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Soil Classification

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The USCS was developed so that soils could be described on a common basis by different investigators and serve as a "shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log, logbook, and/or electronic field data collection device. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand)
- Color, moisture, and odor
- Range of particle sizes and maximum particle size
- Approximate percentage of boulders, cobbles, gravel, sand, and fines
- Plasticity characteristics of the fines
- In-place conditions, such as consistency, density, and structure
- USCS classification symbol

The USCS serves as ”shorthand“ for classifying soil into 15 basic groups:

GW[1] Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)

GP[1] Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)

GM[1] Silty gravel (>50 percent gravel, >15 percent silt)

GC[1] Clayey gravel (>50 percent gravel, >15 percent clay)

SW[1] Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)

SP[1] Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines)

[1] If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

SM[1] Silty sand (>50 percent sand, >15 percent silt)

SC[1] Clayey sand (>50 percent sand, >15 percent clay)

ML[2] Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)

CL[2] Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)

MH[2] Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)

CH[2] Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity)

OL Organic low plasticity silt or organic silty clay

OH Organic high plasticity clay or silt

PT Peat and other highly organic soil

Figure 8-1 defines the terminology of the USCS. Flow charts presented in Figure 8-2 and Figure 8-3 indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.

8.1.1 Estimation of Particle Size Distribution

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.
3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term “trace”.
7. The percentages of gravel, sand, and fines must add up to 100 percent. “Trace” is not included in the 100 percent total.

8.1.2 Soil Dilatancy, Toughness, and Plasticity

8.1.2.1 Dilatancy

To evaluate dilatancy, follow these procedures:

[2] If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words “with sand” or “with gravel” (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words “sandy” or “gravely” (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use “sandy.”

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against 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 note the reaction as none, slow, or rapid in accordance with the criteria in Table 8-1. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

Table 8-1: Criteria for Describing Dilatancy

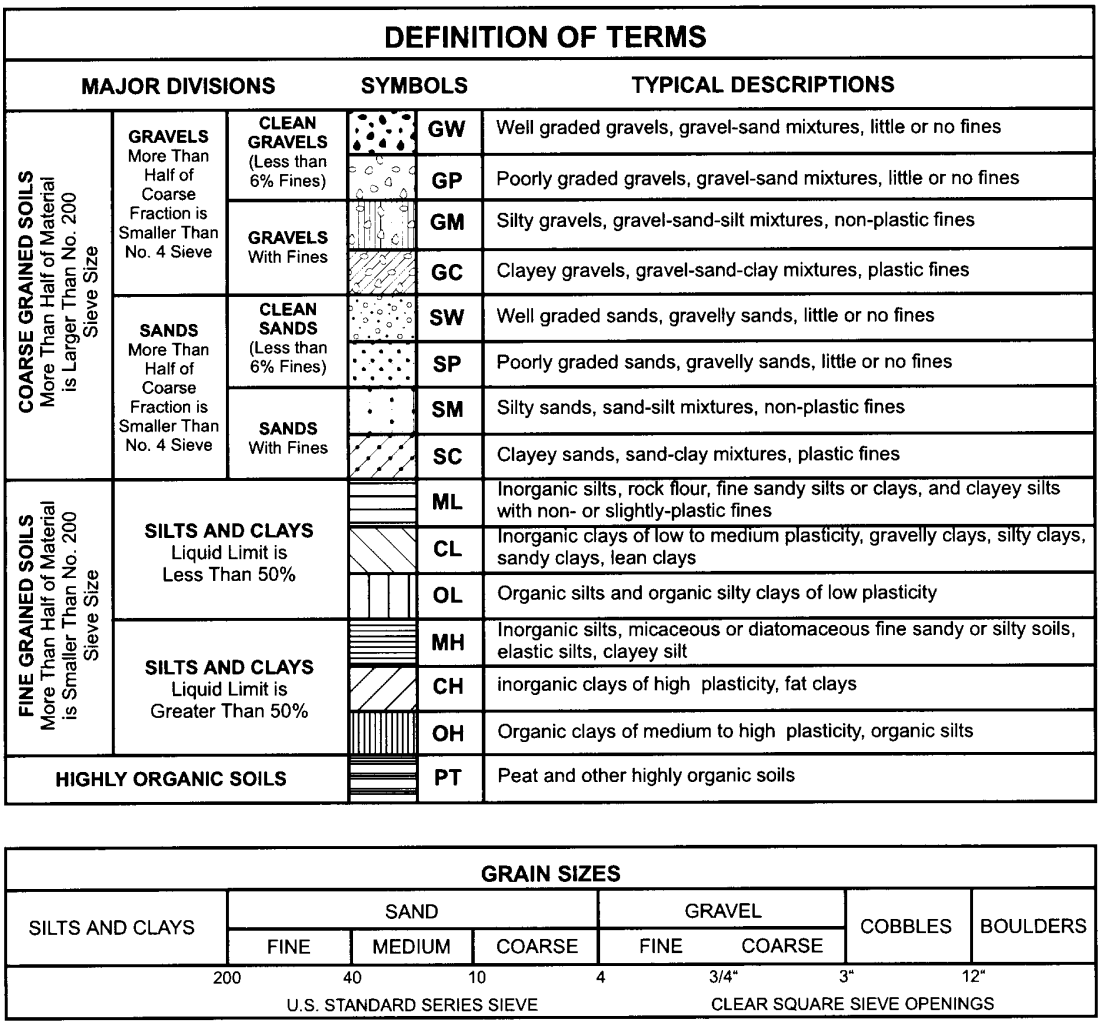
Description	Criteria
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

8.1.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it 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, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 8-2.

Table 8-2: Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.



DEFINITION OF TERMS					
MAJOR DIVISIONS			SYMBOLS		TYPICAL DESCRIPTIONS
COARSE GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	GRAVELS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN GRAVELS (Less than 6% Fines)		GW	Well graded gravels, gravel-sand mixtures, little or no fines
				GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
		GRAVELS With Fines		GM	Silty gravels, gravel-sand-silt mixtures, non-plastic fines
				GC	Clayey gravels, gravel-sand-clay mixtures, plastic fines
	SANDS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN SANDS (Less than 6% Fines)		SW	Well graded sands, gravelly sands, little or no fines
				SP	Poorly graded sands, gravelly sands, little or no fines
		SANDS With Fines		SM	Silty sands, sand-silt mixtures, non-plastic fines
				SC	Clayey sands, sand-clay mixtures, plastic fines
FINE GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	SILTS AND CLAYS Liquid Limit is Less Than 50%			ML	Inorganic silts, rock flour, fine sandy silts or clays, and clayey silts with non- or slightly-plastic fines
				CL	Inorganic clays of low to medium plasticity, gravelly clays, silty clays, sandy clays, lean clays
				OL	Organic silts and organic silty clays of low plasticity
	SILTS AND CLAYS Liquid Limit is Greater Than 50%			MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts, clayey silt
				CH	inorganic clays of high plasticity, fat clays
				OH	Organic clays of medium to high plasticity, organic silts
HIGHLY ORGANIC SOILS				PT	Peat and other highly organic soils

GRAIN SIZES								
SILTS AND CLAYS	SAND			GRAVEL		COBBLES	BOULDERS	
	FINE	MEDIUM	COARSE	FINE	COARSE			
200	40	10	4	3/4"	3"	12"		
U.S. STANDARD SERIES SIEVE				CLEAR SQUARE SIEVE OPENINGS				

Figure8-1: Unclassified Soil Classification System (USCS)

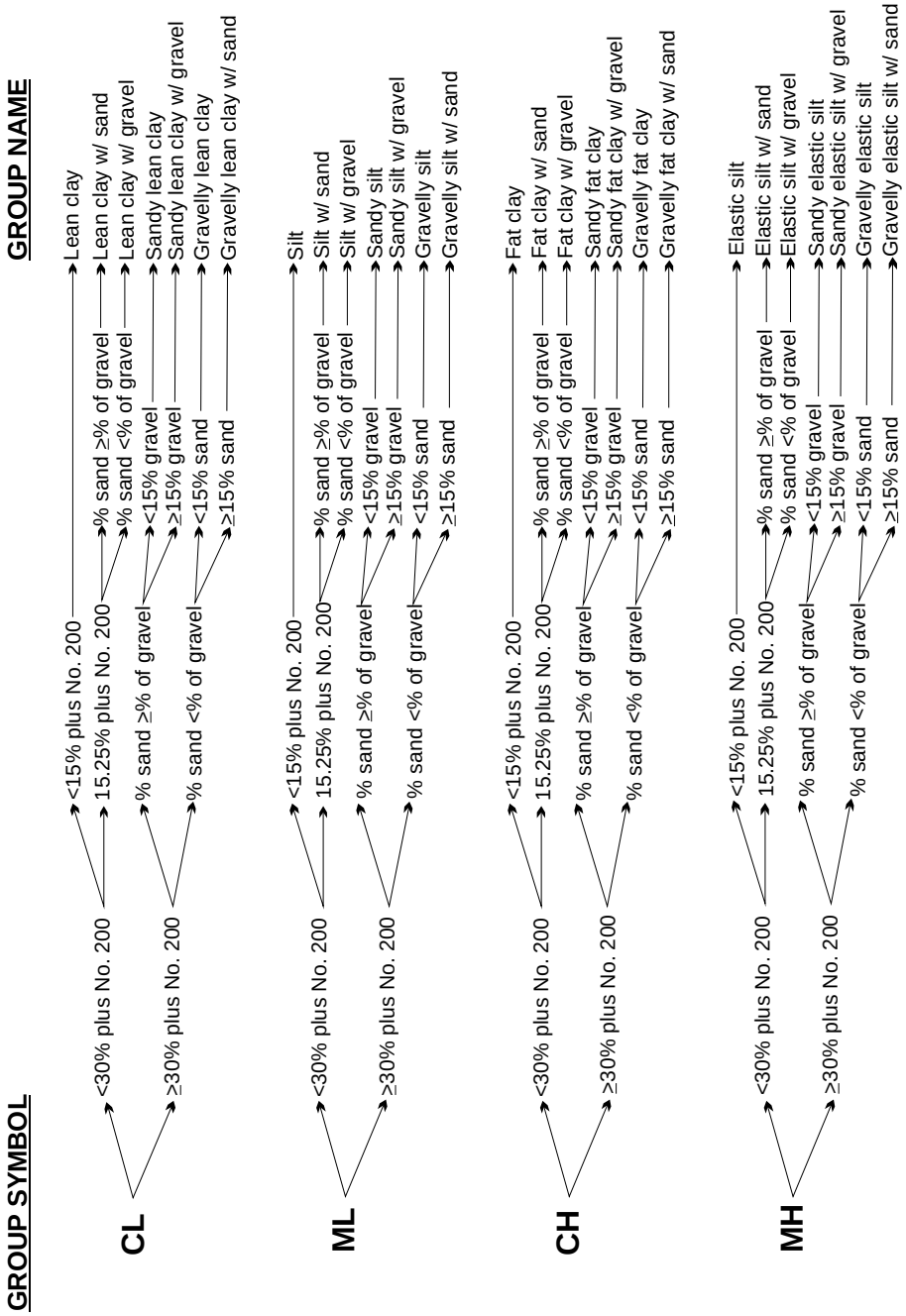


Figure 8-2: Flow Chart for Fine Grain Soil Classification

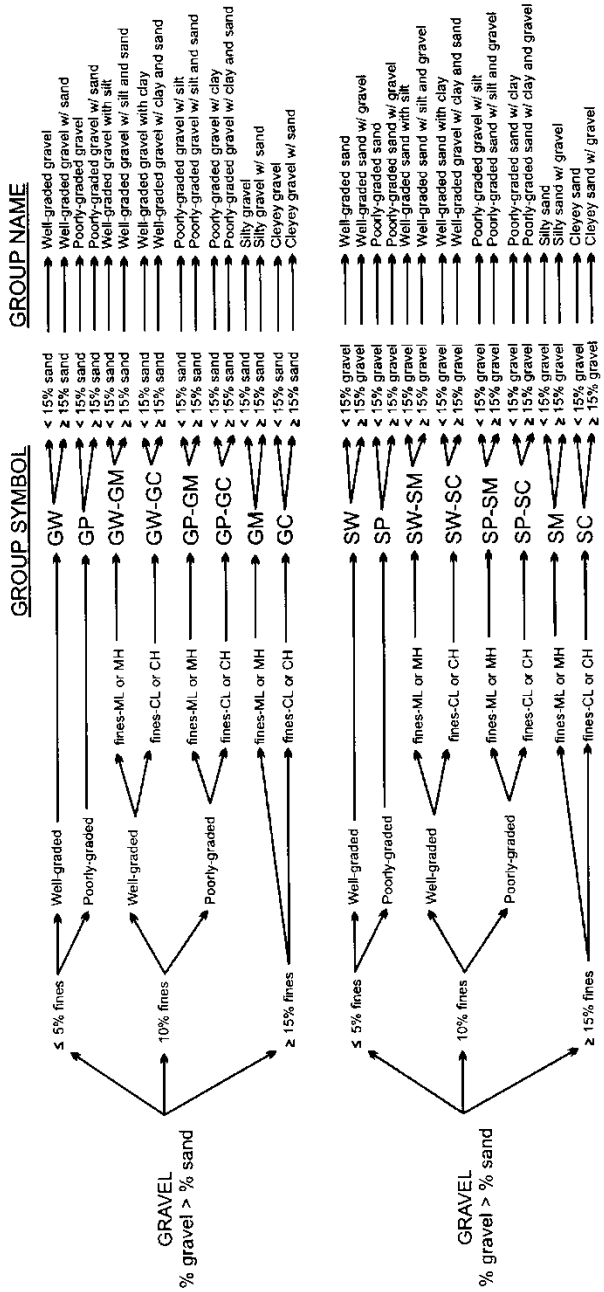


Figure 8-3: Flow Chart for Soil with Gravel

8.1.2.3 Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 8-3 presents the criteria for describing plasticity in the field using the rolled thread method.

Table 8-3: Criteria for Describing Plasticity

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

8.1.3 **Angularity**

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides, but have well-rounded corners and edges.
- **Subangular** particles are similar to angular, but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

8.1.4 **Color, Moisture, and Odor**

The natural moisture content of soil is very important. Table 8-4 shows the terms for describing the moisture condition and the criteria for each.

Table 8-4: Soil Moisture Content Qualifiers

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table

Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like “5YR 5/6, yellowish-red.” Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

8.1.5 In-Place Conditions

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

8.1.5.1 Density/Consistency

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term “density” is commonly applied to coarse to medium-grained sediments (i.e., gravels, sands), whereas the term “consistency” is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the “standard penetration resistance,” or the “N-value.” The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless steel liners and penetrating a cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed “refusal” and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 8-5 and Table 8-6 present representative descriptions of soil density/consistency vs. N-values.

Table 8-5: Measuring Soil Density with a California Sampler – Relative Density (Sands, Gravels)

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43
Dense	30–50	43–71
Very Dense	> 50	> 71

Table 8-6: Measuring Soil Density with a California Sampler – Fine Grained Cohesive Soil

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 8-7):

Table 8-7: Measuring Soil Consistency with a Hand-Held Penetrometer

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8-8.

Table 8-8: Measuring Soil Consistency Using Thumb Pressure

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

8.1.5.2 Cementation

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

- Quartz – siliceous
- Chert – chert-cemented or chalcedonic
- Opal – opaline
- Carbonate – calcitic, dolomitic, sideritic (if in doubt, calcareous should be used)
- Iron oxides – hematitic, limonitic (if in doubt, ferruginous should be used)
- Clay minerals – if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as “kaolin-cemented,” “chlorite-cemented,” etc.
- Miscellaneous minerals – pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

- Weak – friable; crumbles or breaks with handling or slight finger pressure
- Moderate – friable; crumbles or breaks with considerable finger pressure
- Strong – not friable; will not crumble or break with finger pressure

8.1.5.3 Structure

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular – spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified – alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness
- Laminated – alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky – cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed – inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar – particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy – particles are arranged about a horizontal plane

8.1.5.4 Other Features

- Mottled – soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured – breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)
- Slickensided – fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

8.1.6 **Development of Soil Description**

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

8.1.6.1 Coarse-grained Soil

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is "sand-sized." It is classified as a gravel if over 50 percent of the coarse fraction is composed of "gravel-sized" particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

8.1.6.2 Fine-grained Soil

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.

Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

8.1.6.3 Organic Soil

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

8.2 Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

8.3 Rock Classification

The purpose of rock classification is to thoroughly describe the physical and mineralogical characteristics of a specimen and to classify it according to an established system. The generalized rock classification system described below was developed because, unlike the USCS for soils, there is no universally accepted rock classification system. In some instances, a more detailed and thorough rock classification system may be appropriate. Any modifications to this classification system, or the use of an alternate classification system should be considered during preparation of the site work plan. Both the CTO Manager and the QA Manager or Technical Director must approve any modifications to this classification system, or the use of another classification system.

Describing rock specimens on a common basis is essential so that rocks described by different site geologists are comparable. Site geologists describing rock specimens as a part of investigative activities must use the classification system described herein, or if necessary, another more detailed classification system. Use of a common classification system provides the most useful geologic database for all present and future subsurface investigations and remedial activities.

In order to provide a more consistent rock classification between geologists, a rock classification template has been designated as shown in Figure 8-4. The template includes classification of rocks by origin and mineralogical composition. When classifying rocks, all site geologists shall use this template.

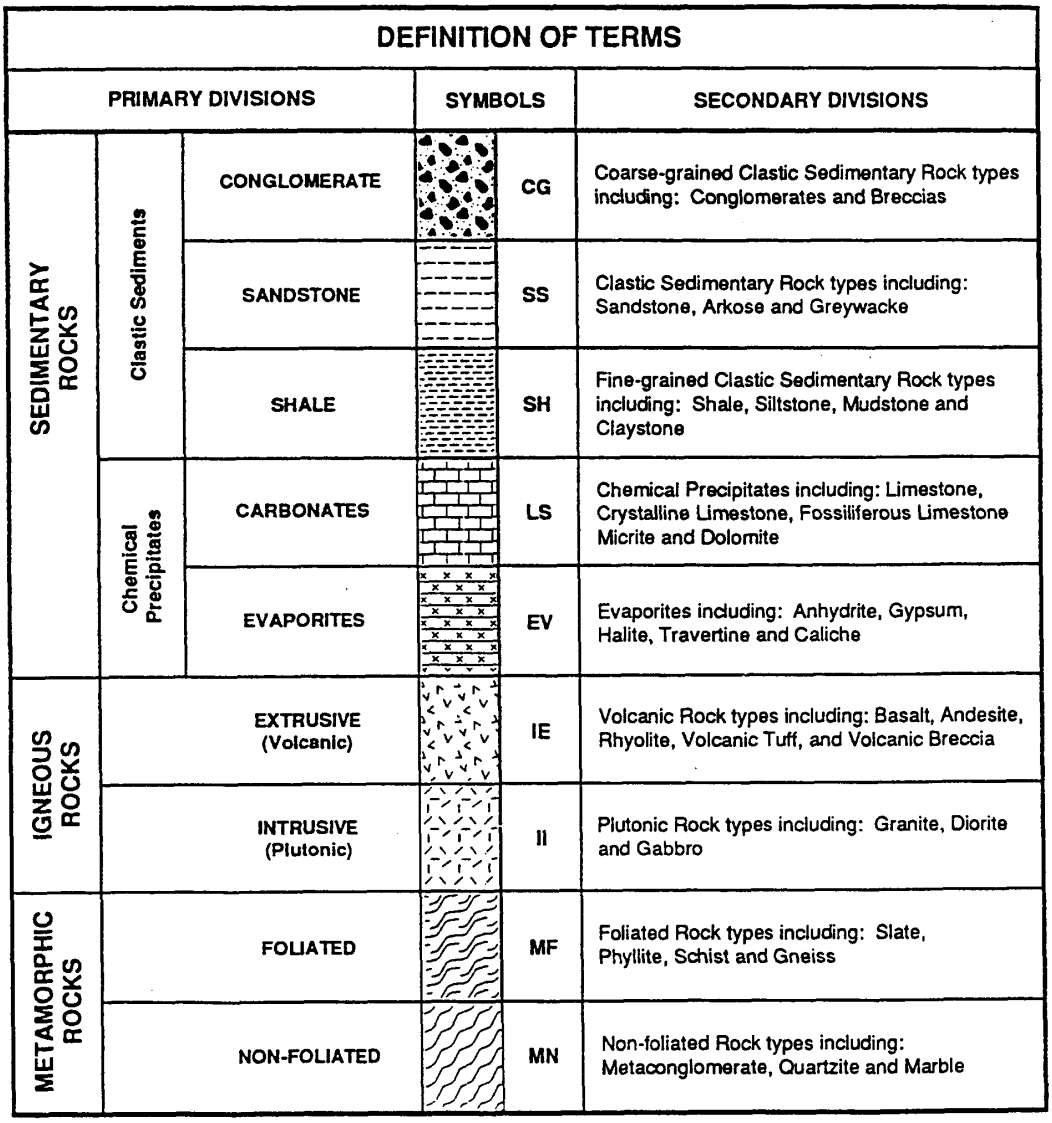
The site geologist shall describe the rock specimen and record the description in a boring log or logbook. The items essential for classification include (i.e., metamorphic foliated):

- Classification Name (i.e., schist)
- Color
- Mineralogical composition and percent
- Texture/Grain size (i.e., fine-grained, pegmatitic, aphlitic, glassy)
- Structure (i.e., foliated, fractured, lenticular)
- Rock Quality Designation (sum of all core pieces greater than two times the diameter of the core divided by the total length of the core run, expressed as a percentage)
- Classification symbol (i.e., MF)

Example: Metamorphic foliated schist: Olive gray, 5Y, 3/2, Garnet 25 percent, Quartz 45 percent, Chlorite 15 percent, Tourmaline 15 percent, Fine-grained with Pegmatite garnet, highly foliated, slightly wavy, MF.

9.0 Quality Control and Assurance

None



DEFINITION OF TERMS					
PRIMARY DIVISIONS			SYMBOLS		SECONDARY DIVISIONS
SEDIMENTARY ROCKS	Clastic Sediments	CONGLOMERATE		CG	Coarse-grained Clastic Sedimentary Rock types including: Conglomerates and Breccias
		SANDSTONE		SS	Clastic Sedimentary Rock types including: Sandstone, Arkose and Greywacke
		SHALE		SH	Fine-grained Clastic Sedimentary Rock types including: Shale, Siltstone, Mudstone and Claystone
	Chemical Precipitates	CARBONATES		LS	Chemical Precipitates including: Limestone, Crystalline Limestone, Fossiliferous Limestone Micrite and Dolomite
		EVAPORITES		EV	Evaporites including: Anhydrite, Gypsum, Halite, Travertine and Caliche
IGNEOUS ROCKS		EXTRUSIVE (Volcanic)		IE	Volcanic Rock types including: Basalt, Andesite, Rhyolite, Volcanic Tuff, and Volcanic Breccia
		INTRUSIVE (Plutonic)		II	Plutonic Rock types including: Granite, Diorite and Gabbro
METAMORPHIC ROCKS		FOLIATED		MF	Foliated Rock types including: Slate, Phyllite, Schist and Gneiss
		NON-FOLIATED		MN	Non-foliated Rock types including: Metaconglomerate, Quartzite and Marble

Figure 8-4: Rock Classification System

10.0 Data and Records Management

10.1 Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be sent to the **CTO Manager** for the project files.

10.2 Field notes will be kept during coring activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. The information pertinent to soil classification activities includes chronology of events, sample locations (x,y,z), time/date, sampler name, methods (including type of core liner/barrel, if applicable), sampler penetration and acceptability, sample observations, and the times and type of equipment decontamination. Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

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Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

AECOM

Boring Log

Project Name: Camp Hero Remedial Investigation	Site:	**Hole ID:**
Project Number: 60443903.4.1	*Northing:*	Total Depth (feet):
Drilling Contractor: New England Geotech	*Easting:*	Date / Time Started:
Driller:	Elevation (feet MSL): *Ground:*	Date / Time Finished:
Drilling Equipment:	▼ Water Depth During Drilling (feet bgs):	Date / Time Completed:
Drilling Method:	Logged By:	Checked By:
Borehole Diameter (inches):	Weather/Comments:	

Depth (feet)	USCS Description	Log: Graphic	Log: USCS or Rock Type	Samples: Attempted Recovered	Samples: Method	Samples: Run Number	Samples: PID/FID (ppm)	Samples: Time	Well Diagram	Remarks (list sample numbers here)
5										**Soil Sample Info** SB S#: SD: ST: Analyses: SB S#: SD: ST: Analyses:
10										**Groundwater Sample Info** Well-head PID (ppm): DTW (ft bgs): GW S#: Screen interval (ft bgs): ST: Analyses: Temp (C): pH: SC (mS/cm): ORP (mV): DO (mg/L): Turbidity (NTU):
15										

Tracking Codes: 10/19/12, 12:21

USCS Name, **Consistency/Density** (predominantly fine: very soft {n=0-1}, soft {n=2-4}, medium stiff {n=5-8}, stiff {n=9-15}, very stiff {n=16-30}, hard {n=31+}/predominently coarse: very loose {n=0-4}, loose {n=5-10}, medium dense {n=11-30}, dense {n=31-50}, very dense {n=51+}). **Moisture**, (dry, moist, wet). **Color**. **Gradation** (relative percentages of soil components). **Plasticity/Cohesiveness** (predominently fine: nonplastic, slightly plastic, low plasticity, medium plasticity, high plasticity)/predominently coarse: cohesionless, slightly cohesive, cohesive). **Stratification/Structure** (blocky, massive, lensed, etc) (contacts: sharp, gradational) (bedding: horizontal, inclined). **Cementation** (none, weak, moderate, strong). **Other descriptive elements; Geologic Origin**
S# = Sample Number, **SD** = Sample Depth, **ST** = Sample Time, **A** = Analysis.
BZ = Breathing Zone, **BG** = Background, **BH** = Borehole, **CB** = Cuttings Bin

AECOM

Boring Log (Continued)

Project Name: Camp Hero Remedial Investigation	Site:	Hole ID:

Depth (feet)	USCS Description	Log: Graphic	Log: USCS or Rock Type	Samples: Attempted Recovered	Samples: Method	Samples: Run Number	Samples: PID/FID (ppm)	Samples: Time	Well Diagram	Remarks (list sample numbers here)
20										
25										
30										

Tracking Codes: 10/19/12, 12:20

Form: 00-BLANK LOG

Direct Push Sampling Techniques

Procedure 3-17

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) provides guidance on the use of direct push techniques for the United States Army Corp of Engineers (USACE).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM for USACE.

1.3 This procedure shall serve as management-approved professional guidance for the Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Project Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

1.4 If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to direct push sampling then those procedures may be added as an appendix to the project-specific SAP.

2.0 Safety

2.1 Field personnel shall perform work in accordance with the site-specific Accident Prevention Plan (APP) and Site-Safety and Health Plan (SSHP). During monitoring well installation, subcontractors in direct contact with potentially contaminated media shall wear the proper personal protective equipment (PPE) as outlined in the site-specific health and safety plan. Failure to comply will result in disciplinary action.

2.2 If circumstances warrant, a real-time immediate response instrument, such as a Miniram Dust Monitor, organic vapor analyzer, HNu, Thermo, Draeger or Sensidyne tubes, or explosimeter, should be used to monitor the work area. When real/time instrument response exceeds the permissible exposure limit, personnel shall don the appropriate PPE and alternate control measures to ensure personnel safety. If safe control measures are not achievable, field activities shall be discontinued immediately. Company-specific APP/SSHPs offer guidelines on air surveillance and on selection of PPE. In addition, the site-specific APP/SSHP includes an air monitoring program and suggested PPE.

2.3 In addition to the aforementioned precautions and depending upon the type of contaminant expected, employ the following safe work practices:

Particulate or Metal Compounds

1. Avoid skin contact and/or incidental ingestion of soil.
2. Wear protective clothing, steel-toed boots, gloves, safety glasses, and hearing protection as warranted.

VOCs

1. Avoid breathing constituents venting from holes by approaching upwind, and/or by use of respiratory protection.
2. Pre-survey the area with a flame ionization detector (FID) or photoionization detector (PID) prior to sampling.

3. If monitoring results indicate organic vapors that exceed action levels as specified in the site-specific APP/SSHP, sampling activities may need to be conducted in Level C protection. At a minimum, skin protection will be required by use of gloves and Tyvek or other media that is protective against the media being encountered.

Flammable or Explosive Conditions

1. Monitor explosive gases as continuously as possible using an explosimeter and oxygen meter.
2. Place all ignition sources upwind or crosswind of the borehole.
3. If explosive gases exceed the designated action levels as specified in the site-specific APP/SSHP, cease operations and evaluate conditions.

Physical Hazards Associated With Soil Sampling

1. To avoid possible back strain associated with sample collection, use the large muscles of the legs, not the back, when retrieving soil samplers.
2. Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
3. To avoid slip/trip/fall hazards, be wary of open trenches, pits, or holes.
4. Be aware of restricted mobility due to PPE.
5. To avoid hand, wrist, arm, shoulder, and back trauma due to the use of slide hammers or hand augers, rotate sampling among field personnel

3.0 Terms and Definitions

3.1 Direct push techniques are methods for subsurface sampling or monitoring that involve the application of downward pressure (usually supplied through hydraulic means) without the benefit of cutting tool rotation to enter soil. A variety of systems are available under several trade names, such as GeoProbe®. Equipment may be skid-mounted, trailered, or mounted directly on the frame of a vehicle.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Project Manager** is responsible for ensuring that these standard direct push technique procedures are followed during projects conducted under the Program and that a qualified individual conducts or supervises the projects. A qualified individual for subsurface sampling or monitoring using direct push techniques is defined as a person with a degree in geology, hydrogeology, or geotechnical/civil engineering with at least 1 year of experience supervising soil boring construction using conventional drilling or direct push techniques. The Project Manager or designee is responsible for ensuring that all personnel involved in direct push

sampling techniques shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1c (DON 2007).

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Site Supervisor is responsible for ensuring that all field personnel follow these procedures.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

5.2.5 The Field Personnel and/or Field Manager is responsible for directly supervising the direct push sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

In addition to those materials provided by the subcontractor, the project **Field Manager/Field Personnel** will require:

- Boring Logs;
- Spoons or scoops;
- Sample kit (bottles, labels, custody records and tape, cooler, ice), if laboratory analysis is required;
- Sample collection pan;
- Folding rule or tape measure;
- Plastic sheeting;
- Utility knife;
- Equipment decontamination materials (as described in SOP 3-06, *Equipment Decontamination*);
- Health and safety equipment (as required by APP/SSHP); and
- Field project notebook/pen.

7.0 Procedure

Direct push techniques may be used as a cost-effective alternative to conventional drilling techniques for obtaining subsurface soil and groundwater samples and for monitoring subsurface conditions.

7.1 Method Selection

Base the decision to use direct push techniques on: (1) their ability to achieve the required information at the required level of quality control and (2) their cost-effectiveness compared to conventional drilling methods. Major limitations of direct push techniques are their inability to penetrate rock or cobbles and a shallow maximum depth of penetration. The capabilities of direct push systems vary significantly among vendors. Consider these differences in capabilities when evaluating the method for a subsurface exploration program.

Use direct push techniques to obtain groundwater samples for confirmatory analyses only if the screen placement method protects the screen from clogging during installation and allows the installation of a sand-pack around the exterior of the well screen.

7.2 Inspection of Equipment

Inspect direct push equipment prior to use for signs of fluid leakage, which could introduce contaminants to the soil. If, at any time during equipment operation, fluid is observed leaking from the rig, cease operations and immediately repair or contain the leak. Collect, containerize, and label soil and other materials affected by the leak for proper disposal (see SOP 3-05, *IDW Management*).

7.3 Preparation of Work Site

Inspect the work site prior to commencing operations to ensure that no overhead hazards exist that could impact the direct push equipment, and the work area should cleared and/or marked by the local underground utility locating service (e.g., DigSafe). In addition, clear locations planned for subsurface exploration using either geophysical methods and/or hand excavate locations to a depth of 2 to 3 feet prior to soil penetration, unless it is certain (by virtue of subsurface clearing activities) that no utilities or other hazardous obstructions will be encountered in the first 2 to 3 feet. Hand excavation may be waived when it is not practical.

Locate the direct push rig so that it is downslope from the penetration point, if the work is to be performed on a grade. Locate the rig downwind or crosswind of the penetration point, if possible. Cover the area surrounding, and in the vicinity of, the penetration point with plastic. Establish required exclusion zones using plastic tape or cones to designate the various areas.

7.4 Equipment Decontamination

To avoid cross-contamination, thoroughly decontaminate equipment used for direct push exploration and sampling as described in SOP 3-06, *Equipment Decontamination*. Decontaminate sampling tools and downhole equipment between each sampling event and between penetration points. At a minimum, steam clean or wash and rinse the equipment. Collect, containerize, and label all wash and rinse water for proper disposal. Clean equipment (e.g., drive rods and samplers) shall not come into contact with contaminated soils or other contaminated materials. Keep equipment on plastic or protect it in another suitable fashion. Store push rods and other equipment removed from a hole on plastic sheeting until properly decontaminated.

7.5 Soil Sampling

This SOP assumes that the subcontractor will perform sampling; therefore, detailed procedures regarding sample acquisition are not provided. Vendors of direct push equipment offer a variety of sampling systems designed specifically for their equipment. Both continuous and discreet soil samples may be obtained using sampling equipment similar to that described in Procedure 3-21, *Surface and Subsurface Soil Sampling*. The preferred methods for soil sampling using direct push techniques use brass or stainless steel split-tube samplers that are driven through the horizon to be sampled. Use plastic sample tubes (e.g., Macro-Core Samplers) only for screening purposes or, in the case of confirmatory sampling, if samples will not be analyzed for volatile organic compounds (VOCs) or semivolatile organic compounds (SVOCs).

7.6 Groundwater Sampling

Direct push vendors offer numerous methods for obtaining groundwater samples. Key differences among methods involve: (1) the maximum well diameter achievable; (2) the ability to protect the well screen from exposure to contaminated overburden soils during installation; (3) the ability to install packing around the screen; (4) flexibility in the size, materials of construction, and design of well screens; and (5) the ability to convert sampling points into permanent monitoring wells. The limitations and abilities of a given system must be thoroughly understood and matched to the needs of the project before committing to the collection of groundwater samples using direct push techniques.

Use direct push techniques only to collect screening samples unless it is confirmed that the system:

1. Effectively protects the well screen from exposure to contaminated overburden soils during installation
2. Allows the installation of effective packing around the well screen
3. Allows the well screen to be effectively sealed against the downward infiltration of overlying groundwater or surface precipitation
4. Is constructed of materials compatible with the intended sampling and analysis goals of the project

5. Allows the use of a well screen properly sized and slotted for the needs of the project

Additional information on the collection of groundwater samples can be found in SOP 3-14 Monitoring Well Sampling.

It is the responsibility of the **Project Manager** to evaluate and determine the appropriateness of direct push systems prior to committing to their use on any project involving groundwater sampling. As part of this evaluation, it is recommended to obtain concurrence from regulatory authorities in advance for the method selection.

7.7 Borehole Abandonment

Methods for abandoning boreholes created with direct push systems will vary among vendors. Coordinate the desired method for abandonment with the vendor in the planning stages of the project to ensure proper abandonment.

Some direct push boreholes will close naturally as the drive rods and sampling tools are withdrawn. This may occur in loose, unconsolidated soils, such as sands. Close all boreholes using one of the procedures described in this procedure, unless natural caving precludes such closure.

The three methods for closing direct push boreholes are:

1. Add granulated or pelletized bentonite and hydrate in layers, proceeding from the bottom of the hole to the surface.
2. Pour premixed cement/water (or cement/water/bentonite) mixture into the hole.
3. Fill the entire hole with granular or pelletized bentonite and hydrate by means of a previously emplaced water tube that is gradually withdrawn as water is supplied to the bentonite.

The second method is recommended. For shallow holes less than 10 feet in depth, pour a cement/water/bentonite mix directly into the opening using a funnel. For deeper holes, use a conductor (tremie) pipe to carry the grout mix to the far reaches of the borehole. Lower the conductor pipe to within 2 inches of the bottom and gradually withdraw it as grout is added, keeping the lower end of the pipe submerged in grout at all times.

The recommended grout mixture for well abandonment is 7 to 9 gallons of water per 94-pound bag of Portland cement, with 3 percent to 5 percent by weight of powdered bentonite added to the mixture. Commercial products, such as Volcay are acceptable with pre-approval of the **Project Manager**.

Seal boreholes to within 0.5 to 2.0 feet of the surface. Inspect the abandoned borehole after 24 hours to ensure that grout shrinkage does not occur. If significant shrinkage has occurred, re-grout the borehole. Fill the remaining portion of the hole with local topsoil or appropriate paving materials.

8.0 Quality Control and Assurance

8.1 Collection of representative samples will be ensured through adherence to the procedures in this SOP and the sampling strategy outlined in the SAP. The field quality control samples identified in the SAP must be collected. These samples may include field duplicates, equipment rinsate blanks, trip blanks, and matrix spike/matrix spike duplicates

9.0 Records, Data Analysis, Calculations

9.1 Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Boring logs;
- Field logbook;

- Sample collection records;
- Chain-of-custody forms; and
- Shipping labels.

9.2 Boring logs (Attachment 1) will provide visual and descriptive information for samples collected at each soil boring and are often the most critical form of documentation generated during a soil sampling program.

9.3 The field logbook is kept as a general log of activities and should not be used in place of the boring log.

9.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

9.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

10.0 Attachments or References

10.1 Attachment 1 – Boring Log

10.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

10.3 SOP 3-05, *IDW Management.*

10.4 SOP 3-06, *Equipment Decontamination.*

10.5 SOP 3-21, *Surface and Subsurface Soil Sampling.*

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (APril 2017)

Attachment 1
Boring Log

Boring ID:

Page 1 of _____

Project Name:	Drilling Company:	Type of Surface Material:
Project Number:	Drilling Method:	Patching Material:
Date Started Drilling:	Rig Type:	Drilling Water Level:
Date Finished Drilling:	Core Size:	Boring Total Depth (bgs):
Physical Location:		Logged By:

(Note: bgs = below ground surface)

Depth Range	Recovery ft/ft	PID (ppm)	Moisture Content	GA Class.	USCS	GA Class: Garfield Avenue Sites classification & Modified Unified Soil Clasiffication System	
						Ground Surface Cover and Thickness:	Sample name & #:
0-1							
1-2							
2-3							
3-4							
4-5							
5-6							
6-7							
7-8							
8-9							
9-10							
10-11							
11-12							
12-13							
13-14							
14-15							
15-16							
16-17							
17-18							
18-19							
19-20							

Stratigraphic Unit Intervals:		Comments:
1.)	5.)	
2.)	6.)	
5.)	6.)	

Field Analysis of Ferrous Iron Using the HACH DR890 Colorimeter and HACH Method 8146

Procedure 3-18

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) is applicable for the field analysis of water, wastewater, and seawater for ferrous iron (Fe^{2+}). Concentrations ranging up to 3.00 mg/L can be analyzed, with higher concentrations analyzed by diluting the samples. The estimated detection limit is 0.03 mg/L. The presence of ferrous iron is indicative of a reducing state, therefore measurement of ferrous iron is useful in determining whether site conditions are reducing.

1.2 The 1,10-phenanthroline indicator in the AccuVac ampul reacts with ferrous iron (Fe^{+2}) in the sample to form an orange color in proportion to the iron concentration. Ferric iron (Fe+3) does not react. The ferric ion concentration can be determined by subtracting the ferrous iron concentration from results of a total iron test.

1.3 Samples are analyzed immediately after collection because ferrous iron readily oxidizes to ferric iron upon exposure to air. A minimum volume of approximately 100 mL of sample is required to complete this analysis. A HACH "AccuVac" ampul (a vacuum-sealed glass ampul containing 1,10-phenanthroline reagent) is broken, tip down in the sample, and allowed to react for the required three minutes. The colorimeter is zeroed and the sample analyzed. The instrument must be zeroed with a sample water blank prior to each analysis.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP). In the absence of an APP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the Site Safety Officer (SSO).

2.2 Caution should be taken when working with all chemicals. Refer to the Material Safety Data Sheets (MSDSs) for the chemicals to be used.

2.3 Caution should be taken by the analyst with the sharp, broken glass that results after activating the AccuVac ampul.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Ferrous iron (Fe^{2+}) oxidizes into ferric iron (Fe^{3+}) rapidly on exposure to air and addition of oxidants. Samples must be analyzed immediately after sample collection.

4.2 Color development due to the reaction of ferrous iron with 1,10-phenanthroline is time dependent. Therefore, it is critical that the reaction time allowed between addition of the reagent from the AccuVac vial and color measurement be consistent for all analyses and carefully timed.

4.3 Care should be taken to avoid cross-contamination between field samples. The analyst should change gloves each time before handling the field sample for analysis. Sample color and turbidity will interfere

with colorimetric measurement. The colorimeter is therefore blank corrected with sample water prior to sample analysis and measurement.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The CTO Manager is responsible for ensuring that field analysis activities comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in the field analysis shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all field analysis is conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

6.1 Equipment

- HACH DR/890 colorimeter;
- 3 HACH "AccuVac ampuls" (to allow for diluted analyses if required) per well;
- HACH colorimeter sample cell (for the blank) 10-20-25 mL, with cap;
- Plastic cups and lids;
- Graduated cylinders;
- Kimwipes®;
- Pipetter; and
- Deionized ultra-filtered water (DIUF) for dilutions.

7.0 Procedure

7.1 Sample Analysis Procedure

Approximately 100 mL of sample will be collected in a plastic cup. Some sample is poured into the HACH 10-20-25 mL sample cell as a blank, and the instrument is zeroed with the blank. An AccuVac ampul is broken into the remainder of the sample in the cup, and set to react for 3 minutes. The sample is then analyzed. The procedures are outlined below.

7.1.1 Turn on the colorimeter by pressing EXIT. Press PRGM. When prompted, enter 33 and press ENTER.

7.1.2 Check historical ferrous iron concentrations for the sample location if available, and determine if sample dilution is necessary: If the anticipated concentration is above 3.00 mg/L, the upper end of the operating range of the colorimeter, a dilution is necessary. Prepare an appropriate dilution with DIUF using the graduated cylinder and pipette as needed. Do not use the volume indicators on the sample cell as these are not sufficiently accurate. Place the required volume of sample into the graduated cylinder, and fill to the desired volume with DIUF to the total volume. Swirl the cylinder by grasping the top with three fingers, and swinging in a circle three times. Record the volumes of sample and DIUF used for the dilution.

7.1.3 Pour at least 10 mL of the sample or diluted sample water into the 10-20-25 mL sample cell (not an AccuVac ampul). This will be used for the blank to zero the colorimeter.

7.1.4 Clean the sample cell with a damp Kimwipe, followed by a dry Kimwipe.

7.1.5 Place the 10-20-25 mL sample cell in the colorimeter, using the same orientation each time.

7.1.6 Cover, and press ZERO.

7.1.7 Pour at least 10mL of the unused sample or diluted sample into a plastic cup. Immerse an inverted AccuVac ampul in the sample water and snap off the tip on the bottom of the cup. Hold the ampul in place until water is no longer entering the ampul (about 4 seconds).

7.1.8 Remove the ampul from the cup, and quickly invert several times to mix. It is not necessary to block the top of the ampul as the liquid will not leak out.

7.1.9 Press TIMER, and the press ENTER on the colorimeter. A three minute reaction period will begin. During this reaction period, wipe off the AccuVac ampul with a Kimwipe.

7.1.10 When the 3-minute timer is up (colorimeter will beep), put the ampul in the colorimeter, cover tightly with the colorimeter cap, press READ, and record the reading.

7.1.11 Record the colorimeter reading on the sample worksheet.

7.1.12 If the reading is >3.00, collect more sample, dilute the sample, and repeat steps 4 through 12. If the sample was diluted and the reading is <0.03, collect more sample, prepare at a lower dilution, and reanalyze.

8.0 Quality Control and Assurance

8.1 Precision, Accuracy, and Contamination

8.1.1 Precision and sample variability is assessed through analysis of 1 field duplicate sample per day, or per 20 field samples, whichever is more frequent. Precision of 30% RPD is considered acceptable for field duplicate analyses. Note that the rapid oxidation of ferrous iron precludes replicate analysis of a sample.

8.1.2 Potential contamination is assessed through the analysis of blanks. The colorimeter must be zeroed with a sample water blank prior to analysis of each sample. For diluted sample analyses, the blank should consist of the diluted sample water at the same dilution as the sample.

8.2 Sample Collection, Preservation, and Storage

8.2.1 Sample preservation and storage is not possible since ferrous iron oxidizes rapidly. Samples should be analyzed immediately after collection.

8.2.2 Use a clean, unused plastic jar to transfer sample.

8.3 Method Performance

The HACH DR/890 colorimeter has an estimated detection limit of 0.03 mg/l Fe^{2+} and an analytical standard deviation (single operator) of 0.009 mg/l Fe^{2+}.

8.4 Pollution Prevention

All dilutions will be carefully recorded and tabulated for use in future site analyses to minimize the number of dilutions and analyses required.

8.5 Waste Management

8.5.1 Unused sample must be disposed of as per the sampling and analysis plan, quality assurance project plan, and/or work plan.

8.5.2 The reacted AccuVac ampul should be placed in the AccuVac Vial Destruct Unit (AVDU) (a 1-L HDPE sample bottle with a large rock), and shaken to break the ampul. Replace the lid of the AVDU with the lid that has holes punctured in the top, and drain all remaining liquid from the AVDU into a container with the sample water for drumming, disposal, etc. Dispose of the glass-filled AVDU as unregulated solid waste.

8.5.3 Liquids drained from the test kits should be diluted five-fold with tap water for discharge to drain, or diluted with purge water and drummed with the purge water for disposal according to the sampling and analysis plan and/or work plan.

9.0 Records, Data Analysis, Calculations

9.1 Results should be reviewed prior to leaving the field to be sure field duplicates were within acceptance range and results did not exceed the instrument's range (3.00 mg/l).

9.2 The dilution factor is calculated as follows:

9.3 DF = (Volume of DIUF water + Volume of sample water) ÷ Volume of sample water

9.4 Concentration of ferrous iron in sample water = reading on colorimeter x DF

9.5 Example: If 10 mL of sample are diluted with 90 mL of DIUF water and the colorimeter reading was 2.3 mg/L, then:

9.6 Ferrous Iron Concentration (mg/L) = 2.3 mg/L x 10 = 23 mg/L

9.7 Unanticipated changes to the procedures or materials described in this POP (deviations) will be appropriately documented in the project records.

9.8 Records associated with the activities described in this POP will be maintained according to the document management policy for the project.

10.0 Attachments or References

10.1 United States Environmental Protection Agency. 2001. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. EPA/240/B-01/004. USEPA Office of Environmental Information, Washington, DC. March 2001.

10.2 *Hach DR/890 Colorimeter Procedures Manual*, Edition 7, HACH Company, December 2005.

10.3 *Hach DR/820, DR/850, DR890 Portable Datalogging Colorimeter Instrument Manual*, Revision 5, HACH Company, 1997 1999.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 – Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Operation and Calibration of a Photoionization Detector

Procedure 3-20

1.0 Purpose and Scope

1.1 Purpose and Applicability

1.1.1 This standard operating procedure (SOP) describes the procedures that will be followed by field staff for operation and calibration of a photoionization detector (PID). The PID is primarily used by AECOM personnel for safety and survey monitoring of ambient air, determining the presence of volatiles in soil and water, and detecting leakage of volatiles.

1.1.2 PIDs routinely used by field personnel include the Photovac Microtip, Thermoelectron 580EZ, and MiniRAE 2000. Personnel responsible for using the PID should first read and thoroughly familiarize themselves with the instrument instruction manual.

1.2 Principle of Operation

1.2.1 The PID is a non-specific vapor/gas detector. The unit generally consists of a hand-held probe that houses a PID, consisting of an ultraviolet (UV) lamp, two electrodes, and a small fan which pulls ambient air into the probe inlet tube. The probe is connected to a readout/control box that consists of electronic control circuits, a readout display, and the system battery. Units are available with UV lamps having an energy from 9.5 electron volts (eV) to 11.7 eV.

1.2.2 The PID analyzer measures the concentration of trace gas present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule (in electron volts (eV)) is less than the energy of the photon. The source of photons is an ultraviolet lamp in the probe unit. Lamps are available with energies ranging from 9.5 eV to 11.7 eV. All organic and inorganic vapor/gas compounds having ionization potentials lower than the energy output of the UV lamp are ionized and the resulting potentiometric change is seen as a positive reading on the unit. The reading is proportional to the concentration of organics and/or inorganics in the vapor.

1.2.3 Sample gases enter the probe through the inlet tube and enter the ion chamber where they are exposed to the photons emanating from the UV lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive- biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This current is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

1.2.4 In service, the analyzer is first calibrated with a gas of known composition equal to, close to, or representative of that to be measured. Gases with ionization potentials near to or less than the energy of the lamp will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials greater than the energy of the lamp will not be detected. The ionization potentials of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to 15.6 eV and are not ionized by any of the lamps available. Gases with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

1.3 Specifications

1.3.1 Refer to the manufacturer's instructions for the technical specifications of the instrument being used. The operating concentration range is typically 0.1 to 2,000 ppm isobutylene equivalent.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). In the absence of a APP, work will be conducted according to the Work Plan (WP) and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Only PIDs stamped Division I Class I may be used in explosive atmospheres. Refer to the project APP/SSHP for instructions pertaining to instrument use in explosive atmospheres.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The Project Manager is responsible for ensuring that the operation and calibration activities comply with this procedure. The Project Manager is responsible for ensuring that all personnel involved in the operation and calibration shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Site Supervisor is responsible for ensuring that all operation and calibration activities are conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

- Calibration Gas: Compressed gas cylinder of isobutylene in air or similar stable gas mixture of known concentration. The selected gas should have an ionization potential similar to that of the vapors to be monitored, if known. The concentration should be at 50-75% of the range in which the instrument is to be calibrated;

- Regulator for calibration gas cylinder;
- Approximately 6 inches of Teflon® tubing;
- Tedlar bag (optional);
- Commercially-supplied zero grade air (optional);
- "Magic Marker" or “Sharpie” or other waterproof marker;
- Battery charger;
- Moisture traps;
- Spare lamps;
- Manufacturer’s instructions; and
- Field data sheets or logbook/pen.

7.0 Procedure

7.1 Preliminary Steps

7.1.1 Preliminary steps (battery charging, check-out, calibration, maintenance) should be conducted in a controlled or non-hazardous environment.

7.2 Calibration

7.2.1 The PID must be calibrated in order to display concentrations in units equivalent to ppm. First a supply of zero air (ambient air or from a supplied source), containing no ionizable gases or vapors is used to set the zero point. A span gas, containing a known concentration of a photoionizable gas or vapor, is then used to set the sensitivity.

7.2.2 Calibrate the instrument according to the manufacturer’s instructions. Record the instrument model and identification number, the initial and adjusted meter readings, the calibration gas composition and concentration, and the date and the time in the field records.

7.2.3 If the calibration cannot be achieved or if the span setting resulting from calibration is 0.0, then the lamp must be cleaned (Section 7.4).

7.3 Operation

7.3.1 Turn on the unit and allow it to warm up (minimum of 5 minutes). Check to see if the intake fan is functioning; if so, the probe will vibrate slightly and a distinct sound will be audible when holding the probe casing next to the ear. Also, verify on the readout display that the UV lamp is lit.

7.3.2 Calibrate the instrument as described in Section 7.2, following the manufacturer’s instructions. Record the calibration information in the field records.

7.3.3 The instrument is now operational. Readings should be recorded in the field records.

7.3.4 When the PID is not being used or between monitoring intervals, the unit may be switched off to conserve battery power and UV lamp life; however, a “bump” test should be performed each time the unit is turned on and prior to taking additional measurements. To perform a bump test, connect the outlet tubing from a Tedlar bag containing a small amount of span gas to the inlet tubing on the unit and record the reading. If the reading is not within the tolerance specified in the project plan, the unit must be recalibrated.

7.3.5 At the end of each day, recheck the calibration. The check will follow the same procedures as the initial calibration (Section 7.2) except that no adjustment will be made to the instrument. Record the information in the field records.

7.3.6 Recharge the battery after each use (Section 7.4).

7.3.7 When transporting, ensure that the instrument is packed in its stored condition in order to prevent damage.

7.4 **Routine Maintenance**

7.4.1 Routine maintenance associated with the use of the PID includes charging the battery, cleaning the lamp window, replacing the detector UV lamp, replacing the inlet filter, and replacing the sample pump. Refer to the manufacturer's instructions for procedures and frequency.

7.4.2 All routine maintenance should be performed in a non-hazardous environment.

7.5 **Troubleshooting Tips**

7.5.1 One convenient method for periodically confirming instrument response is to hold the sensor probe next to the tip of a magic marker. A significant reading should readily be observed.

7.5.2 Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings.

7.5.3 A fogged or dirty lamp, due to operation in a humid or dusty environment, may cause erratic or fluctuating readings. The PID should never be operated without the moisture trap in place.

7.5.4 Moving the instrument from a cool or air-conditioned area to a warmer area may cause moisture to condense on the UV lamp and produce unstable readings.

7.5.5 A zero reading on the meter should not necessarily be interpreted as an absence of air contaminants. The detection capabilities of the PID are limited to those compounds that will be ionized by the particular probe used.

7.5.6 Many volatile compounds have a low odor threshold. A lack of meter response in the presence of odors does not necessarily indicate instrument failure.

7.5.7 When high vapor concentrations enter the ionization chamber in the PID the unit can become saturated or "flooded". Remove the unit to a fresh air environment to allow the vapors to be completely ionized and purged from the unit.

8.0 Quality Control and Assurance

8.1 The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Sampling and Analysis Plan (SAP), hereafter referred to as the project plan.

8.2 Calibration of the PID will be conducted at the frequency specified in the project plan. In the absence of project-specific guidance, calibration will be performed at the beginning of each day of sampling and will be checked at the end of the sampling day or whenever instrument operation is suspect. The PID will sample a calibration gas of known concentration. The instrument must agree with the calibration gas within ±10%. If the instrument responds outside this tolerance, it must be recalibrated.

8.3 Checks of the instrument response (Section 7.5) should be conducted periodically and documented in the field records.

9.0 Records, Data Analysis, Calculations

Safety and survey monitoring with the PID will be documented in a bound field logbook, or on standardized forms, and retained in the project files. The following information is to be recorded:

- Project name and number;
- Instrument manufacturer, model, and identification number;

- Operator's signature;
- Date and time of operation;
- Calibration gas used;
- Calibration check at beginning and end of day (meter readings before adjustment);
- Span setting after calibration adjustment;
- Meter readings (monitoring data obtained);
- Instances of erratic or questionable meter readings and corrective actions taken; and
- Instrument checks and response verifications – e.g., battery check, magic marker response (Section 7.5) or similar test.

10.0 Attachments or References

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, SESD, Enforcement and Investigations Branch, Athens, GA. November 2001.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (APril 2017)

Surface and Subsurface Soil Sampling Procedures

Procedure 3-21

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures for soil sampling. The procedure includes surface and subsurface sampling by various methods using hand auguring, test pit, direct-push, and split-spoon equipment.

1.2 The procedure includes soil sampling for volatile organic compounds (VOCs). For project specific information (e.g. sampling depths, equipment to be used, and frequency of sampling), refer to the Sampling and Analysis Plan (SAP), which takes precedence over these procedures. Surface soil sampling, typically considered to be up to two feet below ground surface by EPA standards, is typically accomplished using hand tools such as shovels or hand augers. Test pit samples are considered subsurface samples, although normally collected via hand tools similar to surface soil sampling or by excavation machinery. Direct-push and split-spoon sampling offer the benefit of collecting soil samples from a discrete or isolated subsurface interval, without the need of extracting excess material above the target depth. These methods dramatically reduce time and cost associated with disposal of material from soil cuttings when compared to test pit sampling. In addition, direct-push and split-spoon sampling methods can obtain samples at targeted intervals greater than 15 feet in depth, allowing for discrete depth soil sampling while speeding up the sampling process. Direct-push methods work best in medium to fine-grained cohesive materials such as medium to fine sands, silts, and silty clay soils. Split-spoon sampling works well in all types of soil, but is somewhat slower than direct-push methods. Samples are composited so that each sample contains a homogenized representative portion of the sample interval. Due to potential loss of analytes, samples for volatile analysis are not composited. Samples for chemical analysis can be collected by any of the above-mentioned sampling methods, as disturbed soil samples. Undisturbed samples are collected, sealed, and sent directly to the laboratory for analysis. For undisturbed samples, the samples are not homogenized.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). In the absence of a APP/SSHP, work will be conducted according to the Work Plan (WP) and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Before soil sampling commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated soil sampling locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Low recovery of soil from sampling equipment will prevent an adequate representation of the soil profile and sufficient amount of soil sample. If low recovery is a problem, the hole may be offset and re-advanced, terminated, or continued using a larger diameter sampler.

4.2 Asphalt in soil samples can cause false positive results for hydrocarbons. To ensure samples are free of asphalt, do not collect samples that may contain asphalt. If the collection of samples potentially containing asphalt is unavoidable, note the sampling depths at which the presence of asphalt are suspected.

4.3 Instrumentation interferences addressed in SOPs for Calibration of the Photoionization Detector (PID), Headspace Screening for Total Volatile Organics, and Equipment Decontamination must also be considered.

4.4 Cross contamination from sampling equipment must be prevented by using sampling equipment constructed of stainless steel that is adequately decontaminated between samples.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The Project Manager is responsible for ensuring that soil sampling activities comply with this procedure. The Project Manager is responsible for ensuring that all personnel involved in soil sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all soil sampling activities are conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

The depth at which samples will be collected and the anticipated method of sample collection (direct-push, split-spoon, hand auger, shovel, or test pits) will be presented in the SAP. The following details equipment typically needed for soil sampling, based on the various methods. See the SAP for specific detail of equipment and supply needs.

6.1 Depending on the nature of suspected contamination, field screening instrumentation may be used for direct sampling. Appropriate instrumentation and calibration standards should be available. If volatile organic contaminants are suspected and a PID will be used, refer to the equipment and instrumentation listed in SOP 3-20 Operation and Calibration of a Photoionization Detector. Equipment in this SOP includes but is not limited to:

- PID/FID;
- Calibration gas; and
- Tedlar® gas bags (for calibration).

6.2 If field screening methods include jar headspace screening for volatile organics, refer to the equipment and procedure in SOP 3-19 Headspace Screening for Total VOCs. Equipment in this SOP includes but is not limited to:

- Clean soil (“drillers jars”) jars; and
- Aluminium foil.

6.3 Appropriate decontamination procedures must be followed for sampling equipment. Refer to SOP 3-06 Equipment Decontamination. Equipment in this SOP includes but is not limited to:

- Phosphate-free detergent;
- Isopropyl Alcohol;
- Tap water;
- Deionized Ultra-Filtered (DIUF) Water;
- Plastic buckets or washbasins;
- Brushes; and
- Polyethylene sheeting.

6.4 The following general equipment is needed for all soil sampling, regardless of method:

- Stainless steel bowls;
- Stainless steel trowels;
- Appropriate sample containers for laboratory analysis;
- Personal Protective Equipment (PPE);
- Logbook;
- Cooler and ice for preservation; and
- Stakes and flagging to document sampling location.

6.5 The following additional equipment is needed for volatile organic sampling:

- Electronic pan scale and weights for calibration; and
- Syringes or other discrete soil core samplers.

6.6 The following additional equipment may be needed for surface and test pit soil sampling:

- Hand Auger

6.7 The following additional equipment may be needed for soil sampling from direct push and/or split-spoon equipment:

- Tape measure or folding carpenter's rule for recording the length of soil recovered.

Note: All subsurface drilling equipment will be provided and maintained by the subcontractor.

7.0 Procedure

7.1 **General Soil Sampling Procedure for All Soil Sampling Methods**

7.1.1 Record the weather conditions and other relevant on-site conditions.

7.1.2 Select the soil sampling location, clear vegetation if necessary, and record the sampling location identification number and pertinent location details.

7.1.3 Verify that the sampling equipment is properly decontaminated, in working order, and situated at the intended sampling location.

7.1.4 Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. Cover surfaces onto which soils or sampling equipment will be placed (i.e. tables with polyethylene sheeting).

7.1.5 Follow the appropriate procedures listed below for either surface, split-spoon, direct push, or test pit sample collection (7.2, 7.3, 7.4, and 7.5 respectively).

7.1.6 Collect soil samples according to procedures listed in Section 7.6 depending on project specific analyses.

7.1.7 Record date/time, sample ID, and sample descriptions in the field logbook or field form. A sketch or description of the location may also be recorded so the sample location can be re-constructed, especially if the location will not be recorded using global positioning satellite (GPS) equipment.

7.1.8 Immediately label the sample containers and place them on ice, if required for preservation. Complete the chain-of-custody form(s) as soon as possible.

7.1.9 Dispose of all excess excavated soil in accordance with the SAP.

7.1.10 If required, mark the sample location with a clearly labelled wooden stake or pin flag. If the location is on a paved surface, the location may be marked with spray paint.

7.1.11 Decontaminate the sampling equipment according to SOP 3-06 Equipment Decontamination.

7.2 Surface Sampling

7.2.1 The criteria used for selecting surface soil locations for sampling may include the following:

- Visual observations (soil staining, fill materials);
- Other relevant soil characteristics;
- Site features;
- Screening results;
- Predetermined sampling approach (i.e. grid or random); and
- Sampling objectives as provided in the SAP.

7.2.2 The following procedures are to be used to collect surface soil samples. Surface soils are considered to be soils that are up to two feet below ground surface, though state regulations and project objectives may define surface soils differently; therefore, the SAP should be consulted for direction on the depth from which to collect the surface soil samples. Sampling and other pertinent data and information will be recorded in the field logbook and/or on field forms. Photographs may be taken as needed or as specified in the SAP.

1. Gently scrape any vegetative covering until soil is exposed. Completely remove any pavement.
2. Remove soil from the exposed sampling area with a trowel, hand auger, or shovel. Put soils within the sampling interval in a stainless steel bowl for homogenizing. Monitor the breathing zone and sampling area as required in the HASP.
3. For VOC analyses, collect representative soil samples directly from the recently-exposed soil using a syringe or other soil coring device (e.g., TerraCore®, EnCore®). Follow procedures in Section 7.6.1 for VOC sampling.
4. Collect sufficient soil to fill all remaining sample jars into a stainless steel bowl. Homogenize the soil samples to obtain a uniform soil composition which is representative of the total soil sample collected according to the following procedure:
    a) Remove all rocks and non-soil objects using a stainless steel spoon or scoop.

b) Form a cone shaped mound with the sample material, then flatten the cone and split the sample into quarters.

c) Use the stainless steel spoon/scoop to mix the quarter samples that are opposite.

d) After mixing the opposite quarters, reform the cone shaped mound.

e) Repeat this procedure a minimum of five (5) times, removing any non-soil objects and breaking apart any clumps.

7.3 Split-Spoon Sampling

7.3.1 At each boring location, the frequency and depth of split-spoon samples will be determined from the SAP. Split-spoon samples may be collected continuously, intermittently, or from predetermined depths.

7.3.2 Split-spoon samplers shall be driven into undisturbed soil by driving the spoon ahead of the drill augers/casing. In cohesive soils, or soils where the borehole remains open (does not collapse), two split-spoon samples may be taken prior to advancing the augers/casing.

7.3.3 After split-spoons are retrieved, open the split-spoon and measure the recovery of soil. If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the SAP, VOC and headspace samples should be collected (see Section 7.6.1) prior to logging the sample.

7.3.4 If headspace screening for VOCs is required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.3.5 Soils collected using the split-spoon sampler will be logged by the field representative using the procedure required in the SAP.

7.3.6 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.3.7 The SAP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from that interval will be set aside in a clearly labelled stainless steel bowl covered with aluminium foil. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.3.8 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

7.4 Direct Push Sampling

At each boring location, the frequency of direct-push samples will be determined from the SAP. Typically, samples with direct-push equipment are collected in 4 foot (ft) intervals, but smaller (e.g., 2 ft) and larger (e.g., 5 ft) intervals are also possible.

1. Sample using Macro-Core samplers with acetate liners to obtain discrete soil samples at the depths specified in the SAP.

2. Cut open the acetate liner. If required in the SAP, immediately scan the recovered soil boring for VOCs using a PID by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the

highest PID reading and the depth at which it was observed along with all other pertinent observations. VOC and headspace samples, if required in the SAP should be collected (see Section 7.6.1) prior to logging the sample.

3. If required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

4. Soils collected using the direct-push sampler will be logged by the by the field representative using the procedure required in the SAP.

5. Collect the remainder of the sample into a stainless steel bowl. Homogenize the soil collected so that the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

6. Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

7.5 Test Pit Sampling

7.5.1 Excavate the test pit to the desired depth.

7.5.2 Using the excavator bucket, collect soil samples as specified in the SAP. Collect a sample and perform screening analyses as required by the SAP. If VOCs contamination is suspected, perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.5.3 Collect the sample from center of the bucket to avoid potential contamination from the bucket.

7.5.4 VOC samples should also be collected from an undisturbed section soil in the excavator bucket. The top layer of exposed soil should be scraped away just prior to collecting the VOC samples.

7.5.5 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.5.6 Dispose of all excavated soil according to the SAP.

7.6 Sample Collection Methods

7.6.1 Volatile Organics Sampling

For soils collected for analyses of volatile organics, including Volatile Petroleum Hydrocarbons (VPH) or other purgable compounds, a closed system is maintained. From collection through analysis, the sample bottles are not opened. The bottle kit for a routine field sample for these analyses will typically include three 40-mL VOA vials and one soil jar. Two 40-mL VOA vials will contain either 5 mL reagent water or 5 mL sodium bisulfate and magnetic stir bars (i.e., low level vials). The third VOA vial will contain 15 mL methanol with no magnetic stir bar (i.e., high level vial). These vials are usually provided by the laboratory and are pre-weighed, with the tare weight recorded on the affixed sample label. No additional sample labels are affixed to the VOA vials, as addition of a label would alter the vial weight. All information is recorded directly on the sample label using an indelible marker. The soil jar is provided for percent solids determination. For VOC or VPH analyses, samples are collected prior to sample homogenization. Collect the VOC sample in accordance with the procedure described below.

1. Determine the soil volume necessary for the required sample weight, typically 5 grams:

   a) Prepare a 5 mL sampling corer (e.g., Terra Core®) or cut-off plastic syringe.

   b) Tare the sampler by placing it on the scale, and zeroing the scale.

   c) Draw back the plunger to the 5 gram mark or 5mL (5cc) mark on cut-off syringe, and insert the open end of the sampler into an undisturbed area of soil with a twisting motion, filling the

sampler with soil. Note the location of the plunger with respect to the milliliter (cc) or other graduation printed on the sampler.

d) Weigh the filled sampler, and remove or add soil until the desired weight is obtained. Note the location of the plunger which corresponds to this weight. Do not use this sample for laboratory analysis.

2. Once the required soil volume has been determined, pull the plunger back to this mark and hold it there while filling the syringe for each sample.

3. Collect 5 grams of soil using the cut-off syringe or Terra Core® sample device. Extrude the 5-grams of soil into one of the low level 40-mL VOA vials. Quickly wipe any soil from the threads of the VOA vial with a clean Kimwipe® and immediately close the vial. It is imperative that the threads be free from soil or other debris prior to replacing the cap on the vial in order to maintain the closed system necessary for the analysis.

4. Gently swirl the vial so that all of the soil is fully wetted with the preservative.

5. Fill the other low level 40 mL VOA vial in this manner.

6. Repeat the process for the high level VOA vials, only for the high level VOA vial three 5 gram aliquots (i.e., 15 grams total) should be extruded into the high level VOA vial.

   NOTE: Depending on the laboratory, some high level VOA vials only contain 5 mL or 10 mL of methanol. If this is the case, either 5 grams total or 10 grams total, respectively, should be extruded into the high level VOA vial. In other words, the mass of soil in grams should be identical to the volume of methanol in mL (i.e., 1:1 ratio of soil to methanol).

7. Collect any additional QC sample collected (e.g., field duplicate, MS, and MSD) in the same manner as above.

8. Fill the 4-oz glass jar with soil from the same area for percent moisture determination.

7.6.2 Soil Sampling Method (All other analyses except VOC/VPH)

When all the required soil for a sampling location has been obtained, the soil can be homogenized as described in section 7.2. Collect sufficient volume to fill all of the remaining sample containers at least ¾ full for all other analyses. Homogenize the soil in a decontaminated stainless steel bowl, removing rocks, sticks, or other non-soil objects and breaking apart any lumps of soil prior to filling the remaining sample containers.

NOTE: Soil samples must contain greater than 30% solids for the data to be considered valid.

8.0 Quality Control and Assurance

8.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the SAP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the SAP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

8.2 Quality control requirements are dependent on project-specific sampling objectives. The SAP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

9.0 Records, Data Analysis, Calculations

All data and information (e.g., sample collection method used) must be documented on field data sheets, boring logs, or within site logbooks with permanent ink. Data recorded may include the following:

- Weather conditions;
- Arrival and departure time of persons on site;
- Instrument type, lamp (PID), make, model and serial number;
- Calibration gas used;
- Date, time and results of instrument calibration and calibration checks;
- Sampling date and time;
- Sampling location;
- Samples collected;
- Sampling depth and soil type;
- Deviations from the procedure as written; and
- Readings obtained.

10.0 Attachments or References

SOP 3-06, Equipment Decontamination

SOP 3-19, Headspace Screening for Total VOCs

SOP 3-20, Operation and Calibration of a Photoionization Detector

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Sediment Sampling

Procedure 3-22

1.0 Purpose and Scope

1.1 Sediment contamination is a widespread environmental problem that can pose a threat to a variety of aquatic ecosystems. Sediment functions as a reservoir for common contaminants such as pesticides, herbicides, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and metals such as lead, mercury, and arsenic. Contaminated sediments represent a hazard to aquatic life through direct toxicity, as well as to aquatic life, wildlife, and human health through bioaccumulation. Accurate assessment of environmental hazards posed by sediment contamination depends in large part on the accuracy and representativeness of sediment collection and analyses (U.S. EPA, 2001).

1.2 Selection and proper use of sediment sampling equipment is essential to the collection of accurate, representative sediment data that will meet the project Data Quality Objectives (DQOs). Most sediment collection devices are designed to isolate and consistently retrieve a specified volume and surface area of sediment, from a required depth below the sediment surface, with minimal disruption of the integrity of the sample and no contamination of the sample. Maintaining the integrity of the collected sediment, for the purposes of the measurements intended, is a primary concern in most studies because disruption of the sediment's structure could change its physiochemical and biological characteristics, thereby influencing the bioavailability of contaminants and the potential toxicity of the sediment (U.S. EPA, 2001).

When selecting the type of sediment sampling equipment to be used for an event, the project DQOs as well as the sediment characteristics should be considered. Related to the project DQOs is the desired depth of sediment sampling. For monitoring and assessment studies where historical contamination is not the focus, the upper 10 to 15 centimeters (cm) is typically the horizon of interest, as this is the horizon that generally contains the most recently deposited sediments and most epifaunal and infaunal organisms (U.S. EPA, 2001). The 0-6 inches interval for sediments with less than two feet of water is also used for human health risk assessment purposes. Sampling of these horizons can usually be done with grab samplers. However, if sediment contamination is being related to organism exposures (e.g. benthic macroinvertebrates and/or fish), or if characterization of deeper sediments is important for comparison of recent surficial versus historical contamination, then more precise sampling of sediment depths might be needed, and a hand corer may be more suitable (U.S. EPA, 2001).

1.3 This standard operating procedure (SOP) describes the procedure for the collection of sediment samples using the Petite Ponar® Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer (or similar sampling devices). The applicability of each of the sediment samplers is described below.

The Petite Ponar® Grab Sampler is used to collect sediment samples in:

- Firm, hard bottoms such as sand, gravel, consolidated marl, and clay
- Mixtures of sand, stones, and coarse debris
- Soft or mucky sediments

The Ekman Bottom Grab Sampler is used to collect sediment samples in:

- Soft, finely divided littoral bottoms free from vegetation and intermixtures of sand, stones, and other coarse debris
- Bottoms composed of finely divided mulch, mud, muck, or submerged fine peaty materials

The Wildco® Hand Corer is used:

- To collect sediment samples for geological characterizations and dating
- To collect sediment samples for programs where it is important to maintain an oxygen-free environment for the sample during collection
- To collect sediment samples from a deeper depth than a grab sampler, and to characterize the depth of contamination at a site
- To investigate the historical input of contaminants to aquatic systems
- To collect sediment samples in semi-consolidated and soft sediment

Pictures and exploded diagrams of the Petite Ponar Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer are presented in Figures 1, 2, and 3, respectively.

1.4 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.5 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first location. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring of sample locations to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and solid or liquid matrix through the use of respirators and disposable clothing.

2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during sediment sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, and waders (if applicable). Refer to the project-specific HASP for the required PPE.

2.3 Handle all sediments removed from potentially contaminated locations in accordance with the IDW handling procedures in SOP 3-05, IDW Management.

2.4 Depending upon the type of contaminant expected or determined in previous sampling efforts, employ the following safe work practices:

- If sampling from a boat, all sampling personnel should wear personal flotation devices (PFDs) when in the boat, and should follow all health and safety protocols for working in a boat presented in the project-specific HASP.
- Lifting the samplers into the boat, dumping its contents, and washing those contents may require leaning over the side of the boat. Care should be taken to keep the boat in proper balance at all times during sampling.
- Severe injury to fingers or hands can be caused by movement of the lever arms of the Petite Ponar® Grab Sampler. Do not handle or move the Petite Ponar® Grab Sampler unless the safety pin is fully inserted in the locking holes.
- Severe injury to fingers or hands can be caused by the closing of the sharpened scoops of the Ekman Bottom Grab Sampler. Handle the Ekman Bottom Grab Sampler very carefully when the springs are set and the cable loops are hooked (armed) on the Twin-Pin™ pins on the release mechanism. Do not “arm” the Ekman Bottom Grab Sampler until the sampler is ready

to be used. The Ekman Bottom Grab Sampler spring-loaded jaws are potentially dangerous; extreme care must be exercised when setting the jaws. To prevent injury (and to extend the life of the springs), unhook both springs from their scoop buttons after each sampling session.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that sediment sampling activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all field sampling personnel involved in sediment sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.

4.4 **Field sampling personnel** are responsible for the implementation of this procedure.

4.5 The field sampler and/or task manager is responsible for directly supervising the sediment sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

5.0 Equipment and Supplies

5.1 For sediment sampling using all types of equipment, the following supplies are required:

- Stainless steel bowls
- Stainless steel hand trowels, spoons, spatulas, and scoops
- Munsell Color Chart
- Particle size chart

5.2 Petite Ponar® Grab Sampler

- 3/16” braided polyester line
- Auxiliary weights

5.3 Ekman Bottom Grab Sampler

- 11 oz split messenger
- 3/16” braided polyester line
- Extension Handle
- Auxiliary weights

5.4 Wildco® Hand Corer

- 3/16” braided polyester line
- Extension handle
- Stainless steel core catchers (for normal sediments)
- Eggshell™ core catchers (for wet sediments)
- Stainless steel nose piece
- Cellulose acetate butyrate (CAB) liners
- Core liner end caps
- Core liner cutter
- Geologists table

- Auxiliary weights

6.0 Procedure

6.1 Depending on the characteristics of the site being investigated, sediment samples may be collected from a boat, or by sampling personnel in waders. In all instances, sediment sampling should begin from the most downstream location and proceed to the most upstream location. If sediment samples are collocated with surface water samples, the surface water sample should be collected prior to the sediment sample in order to avoid increased turbidity from displaced sediment. Regardless of the type of sediment sampling equipment used, documentation of field observations and collection activities should be recorded on the sediment sampling sheet or electronic data collection device. The following observations should be recorded on the sediment sampling form (see Attachment 1) for all sediment sampling activities:

- Sample location
- Weather conditions and other relevant site conditions
- Depth of water to the nearest 0.1 foot. A surveyor rod may be used. If the surveyor rod is used, minimize water turbulence and do not disturb any sediment.
- Physical characteristics of the water body such as estimated current speed (stagnant, slow, medium, or fast) and direction, odor, color, presence of any dead vegetation, surface sheens, etc.
- Sediment color according to the Munsell Color Chart
- Sediment grain size according to a particle size chart

Specific procedures for the collection of sediment samples using the Petite Ponar® Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer are presented below.

6.2 Petite Ponar® Grab Sampler

6.2.1 Inspect the sampler to ensure all parts are in good working condition.

6.2.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.2.3 Attach the 3/16" braided polyester line to the sampler by looping the line through the clevis at the top center of the lever arms and tying securely. Tie the other end of the line to the boat (if applicable), or make sure to hold on to the other end of the line. Strong, tight knots (e.g. bowline, two half hitches) are essential for operator safety and to prevent losing the sampler. If necessary, attach the auxiliary weights to the sampler according the manufacturer's directions.

6.2.4 Insert the Pinch-Pin™ into its hole in the lever arms, making sure to firmly push the Pinch-Pin™ into the hole. As long as the line is taught, the Pinch-Pin™ will stay in its place. When the line becomes the least bit slack (e.g. when the sampler hits the bottom), the Pinch-Pin™ spring will force the Pinch-Pin™ out of its hole, allowing the scoops to close.

6.2.5 Just before lowering the grab into the water, and with the line taught, remove the safety pin so the closing mechanism will release when the sampler is on the bottom. Make sure to keep the line taught, as any loss of tension in the line will cause the Pinch-Pin™ to pop out, closing the sampler.

6.2.6 Lower the sampler into the water in a slow and controlled fashion, especially during the final 1-2', such that the bow wave is minimized, thus minimizing the dispersal of fine material on the sediment surface. At no time should the sampler be allowed to "free fall" down through the water column.

6.2.7 Once the sampler has reached the bottom, release the tension on the line, and allow the sampler to sink into the sediment momentarily. The release of tension on the line will cause the Pinch-Pin™ to pop out.

6.2.8 Collect the sample by pulling on the line, which will cause the lever arms to drive the scoops into the sediment in a closing motion. Keep pulling on the line in a controlled fashion until the scoops drive through the sediment and close.

6.2.9 Once the sampler scoops have closed, continue pulling on the line in a controlled fashion in order to retrieve the sampler back to the surface. When the sampler reaches the surface, lift it clear and bring it above a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.

6.2.10 Prior to sampling and sample homogenization, the overlying water in the sampler should be siphoned off, and not decanted (U.S. EPA 2001).

6.2.11 If acid volatile sulfide/simultaneously extracted metals (AVS/SEM) samples are to be collected, open the top screens of the sampler and collect the AVS/SEM sample directly from the sediment contained in the sampler according to the procedures specified in the project-specific SAP.

6.2.12 If volatile organic compound (VOC) samples are to be collected, open the top screens of the sampler and collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the undisturbed sediment contained within the sampler, making sure to follow all VOC sampling procedures specified in the project-specific SAP. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the undisturbed sediment contained within the sampler.

6.2.13 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), open the sampler by pulling the two scoops open, taking care to keep hands and fingers away from the sharpened edges of the scoops, and allow the sediment to exit the sampler into the decontaminated stainless steel bowl.

6.2.14 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.2.3 through 6.2.12 until an adequate sample volume has been collected, taking care to deploy the sampler to an area that is proximal and upstream, but not on top of, the previous sample location.

6.2.15 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

6.3 Ekman Bottom Grab Sampler with the 11 oz Split Messenger

6.3.1 Inspect the sampler to ensure all parts are in good working condition.

6.3.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.3.3 Attach the 3/16” braided polyester line to the sampler by passing the line through the trip mechanism and knotting it securely below the underlying plate. Thread the 11 oz split messenger on the line, and tie the other end of the line to the boat (if applicable), or make sure to hold on to the other end of the line. Strong, tight knots (e.g. bowline, two half hitches) are essential to prevent losing the sampler. If necessary, attach the auxiliary weights to the sampler according the manufacturer’s directions.

6.3.4 Set the spring on the side of the sampler by hooking the end of the spring onto one scoop button and stretching the spring to reach the second scoop button. Repeat this procedure with the spring on the other side of the sampler.

6.3.5 Arm the scoops by hooking one cable loop to one Twin-Pin™ pin in the trip assembly on the top of the sampler. The white ball on the cable can be used as a hand grip to assist getting the cable loop hooked onto the Twin-Pin™ pin. Repeat for the opposite cable loop. The sampler is now armed and dangerous. Do not allow anything to come in contact with the trip assembly at the top of the sampler, as this may cause a sudden and unexpected closure of the sampler.

6.3.6 Lower the sampler into the water in a slow and controlled fashion, especially during the final 1-2', such that the bow wave is minimized, thus minimizing the dispersal of fine material on the sediment surface. At no time should the sampler be allowed to "free fall" down through the water column.

6.3.7 Once the sampler has reached the bottom, allow the sampler to settle momentarily. Once the sampler has settled, hold the line with just enough tension to keep it straight, and send the 11 oz split messenger down the line. Once the 11 oz split messenger impacts Twin-Pin™ strike pad in the trip assembly on the top of the sampler, the two cable loops will be released from the Twin-Pin™ pins, and the spring-loaded scoops of the sampler will automatically close.

6.3.8 Retrieve the sampler by pulling up the line in with a moderate, steady speed. When the sampler reaches the surface, lift it clear and bring it above a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.

6.3.9 Prior to sampling and sample homogenization, the overlying water in the sampler should be siphoned off, and not decanted (U.S. EPA 2001).

6.3.10 If AVS/SEM samples are to be collected, open the top lids of the sampler and collect the AVS/SEM sample directly from the sediment contained in the sampler according to the procedures specified in the project-specific SAP.

6.3.11 If VOC samples are to be collected, open the top lids of the sampler and collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the undisturbed sediment contained within the sampler, making sure to follow all VOC sampling procedures specified in the project-specific SAP. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the undisturbed sediment contained within the sampler.

6.3.12 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), open the sampler by pulling on the white balls on both cables, opening the spring-loaded scoops and allowing the sediment to exit the sampler into the decontaminated stainless steel bowl. While the spring-loaded scoops are being held open, do not place hands or fingers inside or underneath the sampler.

6.3.13 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.3.4 through 6.3.11 until an adequate sample volume has been collected, taking care to deploy the sampler to an area that is proximal and upstream, but not on top of, the previous sample location.

6.3.14 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

6.4 Ekman Bottom Grab Sampler with the Extension Handle

6.4.1 Inspect the sampler to ensure all parts are in good working condition.

6.4.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.4.3 Attach the extension handle to the top of the sampler with machine bolts.

6.4.4 Arm the sampler according to the procedures described in steps 6.3.3 and 6.3.4 above.

6.4.5 Using the extension handle, lower the sampler to a point 4-6" above the sediment surface, and drop the sampler to the sediment, keeping the sampler vertical at all times.

6.4.6 Trigger the trip assembly by depressing the button on the upper end of the extension handle. This will cause the two cable loops to be released from the Twin-Pin™ pins, and the spring-loaded scoops of the sampler will automatically close.

6.4.7 While keeping the sampler vertical, bring the sampler over to a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.

6.4.8 Collect samples according to the procedures described in steps 6.3.8 through 6.3.13 above.

6.5 Wildco® Hand Corer with the Push Handles

6.5.1 Inspect the sampler to ensure all parts are in good working condition:

- Assemble and disassemble the core tube from the head and nose piece to make sure the threads are not binding. If the threads are binding, consult the manufacturer's directions.
- Make sure that the CAB plastic liner can slide easily in and out of the core tube.
- Make sure the bottom edge of the core tube and nose piece are sharp and free from nicks or dents. If necessary, file smooth using a round file.
- Check the flutter valve for ease of movement.
- Check the flutter valve seat to make sure it is clear of any obstruction, disfigurement, grease, and/or oil that could prevent a tight closure.

6.5.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.5.3 Screw the corer head onto the core tube, and screw the two handles onto the corer head.

6.5.4 Insert a CAB plastic liner into the core tube, insert a core catcher onto the end of the CAB plastic liner (stainless steel for normal sediments, Eggshell™ for wet sediments), and screw the stainless steel nose piece onto the core tube. If using the hand corer from a boat, bridge, high dock, etc., be sure that the appropriate extension handle (5', 10' or 15') is attached to the corer head.

6.5.5 Get in position over the sampling location. If wading in shallow water, be sure to approach the sample location from the downstream side. Line up the sampler, aiming it vertically for the point where the sample is being taken, and push the hand corer in a smooth continuous motion through the water and into the sediment. Increase the thrust as necessary to obtain the penetration desired. Do not hammer or pound the corer into the sediment.

6.5.6 Retrieve the sample by pulling straight up on the handles, keeping the corer as vertical as possible. If the corer has not been completely submerged, close the flutter valve by hand and press it shut while the sample is being retrieved. The flutter valve must be kept very wet if it is to seal properly and prevent sample washout. If the substrate is gripping the corer too tightly, gently rock the top of the corer back and forth horizontally to increase the size of the hole created by the corer and reduce the pull-out suction.

6.5.7 Unscrew the nose piece from the corer and cap the bottom end of the CAB core liner. Release the flutter valve to free the CAB core liner, and slide the CAB core liner from the core tube. Cap the top of the CAB core liner and inspect the CAB core liner for recovery. If the recovery is adequate, proceed to step 6.5.8. If the recovery is not adequate, resample the location by repeating steps 6.5.3 through 6.5.7.

6.5.8 Bring the CAB core liner with the sediment sample over to the geologist table, keeping the core vertical. Place the CAB core liner on the geologist table and cut open with a core liner cutter. If AVS/SEM samples are to be collected, collect the AVS/SEM sample directly from the sediment contained in the core liner according to the procedures specified in the project-specific SAP. If VOC samples are to be collected, collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the sediment core. Consult the project-specific SAP for project-specific VOC sediment sampling procedures. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the sediment core.

6.5.9 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), use a decontaminated stainless steel spoon to transfer the remaining sediment core into a decontaminated stainless steel bowl.

6.5.10 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.5.3 through 6.5.8 until an adequate sample volume has been collected, taking care to deploy the corer to an area that is proximal, but not on top of, the previous sample location.

6.5.11 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

6.6 Wildco® Hand Corer with the Clevis and Line

6.6.1 Inspect the corer as described in step 6.5.1 above.

6.6.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.6.3 Screw the corer head onto the core tube. Attach the 3/16” braided polyester line to the corer by passing the line through the clevis in the corer head and knotting it securely. Strong, tight knots are essential to prevent losing the corer. If necessary, attach the auxiliary weights to the sampler according the manufacturer’s directions.

6.6.4 Insert a CAB plastic liner into the core tube, insert a core catcher onto the end of the CAB plastic liner (stainless steel for normal sediments, Eggshell™ for soupy sediments), and screw the stainless steel nose piece onto the core tube.

6.6.5 Position the corer over the drop point and steady momentarily, making sure to keep the corer vertical at all times. Make sure to arrange the 3/16” braided polyester line to run freely. Since the corer’s penetration is by simple gravity, it is important that there be no restraint on the corer during descent by stricture on the line. Keep a firm hold on the free end of the line, or tie it to the boat (if applicable) or some other permanent fixture.

6.6.6 Drop the corer into the water, and allow the corer to free fall until it hits the sediment surface. The corer should not be dropped to depths greater than 20’ to 30’. Dropping the corer to depths greater than 20’ to 30’ may result in the corer striking the sediment surface at an angle less than 90°, resulting in an unsatisfactory sample.

6.6.7 Once the corer has entered the sediment and is no longer falling, draw the line taut, and then pull on the line to pull the corer from the sediment. Once the corer has been pulled free from

the sediment, bring the corer back to the surface by pulling up the line, using a smooth, hand-over-hand fashion. This movement automatically causes the flutter valve to close, preventing sample washout in all but the soupiest of sediments.

6.6.8 Once the corer has been returned to the surface, lift the corer clear of the water, being careful to keep the corer as vertical as possible at all times.

6.6.9 Collect the sediment sample according to the procedures outlined in steps 6.5.6 through 6.5.11 above.

7.0 Quality Control and Assurance

7.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

7.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

8.0 Records, Data Analysis, Calculations

8.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Sample Collection Records;
- Field logbook;
- Chain-of-custody forms; and
- Shipping labels.

8.2 Sample collection records (Attachment 1) will provide descriptive information for the sediment samples collected at each location.

8.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.

8.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

8.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

9.0 Attachments or References

Attachment 1 – Sediment Sample Collection Record

Figure 1 – Petite Ponar® Grab Sampler and Exploded Diagram

Figure 2 – Ekman Bottom Grab Sampler (Large, Tall, and Standard Sizes) and Exploded Diagram

Figure 3 – Wildco® Hand Corer (with Case and Accessories) and Exploded Diagram

Figure 4 – Illustrations of Acceptable and Unacceptable Grab Samples

NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

U.S. Environmental Protection Agency (U.S. EPA). 2001. *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual.* October.

Wildlife Supply Company. 2003. 2424- Hand Corer Instructions.

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Wildlife Supply Company. 2004. 1728-G30/ 1728-G40 Petite Ponar® Grab.

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Attachment 1

Sediment Sample Collection Record

SEDIMENT SAMPLE COLLECTION FORM

Project Name:			
Date(s):			
Project #:		**Date:**	
Sample Location ID:		**Time:**	
Sample #:		**Weather:**	
Samplers:			
Sample Information:			
Sample Depth:		**Sampling Device:**	
Water Depth:			
Distance from River Bank:			
River Flow Rate:			
Field Decon:	Yes No Dedicated	**Sample Type:**	Grab Composite
Munsell Color:			
Sample Description:			
Other physical characteristics of water body at sample location: (Water color, turbidity, odor, presence of sheens, dead\stressed vegetation)			
Sample Comments/Description:			

Figure 1
Petite Ponar® Grab Sampler and Exploded Diagram

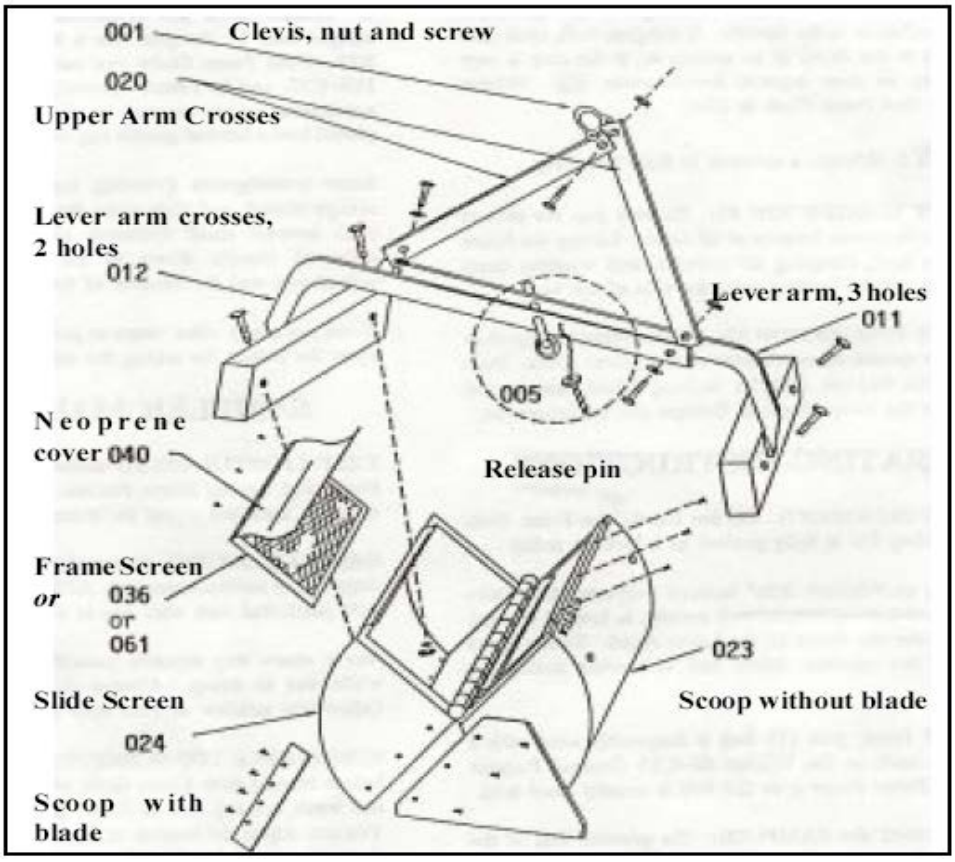
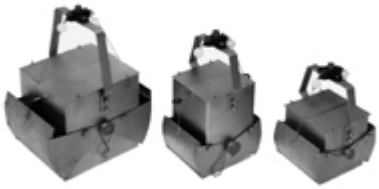


Figure 2

Ekman Bottom Grab Sampler (Large, Tall, and Standard Sizes) and Exploded Diagram



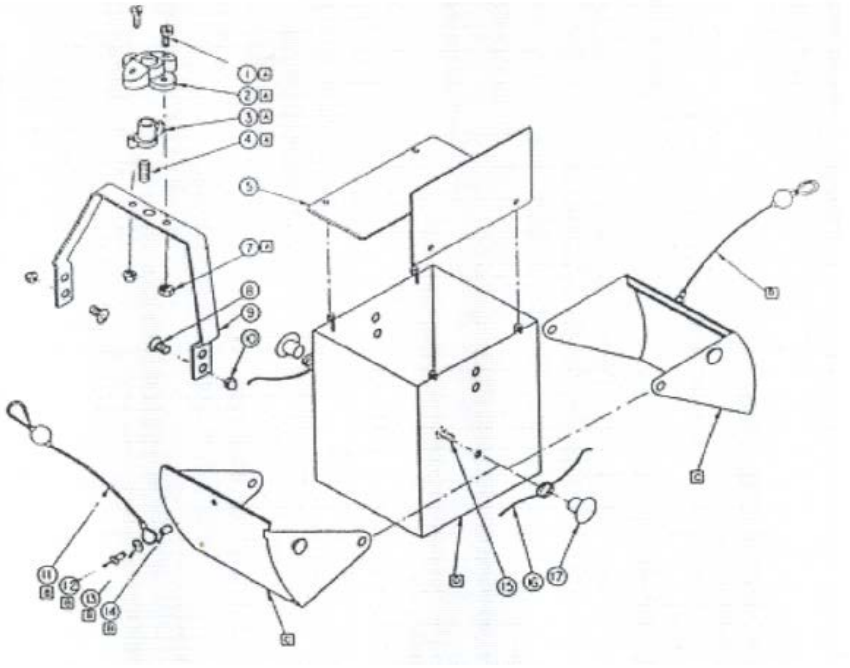
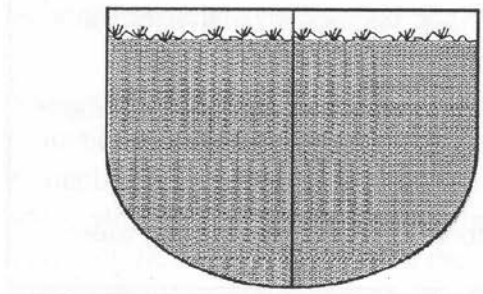
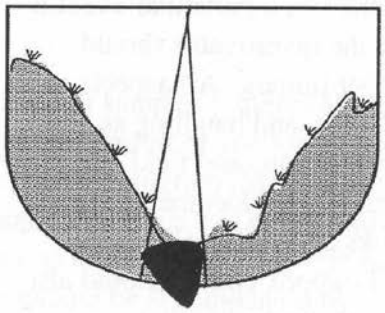


Figure 3
Wildco® Hand Corer (with Case and Accessories) and Exploded Diagram

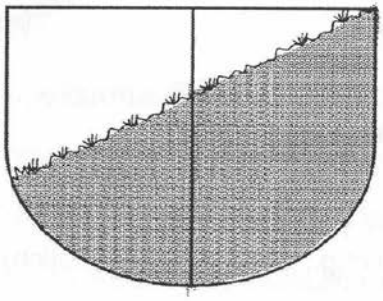
Figure 4
Illustrations of Acceptable and Unacceptable Grab Samples



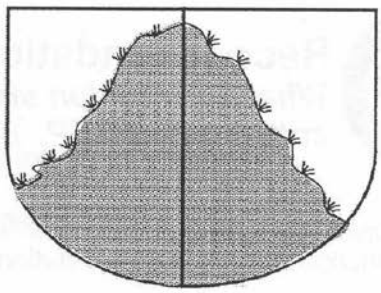
Acceptable if Minimum Penetration Requirement Met and Overlying Water is Present



Unacceptable (Washed, Rock Caught in Jaws)



Unacceptable (Canted with Partial Sample)



Unacceptable (Washed)

Standard Operating Procedure 3-24
Water Quality Parameter Testing for Groundwater Sampling

1.0 PURPOSE

This standard operating procedure (SOP) represents minimum standard of practice. State and federal requirements may vary, and this SOP does not replace state and federal requirements that must be consulted before work begins. Further, if a project-specific work plan has been created, the work plan should be considered the ruling document. This SOP may be modified to meet specific regulatory, client, or project specific criteria.

If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to water quality parameter testing, then those procedures may be added as an appendix to the project-specific Sampling and Analysis Plan (SAP).

2.0 SCOPE

This procedure provides guidance for expected sampling methods and protocols by all personnel related to the measurement of water quality parameters.

Field measurements of water quality parameters are commonly performed to evaluate surface water and groundwater. These tests are often performed to evaluate basic water quality parameters, to evaluate natural attenuation parameters, and to assess the presence of pore water entering a well.

As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by either the Project Manager or the Quality Assurance (QA) Manager, and documented.

3.0 DEFINITIONS

3.1 Barometric Pressure (BP)

The density of the atmosphere, which varies according to altitude and weather conditions.

3.2 Conductivity/Specific Conductance

A measure of the ability of water to pass electrical current, which increases with the amount of dissolved ionic substances (i.e., salts). Conductivity is inversely related to the resistance of a solution and is measured in units of mhos per centimeter (mhos/cm) (inverse ohms/cm, Siemens/cm). The conductivity of water increases with increasing temperature.

Specific Conductance is corrected for 25 degrees Celsius (°C); for this reason, it is best to record Specific Conductance. If Conductivity is recorded, the temperature of the sample MUST recorded.

3.3 Dissolved Oxygen (DO)

The amount of oxygen present in water and available for respiration. DO is typically measured in milligrams per liter (mg/L). Oxygen is less soluble in warm and salty waters, so the instrument compensates the apparent percent saturation for changes in temperature and conductivity. Most probes measure the current resulting from the electrochemical reduction of oxygen (at a gold cathode) diffusing through a selective membrane. Because oxygen is being removed from the sample to perform the measurement, sample flow is required to prevent false low readings due to depletion of oxygen in the solution in front of the probe. Optical DO probes do not remove oxygen from the sample and are less affected by salts. The common range of DO in groundwater is 0.0 to 3.0 mg/L. Measurements outside of this range suggest that the meter may not be operating correctly.

3.4 Nephelometric Turbidity Unit (NTU)

The measurement of light passing through a sample based on the scattering of light caused by suspended particles.

3.5 pH

A measure of acidity and alkalinity of a solution using a logarithmic scale on which a value of 7 represents neutrality, lower numbers indicate increasing acidity, and higher numbers are increasingly basic.

3.6 Oxidation-Reduction Potential (ORP)

Also known as redox or eH, ORP is a measurement of the potential for a reaction to occur, which generally indicates the oxygen status of a sample. The probe consists of a platinum electrode, the potential of which is measured with respect to a reference electrode that rapidly equilibrates with the potential of the sample solution. A positive value indicates that oxygen is present. A negative value indicates an anaerobic environment or reducing condition. For this reason, negative ORP readings should be associated with DO readings of less than 0.5 mg/l; with negative ORP readings the water may exhibit a sulfur odor or gray color. Positive ORP readings should be associated with DO readings greater than 0.5 mg/L and lack of sulfur odors. Because of the complex relationship between ORP and temperature, no compensation is attempted; it is thus best to report both the ORP and temperature of a water sample.

3.7 Total Dissolved Solids

A measure of the quantity of materials in water that are either dissolved or too small to be filtered.

3.8 Turbidity

Measure of the clarity of water in NTUs. Potable water typically has NTU values between 0.0 and 0.3 NTUs, depending on the state or regulatory program.

4.0 RESPONSIBILITIES

The Porject Manager, or designee, is responsible for ensuring that these standard groundwater sampling activities are followed and shall review all groundwater sampling forms at the conclusion of a sampling event. The CTO Manager is responsible for ensuring that all personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks. The QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure. The Field Manager is responsible for ensuring that all project field staff follows these procedures.

Field sampling personnel are responsible for the implementation of this procedure. Personnel are required to be knowledgeable of the procedures in this SOP. Training and familiarization with this SOP shall be documented in the training file for each employee. The field sampler and/or Field Manager is responsible for directly supervising the calibration procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the Project Manager, QA Manager, or Technical Director and then documented in the field logbook and associated report or equivalent document.

5.0 PROCEDURES

5.1 Purpose

The procedures will vary depending on parameters being measured, method of sampling, and the method of measurement used. The information here is a general guidance and the site-specific documents and manufacturer manuals supersede these procedures.

5.2 Cautions

Improper use of water quality testing equipment could result in equipment damage or compromised sampling results. Personnel should be trained to operate the test equipment being used for a field operation and should be trained in the proper techniques for collecting and

logging water quality parameters. Personnel should also be able to recognize problems with test equipment and have someone available for basic troubleshooting and repair.

5.3 Interferences

During field testing, water quality data that is documented from field testing equipment may be influenced by certain outside factors that are unrelated to the actual site water quality. Such parameters and equipment include the following:

pH Meters

- Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use isopropyl alcohol very sparingly so that the electronic surface is not damaged.

- Poorly buffered solutions with low specific conductance (less than 200 microsiemens per centimeter) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

Dissolved Oxygen

- Dissolved gases (e.g., hydrogen sulfide, halogens, sulfur dioxide) are a factor with the performance of DO probes. The effect is less pronounced on optical DO meters. Meter type and potential interferences should be considered based on potential sulfate/sulfide or nitrate/nitrite reducing environments.

- Exposure of the sample to the atmosphere will cause elevated DO measurements.

Turbidity Meter

- If the weather is warm and humidity is high, condensation may collect on the cuvet. To avoid this, allow the sample to warm and dry the outside of the cuvet before making the measurement. One method used to accomplish this is to place the cuvet against one's body (armpits work well).

Temperature

- Sample temperature will change rapidly when there are significant differences between the sample and ambient air.

5.4 Apparatus and Materials

Field personnel shall consult the site work plan and SAP to review the equipment requirements for the sampling procedures to be followed during the sampling effort. The specific apparatus and materials required will depend on the water quality parameters being monitored. Table 1 shows the common equipment used in water quality parameter testing.

Table 1
Water Quality Parameter Testing — Common Equipment

Water Quality Parameter Instrument	Calibration Standards Required	Other Equipment
pH Meter	Yes - 2 or 3 Point Standards depending on groundwater range. Calibration must cover the range to be measured. If samples are above or below typical buffer standards (4, 7 and 10), special order buffers that fall outside groundwater pH range.	Container or flow thru cell for holding sample
Specific Conductance	Yes	Container or flow thru cell for holding sample
ORP Meter	Yes	Container or flow thru cell for holding sample
Turbidity Meter	Yes	Container or flow thru cell for holding sample
DO	No	Container or flow thru cell for holding sample
Thermometer	No	Container or flow thru cell for holding sample
Flow Rate	No	Calibrated Container

Notes:
ORP = Oxidation-Reduction Potential
DO = Dissolved Oxygen

5.5 Instrument or Method Calibration

Most monitoring instruments require calibration before use, and this calibration must be conducted in the field under the ambient climatic conditions that will be present during field sampling. Calibration of monitoring instruments shall be performed in accordance with the manufacturer's specifications and recorded in the provided form in Attachment 1. Site-specific instrument calibration requirements should be specified in the SAP. The following minimum calibration requirements apply to the various types of meters used to gather water quality measurements.

Initial Calibration (IC): Before use, the instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., DO saturation) or a known value of a

calibration standard. An IC is performed in preparation for the first use of an instrument or if a calibration verification does not meet acceptance criteria.

Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following IC by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

Continuing Calibration Verification (CCV): After use, the instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter.

5.5.1 Calibration Checks

Calibration checks are conducted by measuring a known standard. They must be completed after calibration and should be performed at least one other time (i.e., after lunch) and anytime suspect measurements are encountered. Table 2 provides general acceptance ranges to be used during calibration checks. If a meter is found to be outside of the acceptance range, the meter **must** be recalibrated. If the meter remains out of range, the project manager and/or the supplier of the meter should be contacted to determine alternative measures.

Table 2
Calibration Check Acceptance Limits

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

Notes:
mg/L = milligrams per liter
mv = millivolts
NTU = nephelometric turbidity units

5.5.2 Possible and Suspected Ranges

The concentration for each parameter range should be known so that concentrations outside of the range can be noted. Table 3 presents the maximum range of the parameter in groundwater. The table also presents the suspected range. Measurements outside of the maximum/minimum range should be considered in error and the measurement method should be checked. Concentrations outside the normal range should be treated as suspect but may be the result of contaminant impact. For example, a pH of 2.0 would be out of the normally suspected range for groundwater but not at a site impacted with an acid.

Table 3
Minimum and Maximum Result Ranges

Parameter	Units	Possible Min	Possible Max	Normal Min	Normal Max	Notes
Dissolved Oxygen	mg/L	0.0	14.6 (0°C) 10.1 (15°C) 8.3 (2°C)	0.0	5	The colder the sample, the higher the DO reading. DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color. DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
pH	SU	0	14	5	9	pH values exceeding 10 could indicate grout contamination
ORP	mv					DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color. DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
Specific Conductance	µS/cm			varies	varies	
Temperature	°C	0	100	5	30	
Turbidity	NTU	0	Greater than 1,000	0	Greater than 1,000	50 NTU or greater suggests cloudiness.

Notes:

mg/L = milligrams per liter
°C = degrees Celsius
DO = dissolved oxygen
SU = standard units
ORP = oxidation reduction potential
mv = millivolts
mS/cm = micro Siemens per cm
NTU = nephelometric turbidity units

5.5.3 Field Instruments and Calibration Criteria

The calibration acceptance criteria for each instrument are summarized in Table 4 along with special considerations related to each field instrument.

Table 4
Calibration Check Acceptance Limits

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility.
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature.
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

Notes:
mg/L = milligrams per liter
mv = millivolts
NTU = nephelometric turbidity units

pH Meters

- For the most accurate of pH measurements, pH meters should receive a three-point calibration. However, if a two-point calibration will bracket the groundwater pH of the site, a two-point calibration is acceptable. Three-point calibrations typically include calibrating to solutions of pH 7.00, 4.00, and 10.00. If groundwater pH is outside the calibration range of the solution standards, special buffers must be ordered to bracket the pH. Some meters will report the slope of the calibration and this may be used in checking the meter calibration (refer to the meter's manual). When performing an ICV, the result must be within +/- 0.2 pH units of the stated buffer value.

- pH meters should be calibrated across the range of values to be measured. The maximum and minimum calibration solutions shall be outside the range of anticipated values. For example, if the expected range is between 7.50 and 9.00, the 7.00 and the 10.00 standard should be used for calibration. Perform the IC using at least two buffers, and always use the pH 7.00 buffer first. A reading that is above the maximum (or below the minimum) calibration standard is an estimate only and is not valid. This condition requires obtaining a new standard that is above (or below) the reported value, depending on the measurement.

- A percent slope of less than 90 percent indicates a bad electrode that must be changed or repaired. If percent slope cannot be determined, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

Specific Conductivity Meters

- For IC, when the sample measurements are expected to be 100 microsiemens per centimeter (µS/cm) or greater, use two standard potassium chloride (KCl) solutions that bracket the range of expected sample conductivities. Calibrate the instrument with the first standard. Verify the calibration of the instrument with the second standard, bracketing the range of expected sample values.

- If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values.

- When the sample measurements are expected to be less than 100 µS/cm, a lower bracket is not required, but one standard (KCl) solution that is within the range of expected measurements must be used for the IC and the ICV.

- Accept the calibration if the meter reads within +/- 5 percent of the value of any calibration standard used to verify the calibration.

- Most field instruments read conductivity directly. Record all readings and calculations in the calibration records.

- For CCV, check the meter with at least one KCl standard with a specific conductance in the range of conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5 percent of the standard value.

- If new environmental samples are encountered outside the range of the IC, verify the instrument calibration with two standards bracketing the range of sample values. If these calibration verifications fail, recalibrate the instrument.

Dissolved Oxygen Meters

- Before calibrating, check the probe membrane for bubbles, tears, or wrinkles. These conditions require replacement of the membrane in accordance with the manufacturer's directions.

- If the meter provides readings that are off-scale, will not calibrate, or drift, check the leads, contacts, etc., for corrosion and/or short circuits. These conditions require replacement maintenance in accordance with the manufacturer's directions.
- Most DO meters must be calibrated based on an environment of 100 percent humidity and a known elevation and barometric pressure (BP).

- For 100 percent humidity, place the probe in the calibration container with a moist towel and allow the probe to remain, undisturbed, for 10 to 20 minutes.

- The IC is an air calibration at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument. Allow an appropriate warm up period before IC. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100 percent humidity). Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table (see Attachment 2) what DO should measure. The acceptance criterion for DO ICV is +/- 0.3 mg/L.

- Use the same procedure as above for CCV.

ORP Meters

- Verify electrode response before use in the field.

- Equilibrate the standard solution to the temperature of the sample. The standard solution is based on a 25°C temperature; however, the calibration solution standard's value will require adjustment based on the temperature.

- Immerse the electrodes and gently stir the standard solution in a beaker (or flow cell). Turn the meter on, placing the function switch in the millivolt (mv) mode.

- Let the electrode equilibrate and record the reading to the nearest millivolt. The reading must be within ±10 mv from the theoretical redox standard value at that temperature. If not, determine the problem and correct it before proceeding. Switch to temperature display and read the value.

- Record the mv reading and temperature in the field notebook or in form. Rinse the electrode with distilled water and proceed with the sample measurement, unless using a flow cell. If a flow cell is used, rinse between sample locations.

Turbidity Meters

- Perform an initial calibration using at least two primary standards.

- If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard.

- Perform an ICV by reading at least one primary standard as a sample. The acceptance criterion for the ICV depends on the range of turbidity of the standard value:

    1. Standard Value = 0.1 to 10 NTU: the response must be within 10 percent of the standard;

    2. Standard Value = 11 to 40 NTU: the response must be within 8 percent of the standard;

    3. Standard Value = 41 to 100 NTU: the response must be within 6.5 percent of the standard; and

    4. Standard Value greater than 100 NTU: the response must be within 5 percent of the standard.

- Determining the Values of Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards may be used for CCVs.

To initially determine the value of a secondary standard, assign the value that is determined immediately after an ICV or verification with primary standards. This is done by reading the secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10 percent of the assigned standard value. If the +/- 10 percent criterion is not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

- CCV: Perform a CCV using at least one primary or secondary standard. The calibration acceptance criteria are the same as those for an ICV.

5.6 Direct Measurements

Direct measurements with meters are the most common methods and can be accomplished by placing a sample in a container with the probe or by allowing the water to flow past the probe in a flow cell. The use of a flow-through cell improves measurement quality by allowing the constant flow of water over the probes and reduces interaction of the sample with the atmosphere. Sample cups should be avoided. The quantity of samples, timing, and methodology should be described in the project SAP.

Following calibration of required probes, connect the bottom flow-cell port to the discharge line of the pump. Connect the top port to a discharge line directed to a bucket to collect the purge water. Allow the flow cell to completely fill. As the water flows over the probe, record the measurements. Continue to record the measurements at regular intervals, as specified in the SAP.

When the ambient air temperatures are much higher or lower than the temperature of the water sample, it is best to keep the length of tubing between the wellhead and the flow cell as short as possible to prevent heating or cooling of the water. Tubing and flow-through cell should not be exposed to direct sunlight, particularly in the summer, if at all possible, to avoid heating of water samples.

5.7 Data Acquisitions, Calculations, and Data Reduction

5.7.1 Specific Conductivity Correction Factions

If the meter does not automatically correct for temperature (i.e., read Specific Conductivity) record Conductivity and adjust for temperature upon returning to the office. The following equation can be used to convert Conductivity to Specific Conductivity.

$$K = \frac{(Km)(C))}{1 + 0.0191(\mathrm{T} - 25)}$$

Where:

K = Conductivity in μmhos/cm at 25°C

Km = Measured conductivity in μmhos/cm at T degrees Celsius

C = Cell constant

T = Measured temperature of the sample in degrees Celsius;

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(Km)}{1 + 0.0191(\mathrm{T} - 25)}$$

5.7.2 Percentage Difference Calculation

For evaluating slope of readings from either a flow cell or a sample cup.

$$\%Difference = \frac{(Highest\ Value - Lowest\ Value)}{(Highest\ Value)}\ x\ 100$$

5.7.3 Convert mm mercury (mmHG) to inches mercury (inHG)

$$mmHG = inHG\ x\ 25.4$$

5.7.4 True Barometric Pressure

For converting BP obtained from a public domain source that is expressed in BP at sea level to BP at the subject site.

$$TrueBP = (BP) - \frac{(2.5\ x\ [Local\ Altitude])}{100}$$

Where: BP is in mmHG and Local Altitude is in feet

Example: BP at site A is 30.49 inHg and elevation is 544 feet, calculate TrueBP

Convert inHG to mmgHG:

mmHg = 30.49 inHg x 25.4 = 774.4 mmHg

Calculate True BP:

TrueBP = (774.4 mmHg) – [2.5 * (544 /100)] = 774.4-13.6 = 760.8 mmHg

6.0 RECORDS

Data will be recorded promptly, legibly, and in indelible ink on the appropriate logbooks and forms. At the completion of a field effort, all logbooks, field data forms, and calibration logs shall be scanned and made electronically available to the project team. The original field forms, calibrations logs, and log book will be maintained in the project file.

7.0 HEALTH AND SAFETY

Detailed Health and Safety requirements can be found in the site specific Health and Safety Plan. Ensure that a Safe Work Assessment and Permit form is filled out daily prior to any work in the field and reviewed with all project personnel in a daily safety brief.

Safety glasses with side shields or goggles and disposable gloves shall be worn during calibration activities.

8.0 REFERENCES

None

9.0 ATTACHMENTS

Attachment 1: Example Field Instrument Calibration Form
Attachment 2: Solubility of Oxygen at Given Temperatures
Attachment 3: Example Field Data Form

Author	Reviewer	Revisions (Technical or Editorial)
Resolution Consultants		Rev 0
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 – Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Attachment 1
Example Field Instrument Calibration Form

Field Instrument Calibration Form

Calibrated by: _____ Equipment (Make/Model/Serial#): _____

Date: _____ Equipment (Make/Model/Serial#): _____

pH (su) — Standard: ± 0.2 standard units

Initial Calibration / Initial Calibration Verification

	Hach SL	Reading	Pine SL	Reading
pH7				
pH4				

Continuing Calibration Verification

	Hach SL	Reading	Deviation	Acceptable Variance (Y/N)
pH7				
pH4				

DO (mg/L) — Standard: ± 0.3 mg/L of theoretical*

IC (Temp:) / **ICV (Temp:)**

Saturation (%)	Reading (%)	Theoretical (mg/L)	Reading (mg/L)
100			

CCV (Temp:)

Saturation (%)	Reading (%)	Deviation	Acceptable Variance (Y/N)
100			

Theoretical (mg/L)	Reading (mg/L)	Deviation	Acceptable Variance (Y/N)

ORP (mV) — Standard: NA

IC (Zobell SL:) / **ICV (Pine SL:)**

TCS (Std/Temp)	Reading	TCS (Std/Temp)	Reading

CCV (Zobell SL:)

TCS (Std/Temp)	Reading	Deviation	Acceptable Variance (Y/N)

Turbidity (ntu) — Standard: ±10% of Standard

Initial Calibration

Standard	Reading

Continuing Calibration Verification

Standard	Reading	Deviation	Acceptable Variance (Y/N)

Conductivity (ms^{C}/cm) — Standard: ± 5% of standard value

IC (YSI SL:) / **ICV (Pine SL:)**

Standard	Reading	Standard	Reading

CCV (YSI SL:)

Standard	Reading	Deviation	Acceptable Variance (Y/N)

Comments:

Notes:

SL	solution lot		su	standard units		ntu	Nephelometric Turbidity Units
TCS	temperature corrected standard		mV	millivolts		°C	degrees Celsius
Std	standard		%	percent		ms^{C}/cm	millisiemens per centimeter (temperature corrected)
Temp	temperature		mg/L	milligrams per liter		*	Theoretical value

Attachment 2
Solubility of Oxygen at Given Temperatures

Field Measurement of Dissolved Oxygen

Solubility of Oxygen in Water at Atmospheric Pressure			
Temperature	**Oxygen Solubility**	**Temperature**	**Oxygen Solubility**
°C	**mg/L**	**°C**	**mg/L**
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

Notes:

The table provides three decimals to aid interpolation

Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water saturated

°C = degrees Celsius

mg/L = milligrams per liter

Attachment 3
Example Field Data Form

WELL DEVELOPMENT & GROUNDWATER SAMPLING FORM

DATE:	JOB NUMBER:	EQUIPMENT (Make/Model #/Serial #):
PROJECT:	EVENT:	/ /
WELL ID:	LOCATION:	/ /
WEATHER CONDITIONS:	AMBIENT TEMP:	/ /
REVIEWED BY:	PERSONNEL:	/ /

WELL DIA:	**WELL DEVELOPMENT**	
TOTAL DEPTH from TOC (ft.):	START:	FINISH:
DEPTH TO WATER from TOC (ft.):	VOLUME PURGED (gal):	
LENGTH OF WATER COL. (ft.):	**GROUNDWATER SAMPLING**	
1 VOLUME OF WATER (gal):	START:	FINISH:
3 VOLUMES OF WATER (gal):	VOLUME PURGED (gal):	
	ANALYSIS:	

WELL DEVELOPMENT PARAMETERS		GW SAMPLING PARAMETERS	
Temperature:	± 1.0° C	Temperature:	± 0.2° C
pH:	± 0.5 standard units	pH:	± 0.2 standard units
Specific Conductance:	± 10% of the past measurement	Specific Conductance:	± 5% of the past measurement
Turbidity:	relatively stable	DO:	≤ 20% saturation
		ORP:	± 10 millivolts
		Turbidity:	≤ 10 NTU

IN-SITU TESTING

Circle one: DEVELOPMENT SAMPLING							☐ Bailer ☐ Pump		Description:			
Time (hh:mm):												
pH (units):												
Conductivity (mS/cm):												
Turbidity (NTU):												
DO (mg/L): YSI 556												
DO (mg/L): YSI 550												
Temperature (C°):												
ORP (mV):												
Volume Purged (gal):												
Depth to Water (ft):												
								Well Goes Dry While Purging ☐				

SAMPLE DATA

☐ Bailer ☐ Pump Description:

Sample ID	Date (m/d/y)	Time (hh:mm)	Bottles (total to lab)	Filtered (0.45 µm)	Remarks
Purging/Sampling Device Decon Process:					

COMMENTS:

Purge water placed in drum#_____

In-Situ Hydraulic Conductivity Testing via Rising or Falling Head Slug Testing

Procedure 3-35

1.0 Purpose and Scope

1.1 This Standard Operating Procedure (SOP) describes the methods used to obtain in-situ hydraulic conductivity test data via rising or falling head slug testing (also commonly referred to as variable head testing). Slug tests are performed to assess the hydraulic conductivity of the soil or rock surrounding the tested monitoring well. Hydraulic conductivity is a measure of the ease of flow of water through a specific porous medium or fracture when subjected to a hydraulic gradient.

Hydraulic conductivity values are used:

- To estimate rates of groundwater flow;
- To estimate responses of aquifers to applied stresses, such as pumping;
- To estimate the rate of movement of various chemicals in subsurface zones; and
- To construct and calibrate groundwater flow models.

Specific information regarding the slug testing scope of work can be found in the associated Sampling and Analysis Plan (SAP).

1.2 The purpose of this SOP is to provide a description of a specific method or procedure to obtain in-situ hydraulic conductivity test results. In-situ hydraulic conductivity tests can be conducted in open boreholes or in monitoring wells and they can be performed using constant head or variable head test (i.e., slug test) methods. During a constant head test, water levels are maintained at a pre-determined level (relative to static conditions) and the groundwater flow is monitored. During a variable head test (slug test), as it applies to this SOP, a sudden (instantaneous) rising or falling of the static water level in a borehole or monitoring well is produced by injecting or withdrawing a volume or slug of water or solid cylinder. Water levels are monitored and recorded until the water level has returned to static conditions or sufficient data is collected to perform the hydraulic conductivity calculations. The change in water levels can be produced by displacing a known volume of water using a slug. The response of water levels to the test can be monitored using a water level tape or with computerized data loggers. Data loggers are preferred because they can collect many measurements in a short period of time, which is important for evaluating the early-time response of the aquifer to the slug. For the purpose of this SOP and the field program outlined in the SAP, the method to perform a variable head (slug test) in a monitoring well using a computerized data logger will be outlined.

1.3 This procedure is the Program-approved professional guidance for work performed by AECOM.

1.4 As guidance for specific activities, this procedure does not obviate the need for professional judgment or state-specific requirements. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

1.5 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to performing any hydraulic conductivity tests at the first location. All **field testing personnel** responsible for performing any hydraulic conductivity test activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during hydraulic conductivity testing activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 Hydraulic conductivity testing may involve chemical hazards associated with exposure to groundwater and the testing equipment that comes in contact with the groundwater. When conducting hydraulic conductivity tests, adequate health and safety measures must be taken to protect field personnel. These measures are addressed in the project HASP. All work will be conducted in accordance with the HASP.

3.0 Terms and Definitions

None.

4.0 Interferences

Many potential interferences can occur during the performance and analysis of slug tests. For this reason, appropriately trained personnel shall perform the tests in the field and conduct the data analysis. Data and analysis will be reviewed by a professional geologist, or other qualified professional in accordance with state-specific requirements.

Well construction is a common cause of physical slug test interference. For wells screened across the water table where an unsaturated zone of soil is exposed, falling head slug tests (i.e., the instantaneous rise in static groundwater levels) may not provide accurate measures of hydraulic conductivity. While falling head slug tests may be conducted in such wells, the results will not be used quantitatively unless the data analysis indicates that an accurate measurement of hydraulic conductivity has been obtained.

5.0 Training and Qualifications

5.1 Qualifications and Training

5.1.1 The individual(s) executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that the hydraulic conductivity testing activities comply with this procedure. The CTO Manager or designee shall review all hydraulic conductivity forms prior to use. The CTO Manager is responsible for ensuring that all field personnel

involved in hydraulic conductivity testing and data analysis shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all field personnel involved in hydraulic conductivity testing follow these procedures.

5.2.4 **Field testing personnel** are responsible for the implementation of this procedure. Minimum qualifications for field testing personnel require that one individual on the field team shall should be familiar with the theory and practice of slug testing and analysis, as well as with all necessary equipment and field software. Geologists, hydrogeologist, other personnel with geologic or hydrogeologic experience, or other qualifications based on state-specific requirements, should supervise hydraulic conductivity testing.

5.2.5 **Data analysis personnel** are responsible for the review of data associated with this procedure. Minimum qualifications for data analysis personnel require the individual be familiar with the theory and practice of slug testing and analysis, as well as with all necessary software. Geologists, hydrogeologist, other personnel with geologic or hydrogeologic experience, or other qualifications based on state-specific requirements, should review the hydraulic conductivity test results and data interpretation.

5.2.6 The **field testing personnel and/or task manager** is responsible for directly supervising the hydraulic conductivity field testing to ensure that the testing is conducted in accordance with this procedure and state-specific requirements, should they apply, and for recording all pertinent data collected during testing. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the Program Quality Manager and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

General field supplies to perform the slug testing include the following items:

- Boring logs (if available)
- Well construction diagrams (if available)
- Well development logs (if available)
- Water level meter
- Slug (bailer or solid cylinder)
- Nylon string
- Water level indicator
- Pressure transducer(s)
- Data logger(s)
- Computer with appropriate software
- Plastic sheeting
- Equipment decontamination materials (as required by Resolution Consultants SOP No. 3-06 – Equipment Decontamination)
- Health and safety supplies (as required by the HASP)
- Approved plans (e.g., HASP, SAP, QAPP)
- Field project logbook/pen

7.0 Procedure

7.1 General Preparation

The boreholes or monitoring wells to be tested should have been previously developed and had sufficient time to equilibrate before the testing process proceeds (minimum of one week). Well construction diagrams are necessary to determine the depth of the monitoring well and the screened interval.

All equipment that will come into contact with the groundwater will be decontaminated and tested prior to the start of field activities. Any sharp edges of the well casing will be covered with duct tape to protect transducer cables.

An initial round of static water level measurements will be collected prior to initiating the slug test. Wells will be gauged (and subsequently tested) from least contaminated to most contaminated, if known/possible. The static water level in each well will be measured and recorded in the field logbook or field form.

The slug diameter and length to be used in the monitoring well will be determined based on the diameter of the well and the length of the water column. In general, the larger the volume of the slug, the greater the displacement of head and the better the definition of response in the resulting data. However, the slug must be short enough to be completely submerged beneath the static water level, and there must be room beneath the bottom of the slug for the transducer. The transducer and cable should be installed in the well at least two feet from the bottom of the well and be held in place using duct tape to keep the transducer at a constant depth. Therefore, a minimum of seven feet of water within the monitoring well is typically necessary for conducting the test. The transducer will then be connected to the data-logging device and the initial water level recorded. The slug length and diameter will be recorded in the field logbook or field form for use in the data analysis.

Either a pressure transducer connected to a data logger or a programmable down-hole data logger will be used to record the changes in water level during the test. The transducer must be set at least one slug length below the water surface so the slug does not disturb the transducer; several feet deeper is preferable, if possible. After the water level has equilibrated to the static level, the data logger will be programmed according to manufacture's instructions. The data logger will be programmed to record water levels at logarithmically increasing intervals, because early-response data is important for the data analysis.

A measured length of nylon string will be tied to the slug. The line will be of a length that will allow the top of the slug to be submerged beneath the static water level without touching the transducer.

7.2 Falling Head Test

The slug will be lowered part way into the well so that the bottom of the slug is just above the water surface. The data logger will be started and the slug will be simultaneously lowered into the water, so that the top of the slug is below the static water level. Care will be taken to lower the slug fast enough to produce as close to an instantaneous rise in the water level as possible, but not so fast as to produce a wave when the slug enters the water.

When the water level returns to the static level, the falling head test is complete and the rising head test can be started. The data should be saved and a new test set up on the data logger.

If the hydraulic conductivity is low, it may take hours (or more) for the water level to return to static conditions. In this situation, the Field Manager should determine a maximum duration for each test (typically 30 minutes).

The falling head test may not be accurate for wells with screens that bracket the stabilized water table. These tests may be performed in water table wells, but the results will not be used quantitatively unless the data analysis indicates that an accurate measurement of hydraulic conductivity has been obtained.

7.3 Rising Head Test

After the data logger is reset following the falling head test, the test will be started by activating the logger and simultaneously removing the slug. The slug will be quickly removed from the water so that an instantaneous drop in the water level will occur, but it will be done smoothly enough to not disturb the transducer when removing the slug. When the water has returned to a static condition or the maximum duration of time has elapsed (typically 30 minutes), the test will be terminated.

7.4 Data Download

At the completion of the test(s), the data from the slug tests will be downloaded to a laptop computer. If feasible, this data will be plotted on a graph of time versus water level to see if it is acceptable (i.e., adequate water level displacement, sufficient number of data points, a straight-line fit to data, no extraneous fluctuations resulting from inadvertent slug movement and/or pressure waves). If the data are not acceptable, the test(s) will be repeated once water levels have stabilized.

7.5 Equipment Decontamination

All equipment that comes into contact with groundwater (e.g., slugs, transducer, and water level meter) will be decontaminated in accordance with Resolution Consultants SOP No. 3-06 - Equipment Decontamination before moving to the next location. The string should be properly discarded and disposed of.

8.0 QUALITY CONTROL AND QUALITY ASSURANCE

Quality assurance requirements typically suggest the collection of both the rising head and falling head data. Rising head data are preferred for wells screened across the water table because the hydraulic conductivity of the saturated portion of the aquifer is reflected, whereas falling head data may reflect the hydraulic conductivity of the unsaturated zone and capillary fringe.

For quality control purposes, the transducer data logging will be started immediately prior to lowering or removing the slug. The transducer should be activated approximately 1 second prior to ensure that the transducer is recording water level changes and that the transducer is taking readings at frequent intervals during the early part of the well response curve. Care must be taken when lowering or removing the slug to avoid splashing or generating wave effects that would obscure the early-time data.

Data from the rising and falling head tests should be inspected in graphical format to ensure that adequate water level displacement was achieved for both tests, that the data logger recorded all data from the test, and that no fluctuations exist in the data due to violent slug movement.

Both rising and falling head tests may be conducted to help draw attention to any inconsistencies in the data. If the rising and falling head results are not comparable, the data should be investigated to assess which test may more accurately reflect the hydraulic properties of the aquifer, or whether the pressure transducer recorded accurate data. Knowledge of the boring logs is essential to assessing whether the measured response is consistent with the expected response. If a consistent response is not obtained, the tests should be run again.

If applicable per state-specific requirements, the work performed under this SOP will be conducted under the direction of and reviewed by the required trained, certified or licensed personnel.

9.0 Data and Records Management

9.1 Records Management

Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. A field logbook will be maintained to ensure that adequate documentation is made of hydraulic conductivity testing activities. Information, such as background information such as well diameter, depth, and screened interval, will be documented from existing monitoring well construction

logs in the field logbook or other site-specific test log, if applicable. These data will be used in the calculation of hydraulic conductivity, based on the test data. The slug length, slug diameter, water level, well identification number, test type (rising or falling), file name, and time will also be recorded. The actual data of time versus water level will be recorded on the data logger and transferred to a laptop computer. In addition, any problems or unusual conditions that may have occurred during the testing process will also be recorded.

Software files associated with the hydraulic conductivity testing activities will be saved in the associated project files upon completion of the field activities.

The records generated in this procedure will become part of the permanent record supporting the associated fieldwork. All documentation will be retained in the project files following project completion.

The field logbook is kept as a general log of activities and should not be used in place of any site-specific test log, if applicable.

Unanticipated changes to the procedures or materials described in this SOP (deviations) will be appropriately documented in the project records.

Records associated with the activities described in this SOP will be maintained according to the document management policy for the project.

9.2 Data Analysis

Several methods are available for analyzing data obtained from in-situ hydraulic conductivity tests. Most methods incorporate graphical techniques, such as semi-log and log-log plots, to evaluate the data and select values for the calculations.

Inherent in the analytical methods are several simplifying assumptions concerning the aquifer properties and test methods. When selecting a particular analytical method, it is important to consider the basic assumptions that underlie the mathematical expressions. In many cases, it may be advisable to evaluate the data using several methods and examine the range of hydraulic conductivity values that are obtained. For this project, it is expected that the data will be analyzed using appropriate methodologies (e.g., Bouwer and Rice, 1976; Kansas Geological Survey methods [Hyder, et al., 1994]; Cooper, et al, 1967).

10.0 Attachments and References

10.1 Attachments

SOP 3-03, *Recordkeeping, Sample Labelling, and Chain-of-Custody*

SOP 3-06, *Equipment Decontamination*

10.2 References

Bouwer, H and RC Rice. 1976. A slug test for determining hydraulic conductivity of unconfined aquifers with completely or partially penetrating wells. Water Resources Research, Vol. 12, pp. 423-428.

Cooper, H Jr., D Bredehoeft, and S Papadopulos. 1967. Response of a Finite-Diameter Well to an Instantaneous Charge of Water. Water Resources Research, Vol. 3, No. 1.

Hyder, Z, JJ Butler, Jr, CD McElwee, and W Liu. 1994. Slug tests in partially penetrating wells. Water Resources Research, Vol. 30, No. 11: 2945-2957.

USEPA SOP#2046, Slug Tests, October 3, 1994.

Author	Reviewer	Revisions (Technical or Editorial)
Naomi Ouellette, Environmental Scientist	Lauren Roberts, Environmental Scientist	Rev 0 – Initial Issue (November 2012)
Amanda Martin Environmental Scientist	Mark Kauffman Program Manager	Rev 1 - Editorial change only; converted to AECOM SOP for use on USACE HTRW projects (April 2017)

Pore Water Sampling

Procedure 3-45

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for use in sampling pore water. This SOP describes the equipment, field procedures, materials, and documentation procedures necessary for collection of pore water samples. Specific information regarding sample locations can be found in the associated Quality Assurance Project Plan (QAPP).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under contract to the United States Army Corps of Engineers (USACE).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific QAPP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including physical, chemical, and biological hazards are addressed in the project Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). The major health and safety considerations for the work associated with pore water sample collection are the near and on-water safety aspects of the program.

2.2 Daily safety briefs should be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or his/her designee to discuss the day's events and any potential health risks of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP and SSHP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are remedied to the satisfaction of the SSHO.

3.0 Terms and Definitions

3.1 **Global Positioning System (GPS)**

A system of satellites, computers, and receivers that is able to determine the latitude and longitude of a receiver on Earth by calculating the time difference for signals from different satellites to reach the receiver. Interferences

4.0 Interferences

4.1 Cross-contamination of samples may result if sampling equipment is inadequately or improperly decontaminated.

4.2 Contamination of samples may result if samples are exposed to certain environmental conditions. Exposure to potential sources of contamination (e.g., exhaust fumes) should be minimized.

4.3 Care must be taken to avoid surface water intrusion during sampling. Water will flow in a path of least resistance. If space is created around the sides of the sampling end of the pore water device during deployment, surface water may flow down the outside of the device to the screened area and into the intended sample. The pore water device should be used with a sampling flange, especially when collecting pore water near the sediment-surface water interface.

4.4 Inappropriate sampling equipment, such as that manufactured from non-inert plastics, may contaminate samples. Using Teflon, polymer, or stainless steel sampling equipment will minimize contamination during sample collection activities.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The **Project Manager** is responsible for ensuring that pore water sampling activities comply with this procedure. The Project Manager or designee shall review all pore water sampling forms on a minimum monthly basis. The Project Manager is responsible for ensuring that all field sampling personnel involved in pore water sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure. Minimum qualifications for field sampling personnel require that one individual on the field team shall have a minimum of 6 months of experience with pore water sampling.

5.2.5 The **field sampling personnel and/or SS** are responsible for directly supervising the pore water sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the Program Quality Manager and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Approved plans, including target sampling locations
- GPS
- Pore water push-point sampler (i.e. Henry Sampler)
- Peristaltic pump with appropriate battery
- Teflon and medical-grade silicon tubing necessary for peristaltic pump
- Syringe

- Multi-parameter data sonde (YSI or equivalent)
- Laboratory supplied sampling containers
- Ziploc plastic bags for samples and sample jars
- Waterproof project-specific field log book, standardized field forms and pens
- Labels
- Chain of Custody (COC) forms
- Boat with all applicable safety equipment (anchor, etc.)
- Cooler(s)
- Ice
- Decontamination supplies/equipment

7.0 Calibration or Standardization

The multiparameter data sonde should be calibrated daily per the manufacturer's instructions.

8.0 Procedure

Pore water samples will be collected and handled in accordance with United Stated Environmental Protection Agency (USEPA) Region 4 SOP for Pore Water Sampling (SESDPROC-513-R2, dated February 28, 2013).

8.1 Flange/Push-Point/Peristaltic Pump/Tubing Set-up

Navigate to the sample location using a GPS. The flange is first placed at the desired sampling point with the push-point removed to allow water to escape from under the flange. The flange rim should be carefully worked into the sediment until the flange is flush with the sediment surface. The pore water push-point device should then be inserted through the compression adapter on the flange and into the sediment as carefully as possible. When the sampler is inserted to the desired depth, the compression adapter should be tightened. If sampling through a significant amount of surface water, the flange should be connected to the push-point sampler prior to deployment such that the correct depth can be sampled for pore water. The flange and push-point sampler set up are shown in Attachment 1.

Connect tubing from push-point sampler through peristaltic pump and connect the pump to an appropriate battery. Sampling set up is shown on Attachment 2. The tubing should be new and dedicated to the sample location. Install clean, silicone tubing in the pump head, per the manufacturer's instructions. Pharmaceutical-grade silicone tubing (e.g., PharMed tubing) may be required for some projects depending on the analyses required. Refer to the project specific QAPP for specific tubing requirements. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into sample bottles but only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing. (Some types of thinner Teflon tubing may be used.).

If the push-point sampler becomes easily clogged such that pore water cannot be extracted via the sampler, a syringe can be used to extract the sample instead of a peristaltic pump.

8.1.1 Sample Collection

In general, the volume of pore water that can be collected at a given location is limited. Collecting large volumes of pore water will ultimately result in the collection of water from the overlying water body. Therefore, a set of field parameters (color, pH, specific conductivity, temperature, turbidity, salinity, oxidation/reduction potential, and dissolved oxygen) from the surface water located immediately above

the sediment/water interface and from the pore water that is initially extracted will be collected prior to sample collection. If pore water volume is limited, dissolved oxygen and conductivity will be the priority field parameters for the pore water. The pore water sample will only be collected if the dissolved oxygen and conductivity in the pore water and overlying surface water are significantly different. If possible, a final set of field parameters will be collected from the pore water after sample collection.

The samples will be numbered and labeled as described in the project specific QAPP. Following collection, samples will be placed in coolers on ice until transport to the laboratory.

Sample collection information will be recorded at the time of collection in the field logbook and on the form in Attachment 3. This information will include, but not be limited to, the station ID, sample ID, time and date of sample collection, sample collection depth, the sampler's name, description of any sample processing, and any pertinent observations. Initial and final pore water and overlying surface water field parameters should also be recorded.

Samples will be stored cold (4 ± 2°C) until they are packaged for transport/shipment to the laboratories. Samples will be packaged in ice and will be hand delivered or shipped to the appropriate laboratories.

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific QAPP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality Control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific QAPP will provide requirements for sample preservation, holding times, container types, as well as various QC samples such as trip blanks, field blanks, equipment blanks, and field duplicates.

10.0 Data and Records Management

10.1 Field notes will be kept during sampling activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. During the completion of sampling activities, fill out the sample logbook and transmit forms to the Project Manager for storage in project files.

10.2 Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

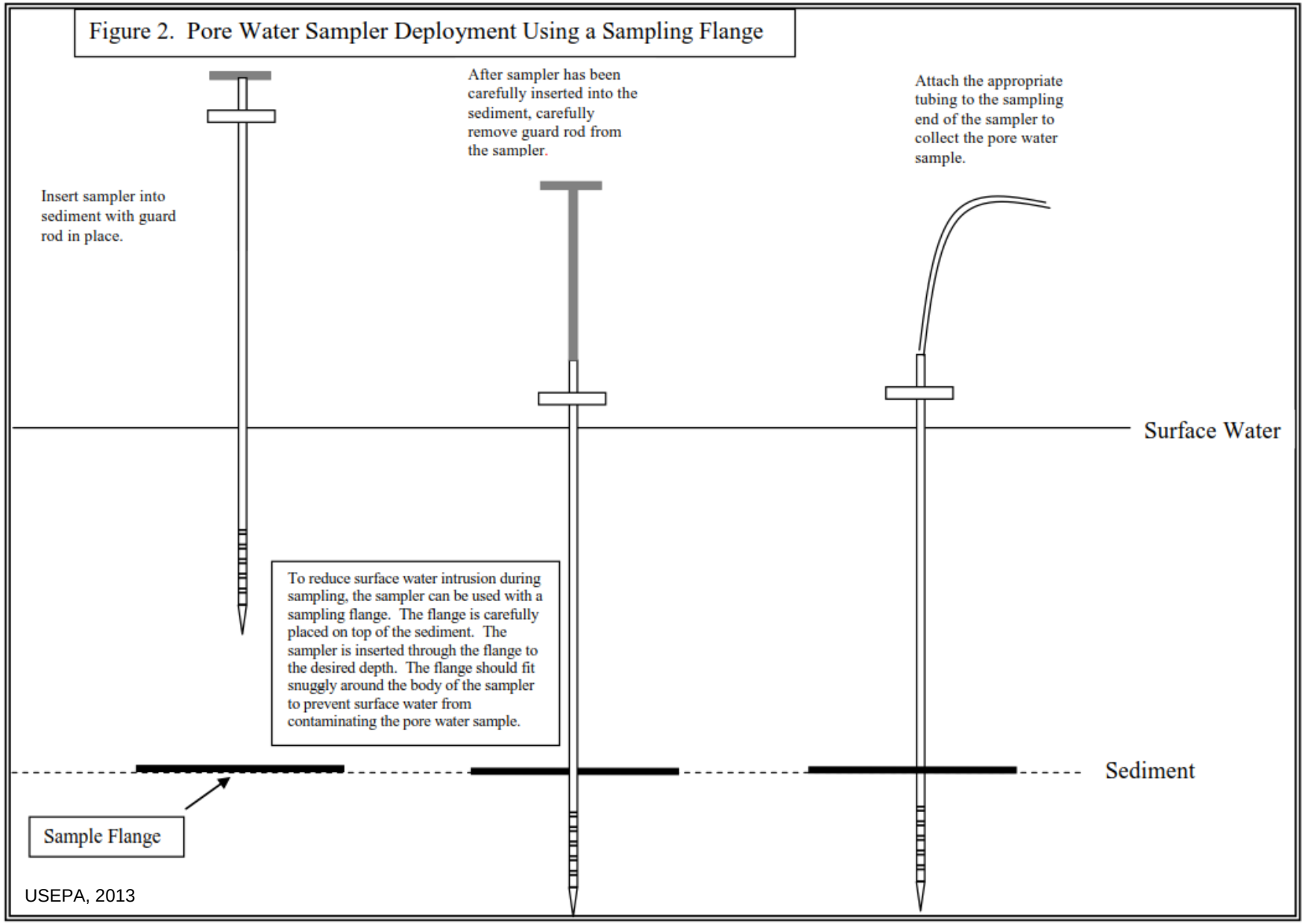
Department of the Navy (DON). 2007. *Navy Environmental and Natural Resources Program Manual*. OPNAV Instruction 5090.1c. October.

United States Environmental Protection Agency (USEPA). 1987. *A Compendium of Superfund Field Operations Methods*. EPA/540/P-87/001, EPA, Office of Emergency and Remedial Response, Washington, D.C.

United States Environmental Protection Agency (USEPA). 2013. *Operating Procedure, Pore Water*. Number SESDPROC-513-R2, EPA, Region 4, Science and Ecosystems Support Division, Athens, GA.

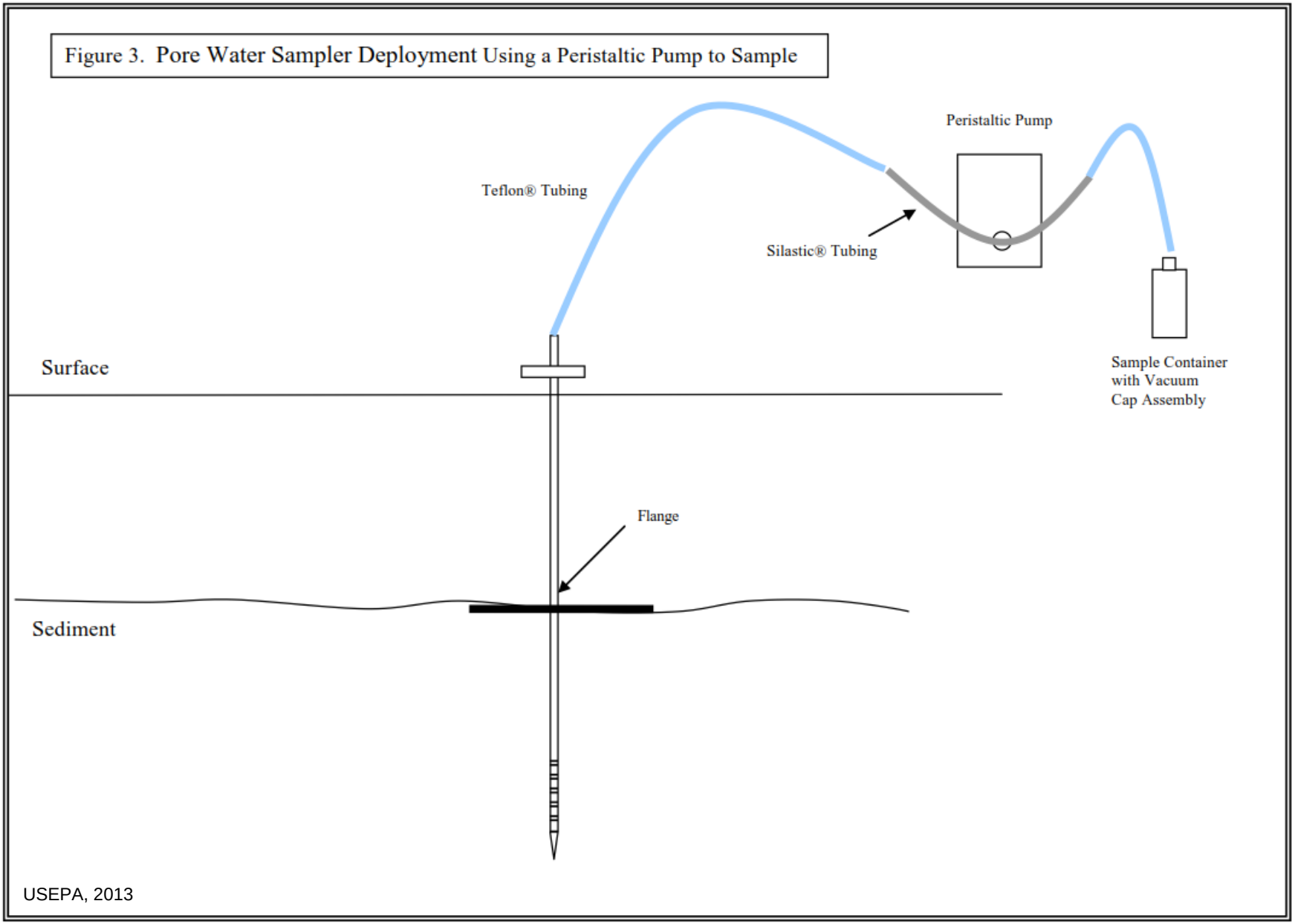
Author	*Reviewer*	*Revisions (Technical or Editorial)*
Helen Jones Senior Scientist	Josh Millard Project Hydrogeologist	Rev 0 – Initial Issue
Amanda Martin Environmental Engineer	Robert Shoemaker Project Manager	Rev 1 – Editorial change only; converted to AECOM SOP for use on USACE projects (September 2019)

Attachment 1 - Pore Water Sampler Deployment



Attachment 2 - Pore Water Sampling Using Peristaltic Pump

Figure 3. Pore Water Sampler Deployment Using a Peristaltic Pump to Sample



USEPA, 2013

Attachment 3 - Pore Water Collection Form

Project Name:				
Date(s):				
Sample Location ID:			Date:	
Sample #:			Time:	
QC (FD or MS/MSD):			Weather:	
GPS Coordinates:				
Samplers:				
Location Information:				
Total Water Depth:			Estimated Current Speed:	
Surface Water Depth:			Porewater Depth:	
Surface Water Field Parameters:				
pH:			Dissolved Oxygen: (mg/l)	
Specific Conductivity: (µmho/cm)			Temperature: (°C)	
ORP: (mv)			Turbidity: (NTU)	
Pore Water Field Parameters (Initial/Final):				
pH:	/		Dissolved Oxygen: (mg/l)	/
Specific Conductivity: (µmho/cm)	/		Temperature: (°C)	/
ORP: (mv)	/		Turbidity: (NTU)	/
Comments/Observations (sheen, odor, other):				
Pore water equipment used (collection equipment and meter make, model, and serial no.):				
Pore water analyses to be performed:				

Appendix D

FLUTe SOPs

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FLUTe Blank Liner Installation and Removal Procedure

(Note: A DVD is available teaching the installation procedure. Call 1-888-333-2433 and request a copy)

Rev. July, 2005

Blank liner installation and removal procedure

(note the starred items may not apply to the simple installation)

The blank liner installation is quite simple when performed in the following sequence:
1. Form the split hose over the top of the casing to protect the liner. Trim the hose to the proper length to cover the entire top edge of the casing. Tape the lower edge of the hose to the casing to hold it in place.
2. Set up the shipping reel on the reel stands with the large axle flange on the side marked "vent" inside the edge of the reel flange*. Make sure the arrow on the protective liner cover points over the top of the reel toward the wellhead. The reel should be about 10 ft from the wellhead.
4. Remove the protective wrapper on the liner. Don't use a sharp knife. Unwrap it.
5. Tag the water level.
6. Assure that the depth of the hole is known and that the hole has not caved, bridged, or otherwise become blocked.
7. Set up the wellhead roller next to the wellhead, well supported (see the attached drawings). Leave room to attach the liner to the casing. Then reposition the roller as shown.
8. Install the air vent tube to the water table in the borehole and make sure that the air vent tube is well tied off with a pipe hitch to prevent its falling down the hole. The air vent tube should be opened with a small scallop of the tube about every 20 ft. to allow easy air flow into the tube. The top of the vent tube should be open to allow the air to flow out of the tube. The tube is usually supplied with the liner. **
9. Cut off the liner about 5 inches above the "GS" mark on the liner (be careful to not cut off the ¼" bubbler tube). Invert about 5 ft of the blank liner as it comes off the shipping reel. (The GS mark will be at the top of the casing.) Slit the liner and the sleeve next to the bubbler tube for about 6 inches.
10. Fold the everted portion of liner lengthwise and slide it down the casing until 5 inches of the top edge is sticking above the casing. Fold the liner over the casing, tape it in place, and clamp the liner to the casing securely. The vent tube must pass through the slit on the liner with the bubbler tube**. The liner is now ready to be installed. The liner is only routed over the large roller for installation, and not under the smaller roller.
11. ***Connect the bubbler tube to the upper right quick connect of the Tagler (bubbler monitor). It is necessary to attach the quick connect fitting to the nut and ferrule on the tube before inserting it into the quick connect on some Taglers. Insert a pressure gauge with a pressure range of about half the depth to the water table in psi (e.g., 30 psi for a 60 ft water table. However, there is no need for a pressure gauge greater than about 30 psi, unless the bubbler is open to the liner far below the water table in the formation.) The bubbler pressure minus 0.433 x (bubbler depth – water table depth) is the excess pressure inside the liner. That should not exceed about 10 psi. The bubbler depth should be equal to or greater than the water table in the formation. The pressure source from the nitrogen bottle (set at about 50 psi) is plugged into the bottom quick connect on the Tagler. The needle valve is set for a flow of about 2 SCFH. Make sure that that flow rate is maintained. (in shallow water table situations, there is usually no need for a bubbler).
12. Add water to the interior of the liner until there is about 5-10 lb of tension on the liner. Then, lower the liner slowly down the hole so as to allow the air to vent from beneath the

liner**. When the liner reaches the water table, the tension will drop to zero as the water slug in the liner descends beneath the water table. If the depth marks on the liner do not correspond to the water table depth, it may be necessary to add more water to cause the liner to descend to the water table. It should not be necessary to fill the liner with more than 10 ft of water to cause it to reach the water table (about 10 gallons for a 5 in. diameter liner). – more water in an angled hole.
13. Once the liner has reached the water table, add water to the interior of the liner, driving the liner down the hole. Note, the bubbler reading will not be correct until the liner has descended to the depth of the bubbler on the liner. (The bubbler pressure on the gauge is the sum of the water height in the liner above the water table in the formation, plus the depth of the bubbler below the water table in the formation. A bubbler is not needed for modest water table depths of about 30 ft.)
14. When the end of the liner comes off the reel, stop the liner. Allow the air trapped in the end of the liner to vent through the check valve on the tether. The liner may need to be squeezed by hand to vent the air. Once the air has been vented from the liner, allow the liner to then continue down the hole. (**Note,** the drawing shows a vacuum pump as used by FLUTe. The pump is not needed for the ordinary installation.)
15. Maintain a modest tension on the liner and then on the tether (about 3-4 lb.) as the liner descends. The tension should be sufficient to prevent the liner from sliding down the hole of its own weight and to be able to easily detect the progress of the eversion as the liner descends.
16. Monitor the bubbler pressure and be careful to not exceed the pressure of 10 psi as calculated in no. 11 above. If one is not adding water, the bubbler pressure will drop as the liner descends. That is a good check that the liner is descending correctly.
17. Let the blank liner descend until its descent velocity has dropped to about 0.1 ft per minute. At that velocity, there is not much remaining transmissivity below the liner. The tether (or the liner wrapped with a kellum strap) should be tied to an anchor that will prevent its further descent. Allowing the liner to descend further will prolong the removal.

Removal of the liner

The removal procedure starts with the wellhead roller situated as shown in the attached drawings. This is the same as the installation geometry, except the tether/liner is routed under the small roller on the back side of the wellhead roller (opposite the well). This routing under the small roller will prevent the roller from tipping over when tension is applied to the tether. The axle of the small roller is removable to allow easier routing of the tether under the small roller.

The wellhead roller must also be anchored to the casing or other secure anchor to prevent the roller from being dragged by the lateral force of the tether as the liner is pulled from the hole.

The procedure is as follows (see the drawings attached):
1. Set up the wellhead roller as shown. Anchor the frame of the roller low to prevent its being dragged away from the wellhead by the tension on the small roller.

2. Set up the winch plate about 50 ft away from the wellhead. Lay out a protective sheet of heavy plastic film between the wellhead roller and the winch to prevent abrasion or thorn/stubble damage to the liner as it is pulled out of the hole.
3. Anchor the winch plate using the cable and the aluminum plate provided. A vehicle can be parked with a tire on the aluminum plate to serve as a good anchor. Make sure that the tire will not be rotated with a 1000 lb force on the cable. A side pull on the tire is more secure for a front wheel.
4. Route the tether as shown and wrap the tether about 4 wraps, clockwise on the capstan of the winch. Then insert the handle and apply a tension to the tether. The tail of the tether, where it comes off the winch, must have some tension on it to prevent slipping on the capstan of the winch. It is usually easier if someone "tails" the tether while someone else cranks the winch. The winch has two directions of travel. The easier direction to turn is geared about 1.6 above the direct gearing of the harder direction. Don't crank too hard on the winch (i.e., the limit of your ability). Just develop a good tension in the tether, which stretches the liner. Then wait as the liner contracts and pulls itself out of the hole. Retighten the tether as needed to remove the liner. As the liner rises in the hole, it will proceed more rapidly as more flow paths are uncovered.
5. In order to pull a blank liner from a hole with a deep water table (>10-15 ft), it is necessary to pump the water out of the liner as the liner rises.
6. **Lower a Grundfos II, or similar pump, to a point <u>no lower</u> than 10-15 ft above the water level in the formation. It is essential that the pump can not pump the water level in the liner lower than that value.** If the liner is over pumped, it may buckle instead of inverting, and become jammed in the hole. That is very awkward and difficult to fix. Turn the pump on while a good tension is applied to the tether, and let the pump run until it stops flowing (i.e., the water level in the liner has dropped to the pump).
7. Increase the tension on the tether. As the liner inverts, the water level in the liner will rise and the pumping can continue. By setting the pump speed to the approximate rate to match the water level rise in the hole as the liner inverts, the pump can run nearly continuously. Wrap the tether neatly onto the shipping reel as the tether comes off the winch. The shipping reel should be positioned between the winch and wellhead, nearer the winch, to later accept the liner as it is pulled out of the hole.
8. **When the liner is <u>at the water table</u>, the pump should be stopped and removed.** This will leave a sufficient amount of water in the liner to allow it to be inverted to the surface. If the pump is not stopped when the liner reaches the water table, there may be insufficient water left in the liner to cause it to invert nicely, and it may again jam in the hole.
9. When the liner follows the tether out of the hole, and over the roller, the liner can be pulled until it reaches the winch and no further. The liner will not go onto the winch. At that time, the Nylon straps provided should be wrapped in the diagonal fashion demonstrated, next to the wellhead roller, so that they operate as a Chinese finger trap or kellum and grip the liner without damaging the coating on the liner. The straps should alternate over and under each other so that they are woven onto the liner. If the straps look like one strap was wrapped on first and the second strap was then wrapped over it for its entire length, it was not properly woven onto the liner.
10. A carabiner is then clipped into the near end of the kellum and connected to a long rope provided to extend to the winch. The tether is then unwrapped from the capstan, and the rope connected to the kellum is wrapped onto the capstan. If the liner has a great deal of tension,

it may be necessary to wrap a kellum onto the liner near the winch, and tie that kellum off to an anchor (the cleat on the winch plate), so that the tether tension can be relaxed without the liner descending back down the hole. Then the rope from the wellhead kellum is wrapped onto the winch capstan.

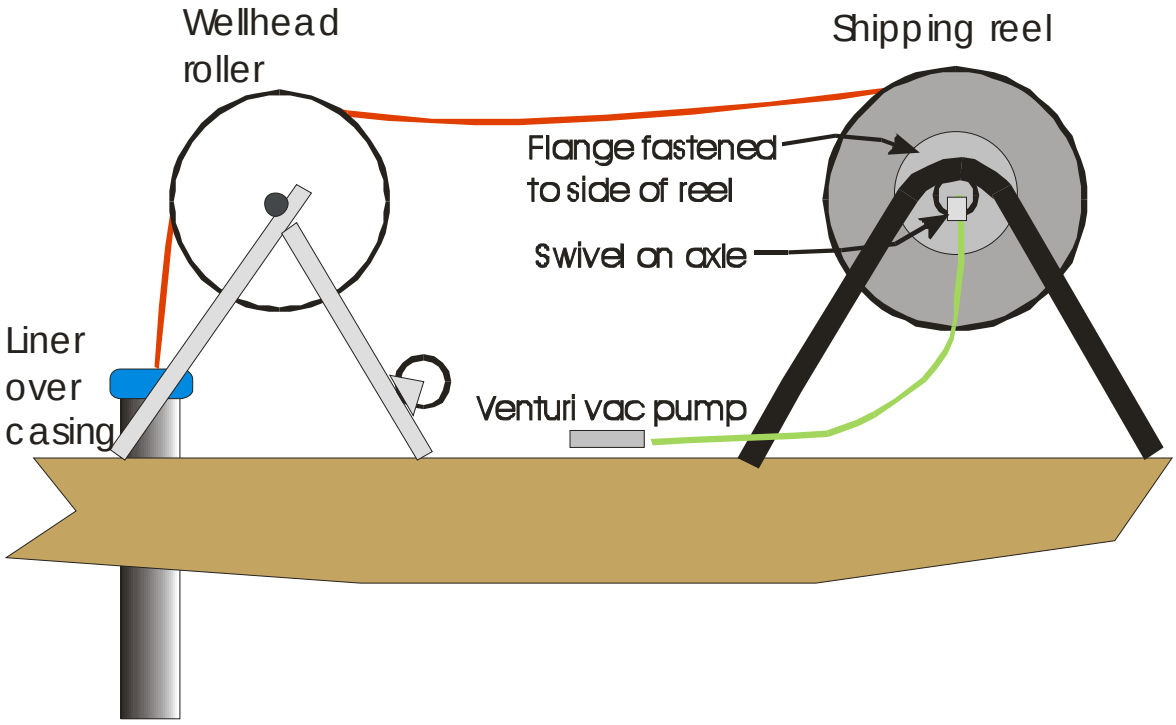
11. As the wellhead kellum is drawn toward the winch, the slack liner is rolled onto the shipping reel as shown. (In fact, the tether should also be wrapped first on the shipping reel as the tether is withdrawn with the winch.) When the kellum nears the winch, a second short rope is tied into the carabiner to anchor the near kellum while the next kellum is attached to the liner near the wellhead. As the tether is unwrapped from the winch, the anchor rope takes up the liner load until the long rope is again wrapped on the winch. (note, only one rope and carabiner is required, since each time that the anchor rope is allowed to take the load, the rope and carabiner can be connected to the kellum at the wellhead.) In this manner, the liner is pulled length after length from the hole. It is obvious that the further the winch is from the wellhead, the less often a kellum must be reattached to the liner.

12. When the liner rises out of the water table in the hole, it can usually be pulled by hand from the borehole by one or more people, saving the time of reattaching kellums. Be sure that the pump is stopped and removed before pulling the liner from the water table. The liner should always be rolled onto the shipping reel as it is pulled from the borehole to prevent damage to the liner and its coating.

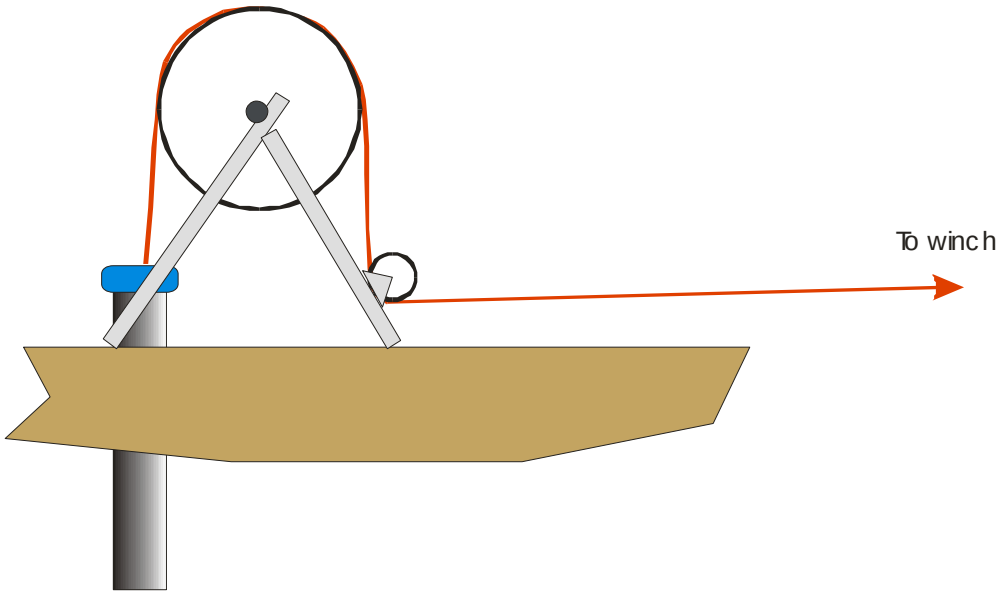
Questions? Call FLUTe at 888-333-2433 or 505-883-4032.

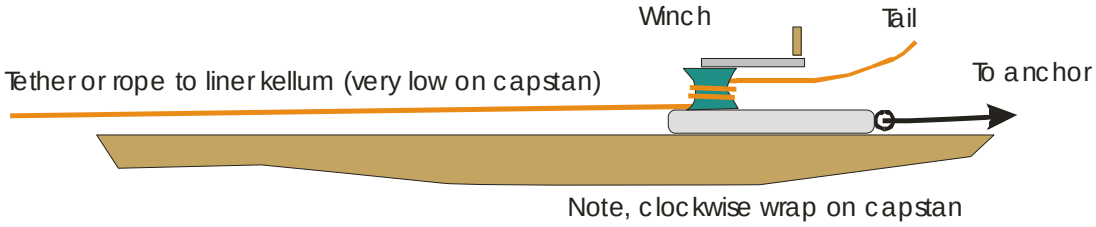
* This detail is only relevant to a reel stand with a vacuum axle. It is not typically used.
** The air vent tube is needed if the casing extends to the water table and there is no flow path in the vadose zone to vent the air trapped beneath the liner.
*** The bubbler tube is a useful monitoring method for the water level in the liner, but much less useful for installations into holes primarily above the water table.

Installation geometry

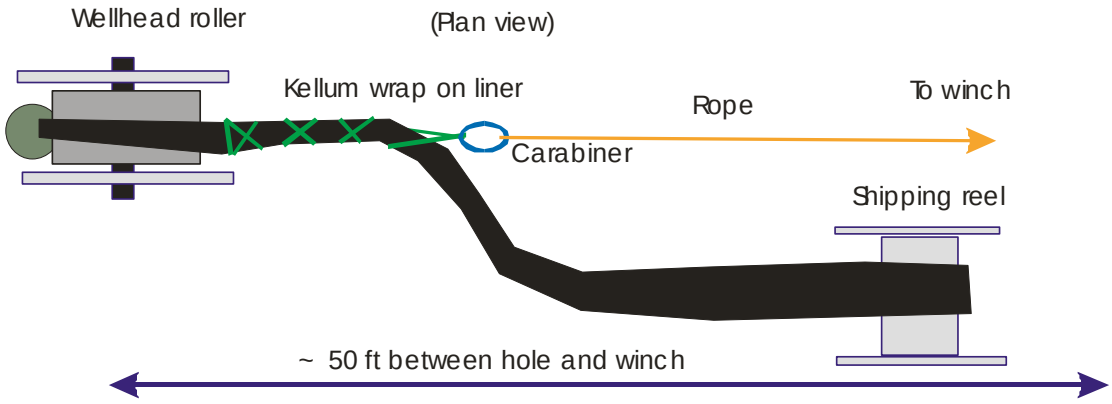


Blank liner removal geometry





Winching geometry for the liner



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Brief Description of Installation Procedure

for

Water FLUTes

Installation procedure for Water FLUTes

Purpose

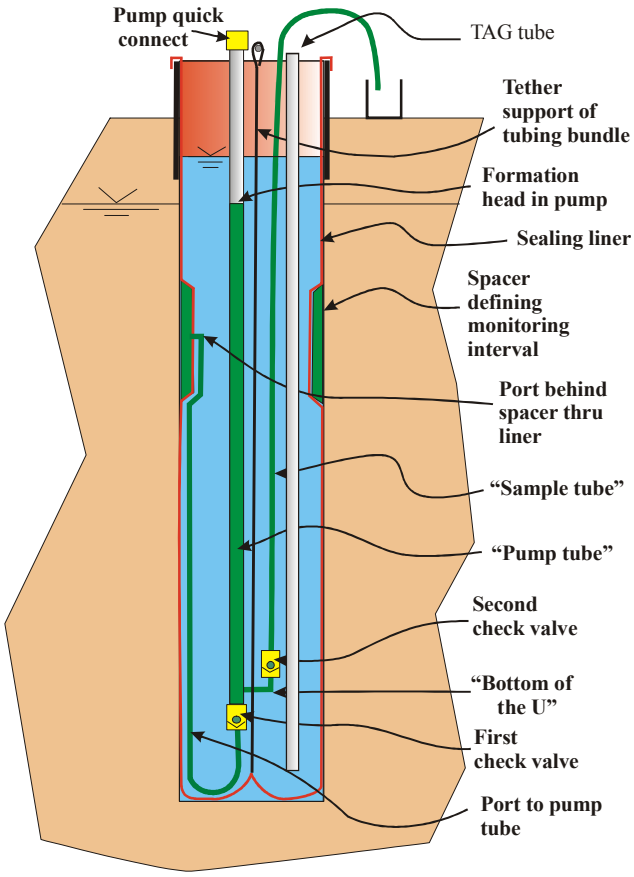
This is intended as a brief general description of the procedure and the equipment used for the Water FLUTe installation method.

The Water FLUTe system

The Water FLUTe system is a multi level ground water sampling system as is described in detail in Cherry, et al[1]. The system consists of a flexible borehole liner composed of a urethane coated nylon fabric with attachments for the purpose of drawing water from the formation and for measurement of the depth of the water table at each sampling interval. Figure 1 depicts the liner as fully installed in a borehole with only one sampling interval shown for clarity. The external annular spacer defines an interval of the borehole that is not sealed by the liner. The ground water sample is drawn from that interval and conducted to the pump system shown in the center of the borehole. The long pump tubing allows a relatively large (~1 gal.) sample to be displaced to the surface by nitrogen gas pressure. The pumping procedure allows a thorough purge of the pumping system and a water sample can then be obtained with essentially no risk of aeration of the sample. The water level at the port is measured with a manual electric tag liner lowered into the pump tube. Pressure transducers are often incorporated into the system to allow a continuous recording of the head variations in the formation.

Fig. 1. Water FLUTe pump system

(Single port system shown for clarity)



The installation procedure

The Water FLUTe system is everted into the borehole as is normally done for many flexible liner systems. Figure 2 shows the main components of the installation procedure (the pumping system is omitted from the drawing). The liner is positioned on a shipping reel near the wellhead. The liner is inside-out relative to its final state in the borehole.

---

[1] *A New Depth-Discrete Multilevel Monitoring Approach for Fractured Rock,* Ground Water Monitoring & Remediation 27, no. 2/ Spring 2007/pages 57–70.

An air vent tube is first located in the borehole to allow the air above the water table to escape as the liner is installed. A second tube called a pump tube is lowered to the bottom of the hole to allow the water to escape beneath the liner as the liner is everted into the hole (eversion is the opposite procedure to inversion). The top end of the liner is fastened to the surface casing with a large hose clamp. Then the liner is pushed into the casing by hand for a depth of ~3 ft to form an annular pocket. Water is added to the annular pocket which pressurizes the liner and drives it down the hole, pulling itself off the shipping reel. The liner passes through itself and is said to be everting down the borehole. The water level inside the liner is well above the water level in the formation so that the liner interior pressure is higher than the formation pressure, causing a seal of the borehole. As the liner descends, it pushes the borehole water into the formation. If the formation is of low transmissivity, the water must be pumped from beneath the liner via the pump tube. When the liner reaches the bottom of the hole, the tether supporting the pump tubing is tied to a strong bar at the wellhead to prevent any further descent of the tubing bundle.

Fig. 2. Typical Water FLUTe Liner Installation

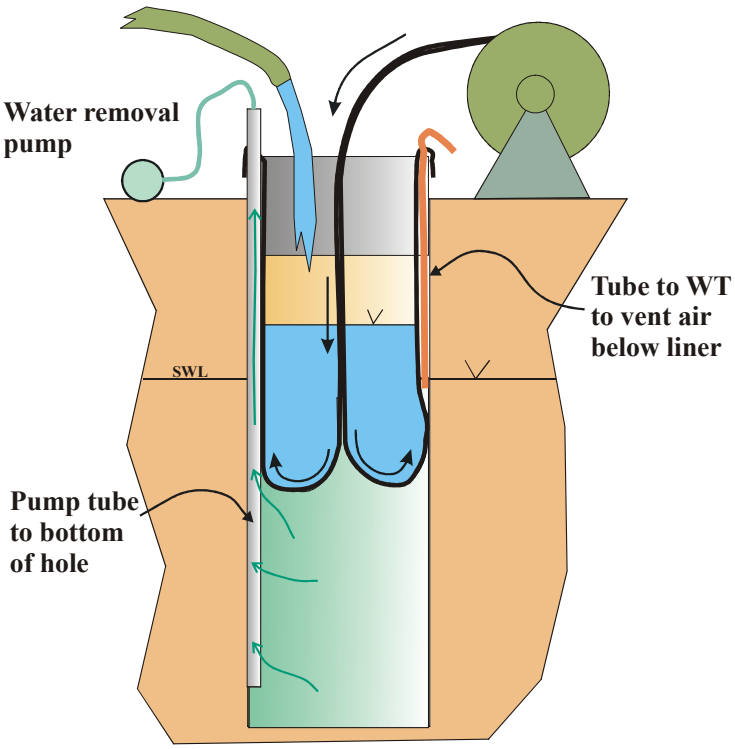


Figure 1 shows the liner fully everted and sealing the borehole. The individual pumping systems are tested to assure that they are fully functional before the pump tube is removed. In order to remove the pump tube, a pump is lowered inside the liner and the water is removed from the liner until the liner begins to collapse. (Sometimes a large tube built into the tubing bundle, called a tag tube, is used as an air lift pump to remove the water from the interior of the liner.) The pump tube is then pulled out of the hole and the liner is refilled to a level about 10 ft above the water table in the formation so as to pressurize the liner and seal the borehole. The sealing liner isolates each sampling interval in the hole to allow a discrete water sample to be drawn from that interval defined by the length of the annular spacer on the exterior of the liner.

The quick connect fittings are added to the top of the pump tubing for connection of the gas source. A nitrogen bottle is used to expel the water from the pumping system as shown in Figure 3.

Special circumstances

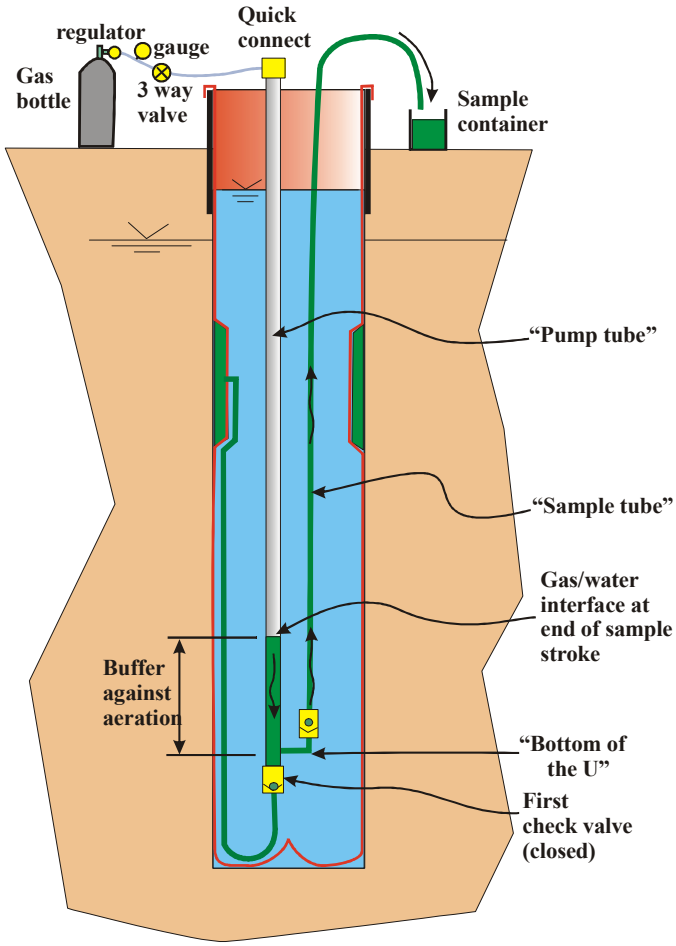
If the water table is very near the surface, a temporary extension of the casing is added to develop a higher driving pressure for the installation of the liner. When the liner is fully

installed, a weighted mud is used as a filling of the liner from the bottom to the top to better pressurize the liner. The mud still allows the liner to be removed by the reverse of the installation process.

In karst formations, a device called an eversion aid can be used inside the bottom end of the liner to cause it to propagate more nearly vertically than a liner driven with water alone. This allows the liner to propagate through large caverns intersected by the borehole.

Water FLUTe liners can be installed equally easily in angled holes or even horizontal holes using the same eversion procedure.

Fig. 3. Pumping Procedure



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FLUTe hydraulic conductivity profiling procedure

Purpose:
The FLUTe hydraulic conductivity profiling procedure, called Profiling for short, is a method invented by FLUTe for obtaining the transmissivity distribution in a borehole while installing a blank sealing liner made of urethane coated nylon. The technique measures the liner installation rate very carefully and from that measurement produces a transmissivity distribution.

The method:
The blank liner installation drives the water from the borehole through all the transmissive features in the borehole. As the liner descends, by the eversion process, it seals the transmissive zones sequentially from the top to the bottom of the hole. When a transmissive zone is sealed, the remaining transmissivity beneath the liner is reduced and that results in an immediate drop in the liner velocity. The velocity change multiplied by the cross section of the hole is the flow rate into that transmissive feature. In that manner, the entire transmissivity of the borehole is mapped in a period of 1-2 hours.

The procedure consists of the careful measurement every half second of the driving pressure in the liner (the excess head above the formation water table), the liner velocity, the tension on the liner at the surface, the pressure in the borehole beneath the liner, and the time of each recording. The procedure requires that the liner tension and the driving pressure in the liner be maintained relatively constant. A recording transducer is positioned at the bottom of the hole to measure the pressure beneath the descending liner.

The procedure
The blank liner is positioned at the wellhead. The profiling machine is set up over the top of the wellhead and the liner is passed through the machine and attached with a clamp to the top of the casing. The liner is pushed into the casing to form an annular space which is then filled with water. For a very shallow water table, a weighted mud may be added to the liner. The profiling machine controls the tension, measures the tension, and measures the velocity and position of the liner. A bubbler is built into the liner to monitor the level of the water inside of the liner (the driving pressure).

The recording system is connected to the profiling machine. The data is recorded into a laptop computer as the liner descends. The liner descent is driven by the addition of water to the inside of the liner from a nearby water storage tank. The water flow rate is controlled by a valve to maintain a constant water level inside of the liner. As the liner is pulled through the machine by the water pressure in the liner, the operator assures that the

data is being well recorded and that the water addition is appropriate. A vacuum line is connect to the interior of the liner on the shipping reel via a hollow axle inside the reel in order to remove any air trapped inside the liner that would otherwise become trapped in the end of the liner and interfere with its free descent.

As the end of the liner passes into the borehole (this occurs when the liner has traveled half the liner length), the liner is followed by a tether attached to the end of liner. This tether in passing though the profiling machine continues the velocity recording process.

When the liner has sealed most of the flow paths in the borehole, the liner velocity slows to a very low rate. When the rate drops to less than 0.001 ft/second, the installation is halted because the hole has been effectively sealed and there is little value in continuing the slow rate of measurement. From the final velocity the conduct/transmissivity of the remainder of the borehole can be calculated.

The liner tether is secured to an anchor at the wellhead to prevent further descent of the liner, and to maintain an excess head in the liner to provide a good seal of the borehole. The removal time for the liner is very similar to the installation time.

The data reduction

The data recorded are entered into a data reduction program which allows various tests of the data validity and then the data are used to calculate the transmissivity distribution in the borehole. The velocity and transmissivity data are plotted versus hole depth and are also provided to the customer in digital form to allow his assessment and use of the transmissivity distribution in the formation intersected by the borehole.

Further information

Numerous technical papers have been published on this procedure and its comparison with the more traditional transmissivity measurements such as straddle packers. Those papers are available from FLUTe at info@flut.com. The main advantage of the method is the low cost of the data beyond the cost of installing a sealing liner, the short amount of time required to perform the measurement, and the high spatial resolution of the transmissivity distribution. When the measurement is completed, the borehole is sealed by the liner.

Questions on this procedure should be addressed to Carl Keller at carl@flut.com.

6 Easy St., Santa Fe, NM 87504
505-455-1300, www.flut.com

Sampling guidelines for *Water FLUTe* systems installed after May, 2009

Rev. April, 2010

Water level in the liner.

The liner water level should be ~10 ft above the highest formation water level to provide a good seal of the liner in the hole (5 ft minimum excess head). The formation water level can be measured via the "pump tube" for each port. The water level inside the liner should be tagged in the ½ x 5/8" tube labeled "TAG" adjacent to the sampling tubes. If the water level inside the liner is measured in the liner, outside the Tag Tube, lower the weighted tag line very slowly to avoid damage to the liner. Water can be added to the liner by simply pouring water into the liner or through the TAG tube, whichever is easier. Do not fill the liner more than 10 ft above the highest formation water level. The water level in the liner should be checked prior to each sampling episode. (Beware that filling the liner with de-ionized water can give a false water level reading.) It is not recommended to manually tag water levels more than 200 ft below the surface. The wet film adhesion may prevent the removal of the tag line. A special Teflon coated tag line can be used to extend that limit.

Water flow

The water flow into the pumping system is shown in Fig. 1. Water flows from the formation through the spacer pore space, through the port tube, through the first check valve, and fills the "pump tube". The "sample tube" is also filled at the same time. The water level rises in the pump tube to the water table for that port.

Setting up the gas pressure source

The water is pumped with gas pressure. The FLUTe pump design is such that there is very low risk of aeration of the sample. The gas source is usually a nitrogen bottle with a regulator for setting the prescribed driving pressure. The arrangement of the FLUTe gas drive system is shown in Fig. 2. The regulator is set to the proper gas pressure defined later by closing the three way valve to prevent gas flow out of the quick connect fitting. The

pressure gauge on the FLUTe pump driver is much more sensitive than the regulator for setting the regulator pressure. The FLUTe pump driver must be securely connected to the regulator at the normal ¼" NPT connection on the regulator outlet.
The regulator is first attached to the top fitting on the gas bottle (a special nitrogen regulator fitting connects to a nitrogen bottle). Tighten the nut securely. Turn the pressure regulator handle counter-clockwise until is moves freely (the no pressure position). Rotate the main valve on the regulator (nearer the bottle) clockwise to fully closed. Open the valve on the bottle (counter clockwise). The main bottle pressure gauge on the regulator will rise to the bottle pressure. Close the regulator valve (clockwise) until the pressure starts to rise on the pressure gauge on the FLUTe pump driver (three way valve closed with no flow out of the quick connect). Adjust the regulator to the desired pressure for purging, provided by FLUTe. Connect the quick connect to the top fitting of the pump tube (see Fig. 2). Open the three way valve to drive the water out of the pump.

Purging

Water is pumped from the tubing by applying the gas pressure to the interface at the static water level in the pump tube (Fig. 1 and 2). The water is driven down in the pump tube and up through the second check valve to the surface via the sample tube. By driving the water with a sufficient gas pressure (the "recommended purge pressure") to drive all of the water in the pump tube and the sample tube to the surface, the water in the pump tubing is nearly all expelled. The purge stroke (~1 gal. of water) is complete when gas is expelled from the sample tube following the water flow. <u>The pressure in the system must then be vented (i.e., dropped to atmospheric by turning the three way valve to the vent position)</u>, to allow the pump tube to refill by flow via the port tube. The recharge flow from the port tube consists of the port tube water, the water in the pore space of the spacer, and water from the medium. Because of the relatively large volume in the pump tube, most of the recharge is from the medium. The recharge will take about as long as the first purge stroke. However, a low conductivity medium will require more time.

Purging the pump tube a second time will remove any of the water that has resided in the spacer and port tube volume. That is highly recommended, since the water resident in the tubing and spacer is probably not typical of the formation water. If the refill has been prompt, the second purge water

volume will be similar to the first stroke. Two more purge strokes, for a total of four purge strokes, are recommended to remove water that may have been in long contact with the liner or spacer. (Note, systems manufactured before May, 2009 use larger pumps and were only stroked twice. The purge volume is slightly larger for this new procedure and takes about the same time as the two stroke system. This new system stresses the liner less at the spacer and has numerous other advantages.)

Sampling

The sampling flow is best driven on the fifth cycle using a "recommended sampling pressure" which is less than that needed to drive gas through the bottom of the pump tube. The pressure recommended is that which will drive the water to near, but not out of, the bottom of the large tube. That recommended pressure, "the sampling pressure," is calculated in the spreadsheet provided with each system. The pressure regulator is set to the sample pressure, which is lower than the purge pressure. Opening the three way valve will now apply the sample pressure to the system causing flow from the sample tube.

The first flow of the sampling cycle sweeps along droplets of water left in the tubing from the purge cycle. That residual water is depleted of volatile components. Tests have shown that the first tube volume of the sample flow should be discarded as depleted in volatiles (the "discard volume" is also calculated in the spreadsheet). Thereafter, the samples can be collected from the sample tube outflow. The volume to be discarded is shown in the spreadsheet as "discard volume". The sample tube water flow rate will start fast, then slow, and finally stop. That occurs as the water column being driven approaches the applied pressure/head. The typical sampling pressure drives to within 25 ft. of the bottom of the pump tube (the U). The large buffer zone remaining in the pump tube assures against aeration of the sample.

This procedure should provide an ample sample (~3 liters) of good quality drawn directly from the formation. If a larger sample volume is needed, simply drop the pressure (i.e., vent the three way valve again), let the pump refill and apply the pressure again. No discard is needed for subsequent sampling flows.

Caution: If the pumping system refills very slowly, there may not be sufficient water in the pump to fill the "sample tube" to the surface when the stroke is performed. In that case, there will be spitting of gas from the sample water and it will be followed by a flow of gas only. The sample water should never show "spitting" and the sample stroke should never end with gas flow from the sample tube. The proper sample flow will slow until it stops flowing. Should this evidence of insufficient recharge be observed, allow the pump to refill for a longer time and repeat the sample stroke. One can tag the water level in the large tube, as described in the head measurement procedure, to assure that the pumping system has been sufficient refilled.

Measuring the head in the system

The water level at each port can be manually measured by removing the plug from the top of the pump tube and lowering a slender (~1/4") electric water level meter until it contacts the water level in the pump tube. It is not recommended to manually tag water levels more than 200 ft below the surface. The wet film adhesion may prevent the removal of the tag line. A special Teflon coated tag line can be used to extend that limit.

The water level in the large tubes may not be the current water level. After sampling, if there is any leakage of the second check valve (sand in the tube, etc…) the water in the sample tube can backflow into the larger tube, adding to the water that fills the large tube during the recharge. Also, if the water level in the formation is dropping between head measurements, the water level in the pump tube will not follow the descent if the first check valve is a good seal. For these two reasons, and for the freezing concern below, it is best to finish the sampling stroke by raising the pressure to the "purge pressure" value to purge the pumping system of all water. Then upon refilling, the level is the current head for each port. If head measurements are made between sampling events, each port's pumping system should be first be purged one stroke to allow the tubing to refill to the current head value. Always replace the plugs in the top of the pump tubes when finished sampling.

If the water might freeze in the sampling tubing near the surface, purge the entire volume of water from each sampling line, after sampling, before leaving it. Use the recommended purge pressure to remove all water, not the sampling pressure. **Each line should be blowing gas when the purge is**

complete. If the tubes were purged after sampling prior to head measurements, that is sufficient.

Since the Water FLUTe uses PVDF tubing, the purge of the entire system after sampling should not be neglected, even if head measurements are not to be made. This removes the water column in the sampling tube. For deep water tables, the long term pressure of the standing water in the sampling tube might lead to excessive creep of the tubing which is susceptible to "cold flow", a characteristic of Teflon like materials. (This is not a concern except for very deep water tables (>300 ft).

In most cases, the performance of a final purge of the system after sampling is useful, even if not essential.

Simultaneous purge and sampling of all tubes

The FLUTe pumping system for each port is essentially identical in length, pump volume and elevation in the hole. This allows all ports to be purged and sampled simultaneously for a great saving in sampling time. The only difference for simultaneous sampling is that the pressure source must include a tube to each port fitting at the wellhead. FLUTe offers a manifold pump driver system at extra cost (the single port driver is provided with the Water FLUTe). The recommended purge and sample pressures are the same as used for single port sampling.

In some cases, the buoyancy of the sampling system is so great when emptied of water during the simultaneous purge that the tubing bundle can cause the liner to invert. The sampling volume spreadsheet provided with the liner notes whether the system can be purged simultaneously. This is only a problem for smaller hole diameters, many ports, and a small excess head in the liner. The new pump design allows simultaneous sampling in most situations.

A short summary is provided as the following checklist:

<u>Check List</u>

1. Check/restore the water level in the liner.
2. Connect the gas driver source to the gas drive (pump) tube for the port.

3. Set the regulator to the recommended purge pressure.
4. Turn the three way valve and expel the tube water at the suggested purge pressure. Collect the purged water volume for verification of a good purge. Note the water flow time of the purge stroke (~4 min.).
5. Allow the tubing to refill. Repeat the purge. Collect the purge volume to assure the amount removed is at least the "port tube volume". Was the refill long enough?
6. Purge a total of four times, more if desired.
7. Allow the tubing to refill for the sample stroke.
8. Reduce the driving pressure to the "sampling pressure". Apply the pressure and collect the first flow to measure the discard volume. Discard that water. Collect the samples.
9. Perform a final purge of the water out of the sampling lines by raising the driving pressure to the purge pressure value.
10. When the sampling system has refilled, tag the water level, if desired, for the current water table. If a port system is refilling very slowly, tag it at a later time.

See the spreadsheet provided with each *Water FLUTe* for the recommended purge and sampling pressures. Those are the pressures that can also be used for a simultaneous purge of the several ports. The spreadsheet flags the condition where all ports should not be purged simultaneously. In most cases, several, to all, of the ports can be purged simultaneously.

Optimum sampling procedure:
Since it is often desirable to minimize the amount of time that the sample water resides in the pumping tubing, it is useful to note the actual time that is required for the recharge of the system. Since the fill rate slows dramatically for the last portion of the recharge, it is not necessary to wait for a complete refill. For most formations, the recharge is dominated by the tubing pressure drop. In that case, the time required for the purge stroke to be completed is about the same time required for the refill. (The exception is for a tight formation that recharges the tubing very slowly.) Hence the second purge can be started after waiting the same length of time as the first purge endured. If the second purge is of a similar volume (usually somewhat less) than the first purge volume, the refill time was long enough. After the same delay, the sampling stroke can be initiated. This timing of the strokes allows one to reduce the retention time in the pumping system. For the very large sample volumes produced, the refill time can be shortened

even more, as long as the sample volume is adequate after the discard of the first flow.

In some situations, the retention time is still too long. FLUTe can often increase the sample tube and port tube diameters for greater flow rates. However, the standard design is well matched for to a wide range of hole diameters, depths, and water table elevations. For very deep wells, the tubing may need to be of higher pressure capacity for the required driving pressures. For water table depths below 700 ft., this may be a concern. FLUTe initiated a design change from Nylon 11 to PVDF tubing in the Water FLUTe systems in 2002 to avoid any concern about tubing interaction with the sample water. However, the prescribed purge is sufficient for the use of Nylon tubing systems.

For special situations such as a very large difference (>50ft) between the water tables at the ports or large fluctuations in the water table, the pumping system may be extended to greater depths. However, the sampling procedure above is sufficient for that situation also.

Questions: Call 888-333-2433 and ask for Carl Keller, or a field engineer.

Figure 1. Water FLUTe pump system

(Single port system shown for clarity)

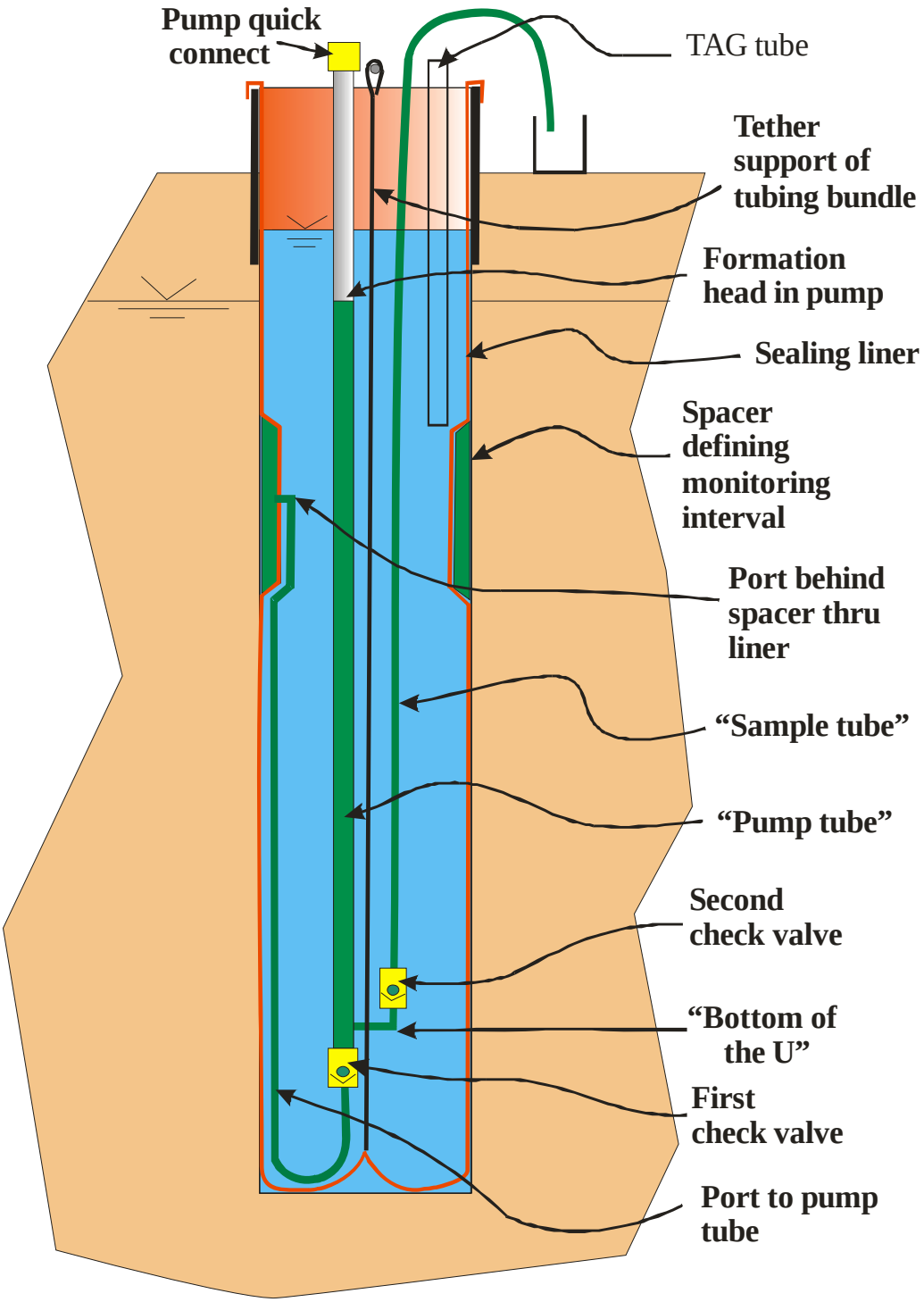
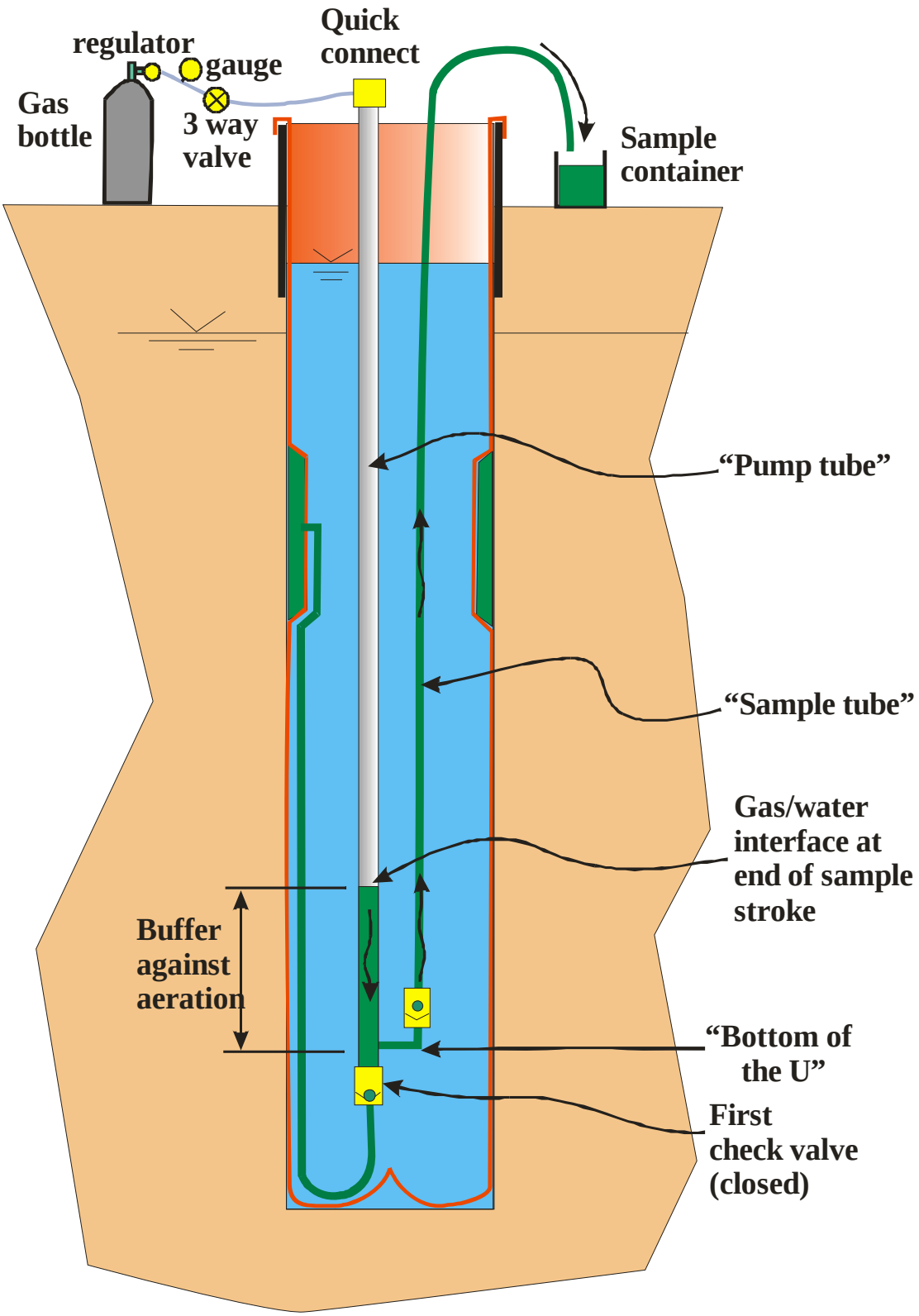


Fig. 2. Pumping Procedure



FLUTe™ Flexible Liner Underground Technologies, Ltd. L.C.
For a medley of innovative designs
6 Easy St., Santa Fe, NM 87504
505-455-1300, www.flut.com

Sampling guidelines for *Water FLUTe* systems installed prior to May, 2009

Rev. April, 2010

Water level in the liner.

The liner water level should be 10 ft above the highest formation water level to provide a good seal of the liner in the hole (5 ft minimum excess head). The formation water level can be measured via the "pump tube" for each port. The water level inside the liner should be tagged in the ½" id tube labeled "TAG" adjacent to the sampling tubes. If the water level inside the liner is measured in the liner, outside the Tag Tube, lower the weighted tag line very slowly to avoid damage to the liner. Water can be added to the liner by simply pouring water into the liner or through the TAG tube, whichever is easier. Do not fill the liner more than 10 ft above the highest formation water level. The water level in the liner should be checked prior to each sampling episode. (Beware that filling the liner with de-ionized water can give a false water level reading.)

Water flow

The water flow into the pumping system is shown in Fig. 1. Water flows from the formation through the spacer pore space, through the port tube, through the first check valve, and fills the "pump tube". The "sample tube" is also filled at the same time. The water level rises in the pump tube to the water table for that port.

Setting up the gas pressure source

The water is pumped with gas pressure. The FLUTe pump design is such that there is very low risk of aeration of the sample. The gas source is usually a nitrogen bottle with a regulator for setting the prescribed driving pressure. The arrangement of the FLUTe gas drive system is shown in Fig. 2. The regulator is set to the proper gas pressure defined later by closing the three way valve to prevent gas flow out of the quick connect fitting. The pressure gauge on the FLUTe pump driver is much more sensitive than the regulator for setting the regulator pressure. The FLUTe pump driver must be securely connected to the regulator at the normal ¼" NPT connection on the regulator outlet.

The regulator is attached to the top fitting on the gas bottle (a special nitrogen regulator fitting connects to a nitrogen bottle). Turn the pressure regulator handle counter-clockwise until is moves freely (the no pressure position). Rotate the main valve on the regulator (nearer the bottle) clockwise to fully closed. Open the valve on the bottle (counter clockwise). The main bottle pressure gauge on the regulator will rise to the bottle pressure. Close the regulator valve (clockwise) until the pressure starts to rise on the pressure gauge on the FLUTe pump driver (three way valve closed with no flow out of the quick connect). Adjust the regulator to the desired pressure for purging, provided by FLUTe. Remove the plug of each pump tube and connect the quick connect to the top fitting of the pump tube (see Fig. 2). Open the three way valve to drive the water out of the pump.

Purging

Water is pumped from the tubing by applying the gas pressure to the interface at the static water level in the pump tube (Fig. 1 and 2). The water is driven down in the pump tube and up through the second check valve to the surface via the sample tube. Drive the water with a sufficient gas pressure (the "recommended purge pressure") to drive all of the water in the pump tube and the sample tube to the surface, the water in the pump tubing is nearly all expelled. The purge stroke is complete when gas is expelled from the sample tube following the water flow. The pressure in the system must then be vented (i.e., dropped to atmospheric by turning the three way valve to the vent position), to allow the pump tube to refill with flow via the port tube. The recharge flow from the port tube consists of the port tube water, the water in the pore space of the spacer, and water from the medium. Because of the relatively large volume in the pump tube, most of the recharge is from the medium. The recharge will take about as long as the first purge stroke. However, a low conductivity medium will require more time.

Purging the pump tube a second time will remove any of the water that has resided in the spacer and port tube volume. That is highly recommended, since the water resident in the tubing and spacer is probably not typical of the formation water. If the refill has been prompt, the second purge water volume will be similar to the first stroke. If in doubt, or if in a sedimentary formation or screened well, a third purge stroke is recommended to remove water that may have been in long contact with the liner or spacer.

Sampling
The sampling flow is best driven on the third (or fourth) cycle by a "recommended sampling pressure" which is less than that needed to drive gas through the bottom of the pump tube. The pressure recommended is that which will drive the water to near, but not out of, the bottom of the large tube. That recommended pressure, "the sampling pressure," is calculated in the spreadsheet provided with each system. The pressure regulator is set to the sample pressure, which is lower than the purge pressure. Opening the three way valve will now apply the sample pressure to the system causing flow from the sample tube.

The first flow of the sampling cycle sweeps along droplets of water left in the tubing from the purge cycle. That residual water is depleted of volatile components. Tests have shown that the first tube volume of the sample flow should be discarded as depleted in volatiles (the "discard volume" is also calculated in the spreadsheet). Thereafter, the samples can be collected from the sample tube outflow. The volume to be discarded is shown in the spreadsheet as "discard volume". The sample tube water flow rate will start fast, then slow, and finally stop. That occurs as the water column being driven approaches the applied pressure/head. The typical sampling pressure drives to within 25 ft. of the bottom of the pump tube (the U). The large buffer zone remaining in the pump tube assures against aeration of the sample.

This procedure should provide an ample sample of good quality drawn directly from the formation.

Caution: If the pumping system refills very slowly, there may not be sufficient water in the pump to fill the "sample tube" to the surface when the stroke is performed. In that case, there will be spitting of gas from the sample water and it will be followed by a flow of gas only. The sample water should never show "spitting" and the sample stroke should never end with gas flow from the sample tube. The proper sample flow will slow until it stops flowing. Should this evidence of insufficient recharge be observed, allow the pump to refill for a longer time and repeat the sample stroke. One can tag the water level in the large tube, as described in the head

measurement procedure, to assure that the pumping system has been sufficient refilled.

Measuring the head in the system

The water level at each port can be manually measured by removing the plug from the top of the pump tube and lowering a slender (~1/4") electric water level meter until it contacts the water level in the pump tube. It is not recommended to manually tag water levels more than 200 ft below the surface. The wet film adhesion may prevent the removal of the tag line. A special Teflon coated tag line can be used to extend that limit.

The water level in the large tubes may not be the current water level. After sampling, if there is any leakage of the second check valve (sand in the tube, etc…) the water in the sample tube can backflow into the larger tube, adding to the water that fills the large tube during the recharge. Also, if the water level in the formation is dropping between head measurements, the water level in the pump tube will not follow the descent if the first check valve is a good seal. For these two reasons, and for the freezing concern below, it is best to finish the sampling stroke by raising the pressure to the "purge pressure" value to purge the pumping system of all water. Then upon refilling, the level is the current head for each port. If head measurements are made between sampling events, each port's pumping system should be first be purged to allow the tubing to refill to the current head value. Always replace the plugs in the top of the pump tubes when finished sampling.

If the water might freeze in the sampling tubing near the surface, purge the entire volume of water from each sampling line, after sampling, before leaving it. Use the recommended purge pressure to remove all water, not the sampling pressure. **Each line should be blowing gas when the purge is complete.** If the lines were purged after sampling for head measurements, that is sufficient.

If the Water FLUTe uses PVDF tubing, the purge of the entire system after sampling should not be neglected, even if head measurements are not to be made. This removes the water column in the sampling tube. For deep water tables, the long term pressure of the standing water in the sampling tube might lead to excessive creep of the tubing which is susceptible to "cold flow", a characteristic of Teflon like materials. (This is not a concern except for very deep water tables (>300 ft).

In most cases, the performance of a final purge of the system after sampling is useful, even if not essential.

Simultaneous purge and sampling of all tubes

The FLUTe pumping system for each port is essentially identical in length, pump volume and elevation in the hole. This allows all ports to be purged and sampled simultaneously for a great saving in sampling time. The only difference for simultaneous sampling is that the pressure source must include a tube to each port fitting at the wellhead. FLUTe offers a manifold pump driver system at extra cost (the single port driver is provided with the Water FLUTe). The recommended purge and sample pressures are the same as used for single port sampling.

In some cases, the buoyancy of the sampling system is so great when emptied of water during the simultaneous purge that the tubing bundle can cause the liner to invert. The sampling volume spreadsheet provided with the liner notes whether the system can be purged simultaneously. This is only a problem for smaller hole diameters, many ports, and a small excess head in the liner. However, increasing the excess head in the liner to overcome the buoyancy of the tubing can be a hazard to the liner.

A short summary is provided as the following checklist:

<u>Check List</u>

1. Check/restore the water level in the liner.
2. Connect the gas driver source to the gas drive tube for the port.
3. Set the regulator to the recommended purge pressure.
4. Expel the tube water at the suggested purge pressure. Collect the purged water volume for verification of a good purge. Note the water flow time of the purge stroke.
5. Allow the tubing to refill. Repeat the purge. Collect the purge volume to assure the amount removed is at least the “port tube volume”. Was the refill long enough?
6. Purge a third time, if desired.
7. Allow the tubing to refill for the sample stroke.

8. Reduce the driving pressure to the "sampling pressure". Apply the pressure and collect the first flow to measure the discard volume. Discard that water.
9. Reduce the pressure, if needed, to slow the flow and collect the samples.
10. Perform a final purge of the water out of the sampling lines by raising the driving pressure to the purge pressure value.
11. When the sampling system has refilled, tag the water level, if desired, for the current water table. If a port system is refilling very slowly, tag it at a later time.

See the spreadsheet provided with each *Water FLUTe* for the recommended purge and sampling pressures. Those are the pressures that can be used for a simultaneous purge of the several ports, but be sure that the buoyancy of the tubing will not lift the tubing, and the wellhead. The spreadsheet flags the condition where all ports should not be purged simultaneously. In most cases, several, to all, of the ports can be purged simultaneously.

Optimum sampling procedure:
Since it is often desirable to minimize the amount of time that the sample water resides in the pumping tubing, it is useful to note the actual time that is required for the recharge of the system. Since the fill rate slows dramatically for the last portion of the recharge, it is not necessary to wait for a complete refill. For most formations, the recharge is dominated by the tubing pressure drop. In that case, the time required for the purge stroke to be completed is about the same time required for the refill. (The exception is for a tight formation that recharges the tubing very slowly.) Hence the second purge can be started after waiting the same length of time as the first purge endured. If the second purge is of a similar volume (usually somewhat less) than the first purge volume, the refill time was long enough. After the same delay, the sampling stroke can be initiated. This timing of the strokes allows one to reduce the retention time in the pumping system. For very large sample volumes produced, the refill time can be shortened even more, as long as the sample volume is adequate after the discard of the first flow.

In some situations, the retention time is still too long. FLUTe can often increase the sample tube and port tube diameters for greater flow rates. However, the standard design is well matched for to a wide range of hole diameters, depths, and water table elevations. For very deep wells, the

tubing may need to be of higher pressure capacity for the required driving pressures. For water table depths below 700 ft., this may be a concern. FLUTe initiated a design change from Nylon 11 to PVDF tubing in the Water FLUTe systems in 2002 to avoid any concern about tubing interaction with the sample water. However, the prescribed purge is sufficient for the use of Nylon tubing systems.

Questions: Call 888-333-2433 and ask for Carl Keller, or a field engineer.

Figure 1. Water FLUTe pump system

(Single port system shown for clarity)

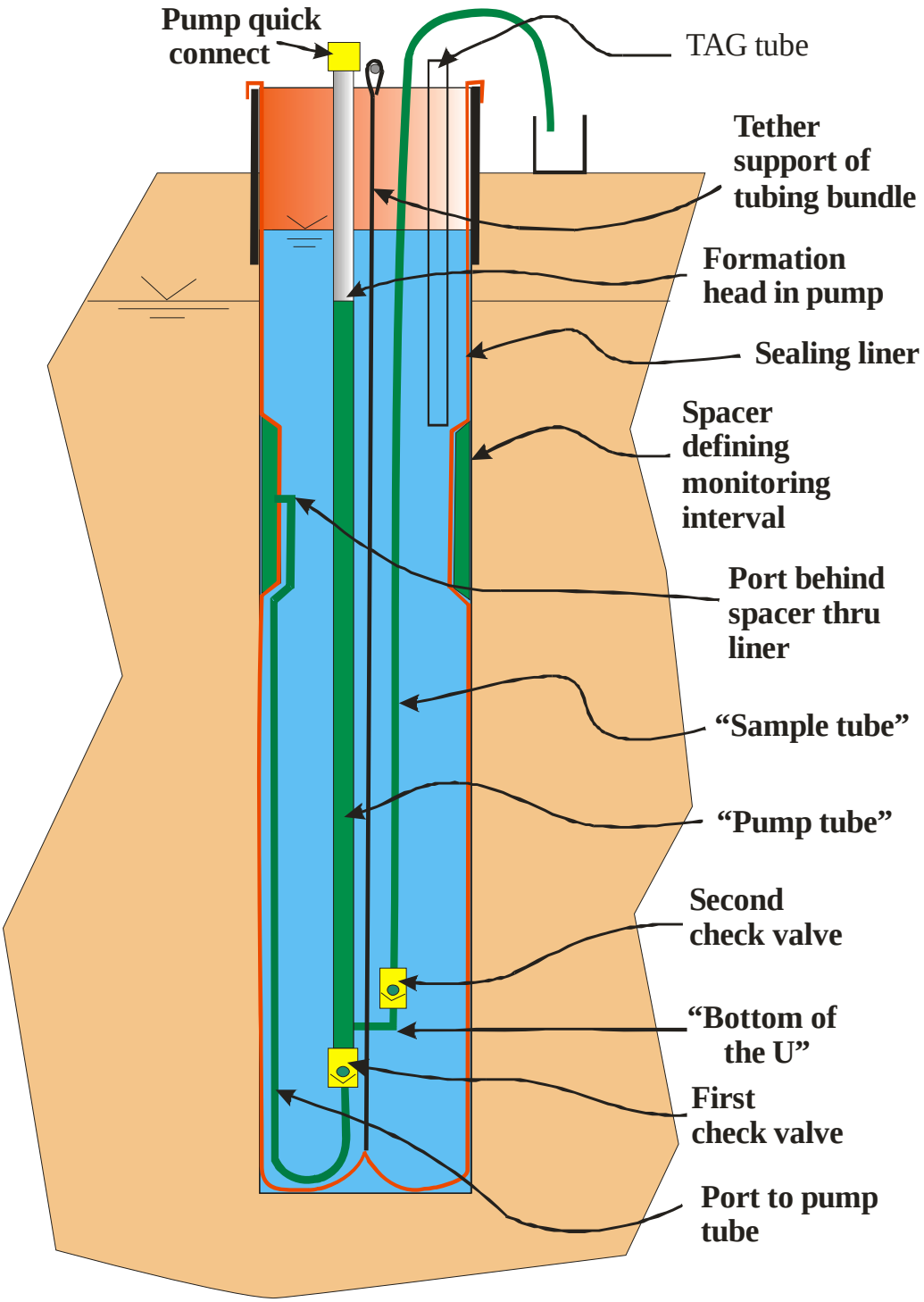
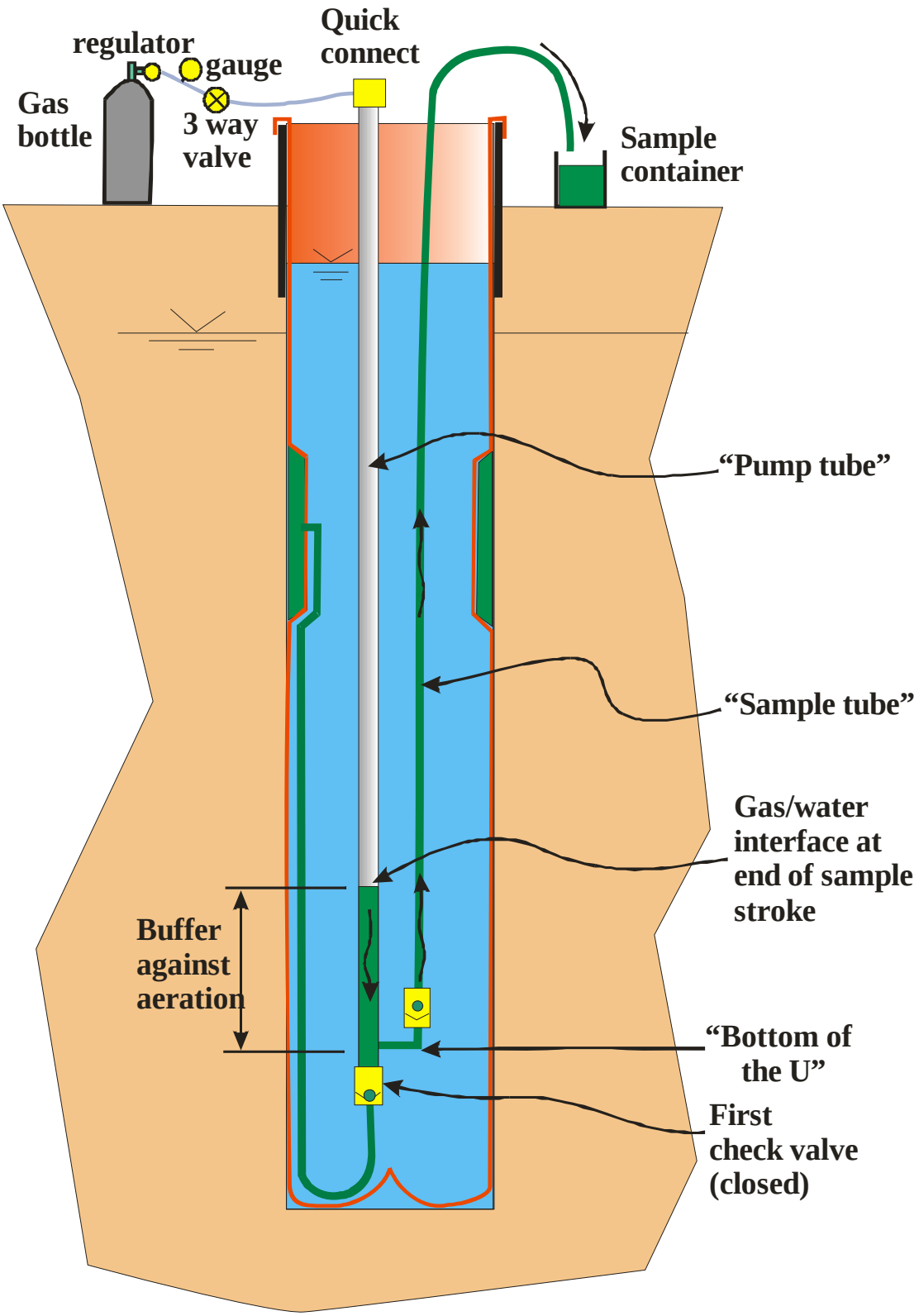
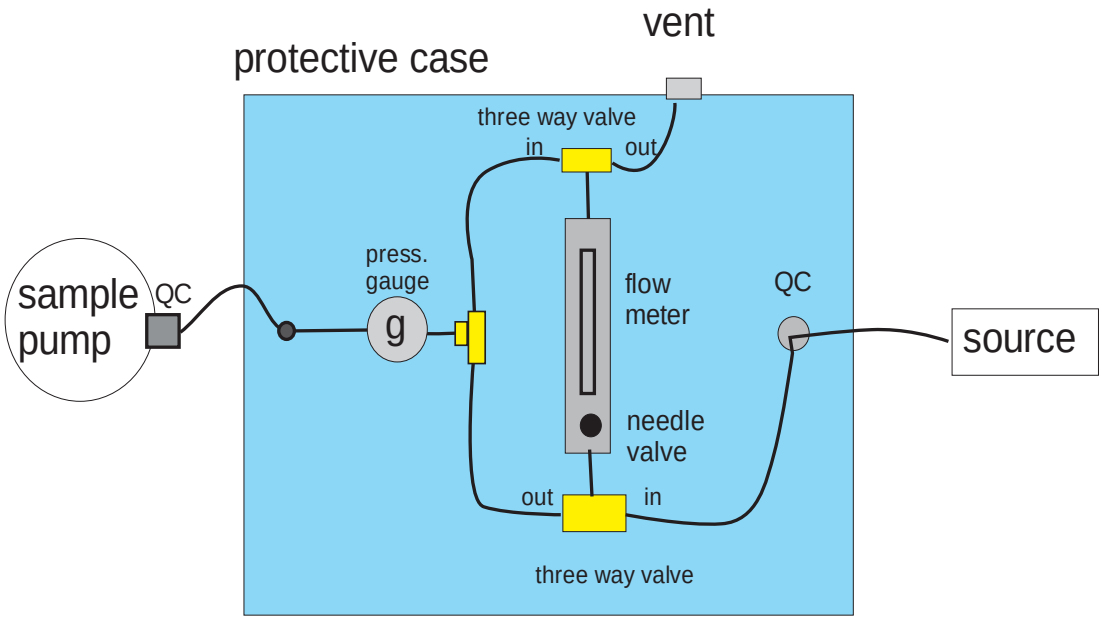


Fig. 2. Pumping Procedure



FLUTe™ Flexible Liner Underground Technologies, LLC
P. O. Box 340, Alcalde, NM 87511

Flow Control system for Water FLUTe



Price: $855

FLUTe™ Flexible Liner Underground Technologies, LLC
P. O. Box 340, Alcalde, NM 87511

Flow rates for microseeps flow controller

enter numbers in bold outlined cells only

depth of pump
130

water table
30

volume of pump tube
0.17726 cubic ft.
1.329449 gal.

time to refill
5 min.

time to flow throught cell
20 min

pressure in pump
4.829252 bars abs.

gauge press.
3.829252 bars

	Flow rate to refill	flow rate to flow through cell
	0.135755	0.033939
	8.145271 SCFH	2.036318 SCFH

Appendix E

Laboratory Certifications

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 5.1.1 February 2018 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program
(DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:
Environmental Testing
(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Tracy Szerszen
President/Operations Manager

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
January 22, 2010	February 4, 2018	March 31, 2020
Revision Date:	*Accreditation No.:*	*Certificate No.:*
December 13, 2018	65817	L18-62-R1

Perry Johnson Laboratory Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Contact Name: Vicky Collom Phone: 585-288-5380

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	ASTM D6919	IC	Ammonia
Aqueous	EPA 200.7	ICP	Aluminum
Aqueous	EPA 200.7	ICP	Antimony
Aqueous	EPA 200.7	ICP	Arsenic
Aqueous	EPA 200.7	ICP	Barium
Aqueous	EPA 200.7	ICP	Beryllium
Aqueous	EPA 200.7	ICP	Boron
Aqueous	EPA 200.7	ICP	Cadmium
Aqueous	EPA 200.7	ICP	Calcium
Aqueous	EPA 200.7	ICP	Chromium
Aqueous	EPA 200.7	ICP	Cobalt
Aqueous	EPA 200.7	ICP	Copper
Aqueous	EPA 200.7	ICP	Iron
Aqueous	EPA 200.7	ICP	Lead
Aqueous	EPA 200.7	ICP	Magnesium
Aqueous	EPA 200.7	ICP	Manganese
Aqueous	EPA 200.7	ICP	Molybdenum
Aqueous	EPA 200.7	ICP	Nickel
Aqueous	EPA 200.7	ICP	Potassium
Aqueous	EPA 200.7	ICP	Selenium
Aqueous	EPA 200.7	ICP	Silver
Aqueous	EPA 200.7	ICP	Sodium
Aqueous	EPA 200.7	ICP	Strontium
Aqueous	EPA 200.7	ICP	Thallium
Aqueous	EPA 200.7	ICP	Tin
Aqueous	EPA 200.7	ICP	Titanium
Aqueous	EPA 200.7	ICP	Vanadium
Aqueous	EPA 200.7	ICP	Zinc
Aqueous	EPA 200.8	ICP/MS	Aluminum
Aqueous	EPA 200.8	ICP/MS	Antimony
Aqueous	EPA 200.8	ICP/MS	Arsenic
Aqueous	EPA 200.8	ICP/MS	Barium
Aqueous	EPA 200.8	ICP/MS	Beryllium
Aqueous	EPA 200.8	ICP/MS	Cadmium

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 200.8	ICP/MS	Chromium
Aqueous	EPA 200.8	ICP/MS	Cobalt
Aqueous	EPA 200.8	ICP/MS	Copper
Aqueous	EPA 200.8	ICP/MS	Lead
Aqueous	EPA 200.8	ICP/MS	Manganese
Aqueous	EPA 200.8	ICP/MS	Nickel
Aqueous	EPA 200.8	ICP/MS	Selenium
Aqueous	EPA 200.8	ICP/MS	Silver
Aqueous	EPA 200.8	ICP/MS	Thallium
Aqueous	EPA 200.8	ICP/MS	Vanadium
Aqueous	EPA 200.8	ICP/MS	Zinc
Aqueous	EPA 218.6	IC-UV	Chromium, Hexavalent
Aqueous	EPA 245.1	CVAA	Mercury
Aqueous	EPA 300.0	IC	Bromide
Aqueous	EPA 300.0	IC	Chloride
Aqueous	EPA 300.0	IC	Fluoride
Aqueous	EPA 300.0	IC	Nitrate
Aqueous	EPA 300.0	IC	Nitrite
Aqueous	EPA 300.0	IC	Sulfate
Aqueous	EPA 335.4	Colorimetric	Cyanide
Aqueous	EPA 351.2	Colorimetric	TKN
Aqueous	EPA 410.4	Colorimetric	COD
Aqueous	EPA 420.4	Colorimetric	Phenols
Aqueous	EPA 608.3	GC/ECD	4,4-DDD
Aqueous	EPA 608.3	GC/ECD	4,4-DDE
Aqueous	EPA 608.3	GC/ECD	4,4-DDT
Aqueous	EPA 608.3	GC/ECD	a-BHC
Aqueous	EPA 608.3	GC/ECD	Aldrin
Aqueous	EPA 608.3	GC/ECD	Alpha-chlordane
Aqueous	EPA 608.3	GC/ECD	Aroclor 1016
Aqueous	EPA 608.3	GC/ECD	Aroclor 1221
Aqueous	EPA 608.3	GC/ECD	Aroclor 1232
Aqueous	EPA 608.3	GC/ECD	Aroclor 1242
Aqueous	EPA 608.3	GC/ECD	Aroclor 1248
Aqueous	EPA 608.3	GC/ECD	Aroclor 1254
Aqueous	EPA 608.3	GC/ECD	Aroclor 1260

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Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 608.3	GC/ECD	b-BHC
Aqueous	EPA 608.3	GC/ECD	Chlordane
Aqueous	EPA 608.3	GC/ECD	d-BHC
Aqueous	EPA 608.3	GC/ECD	Dieldrin
Aqueous	EPA 608.3	GC/ECD	Endosulfan I
Aqueous	EPA 608.3	GC/ECD	Endosulfan II
Aqueous	EPA 608.3	GC/ECD	Endosulfan sulfate
Aqueous	EPA 608.3	GC/ECD	Endrin
Aqueous	EPA 608.3	GC/ECD	Endrin aldehyde
Aqueous	EPA 608.3	GC/ECD	Endrin ketone
Aqueous	EPA 608.3	GC/ECD	Gamma-chlordane
Aqueous	EPA 608.3	GC/ECD	g-BHC
Aqueous	EPA 608.3	GC/ECD	Heptachlor
Aqueous	EPA 608.3	GC/ECD	Heptachlor epoxide
Aqueous	EPA 608.3	GC/ECD	Methoxychlor
Aqueous	EPA 608.3	GC/ECD	Toxaphene
Aqueous	EPA 624.1	GC/MS	1,1,1,2-Tetrachloroethane
Aqueous	EPA 624.1	GC/MS	1,1,1-Trichloroethane
Aqueous	EPA 624.1	GC/MS	1,1.2-Trichloroethane
Aqueous	EPA 624.1	GC/MS	1,1,2,2-Tetrachloroethane
Aqueous	EPA 624.1	GC/MS	1,1-Dichloroethane
Aqueous	EPA 624.1	GC/MS	1,1-Dichloroethylene
Aqueous	EPA 624.1	GC/MS	1,1-Dichloropropene
Aqueous	EPA 624.1	GC/MS	1,2-Dichloropropane
Aqueous	EPA 624.1	GC/MS	1,2,3-Trichlorobenzene
Aqueous	EPA 624.1	GC/MS	1,2,3-Trichloropropane
Aqueous	EPA 624.1	GC/MS	1,2,4-Trichlorobenzene
Aqueous	EPA 624.1	GC/MS	1,2,4-Trimethylbenzene
Aqueous	EPA 624.1	GC/MS	1,2-Dibromo-3-chloropropane
Aqueous	EPA 624.1	GC/MS	1,2-Dibromoethane
Aqueous	EPA 624.1	GC/MS	1,2-Dichlorobenzene
Aqueous	EPA 624.1	GC/MS	1,2-Dichloroethane
Aqueous	EPA 624.1	GC/MS	1,3,5-Trimethylbenzene
Aqueous	EPA 624.1	GC/MS	1,3-Dichlorobenzene
Aqueous	EPA 624.1	GC/MS	1,3-Dichloropropane
Aqueous	EPA 624.1	GC/MS	1,4-Dichlorobenzene

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Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 624.1	GC/MS	2,2-Dichloropropane
Aqueous	EPA 624.1	GC/MS	2-Butanone (MEK)
Aqueous	EPA 624.1	GC/MS	2-Chloroethylvinyl ether
Aqueous	EPA 624.1	GC/MS	2-Chlorotoluene
Aqueous	EPA 624.1	GC/MS	2-Hexanone
Aqueous	EPA 624.1	GC/MS	4-Chlorotoluene
Aqueous	EPA 624.1	GC/MS	4-Isopropyltoluene
Aqueous	EPA 624.1	GC/MS	4-Methyl-2-pentanone (MIBK)
Aqueous	EPA 624.1	GC/MS	Acetone
Aqueous	EPA 624.1	GC/MS	Benzene
Aqueous	EPA 624.1	GC/MS	Bromobenzene
Aqueous	EPA 624.1	GC/MS	Bromochloromethane
Aqueous	EPA 624.1	GC/MS	Bromodichloromethane
Aqueous	EPA 624.1	GC/MS	Bromoform
Aqueous	EPA 624.1	GC/MS	Bromomethane
Aqueous	EPA 624.1	GC/MS	Carbon disulfide
Aqueous	EPA 624.1	GC/MS	Carbon tetrachloride
Aqueous	EPA 624.1	GC/MS	Chlorobenzene
Aqueous	EPA 624.1	GC/MS	Chloroethane
Aqueous	EPA 624.1	GC/MS	Chloroform
Aqueous	EPA 624.1	GC/MS	Chloromethane
Aqueous	EPA 624.1	GC/MS	cis-1,2-Dichloroethene
Aqueous	EPA 624.1	GC/MS	cis-1,3-Dichloropropene
Aqueous	EPA 624.1	GC/MS	Dibromochloromethane
Aqueous	EPA 624.1	GC/MS	Dibromomethane
Aqueous	EPA 624.1	GC/MS	Dichlorodifluoromethane (Freon 12)
Aqueous	EPA 624.1	GC/MS	Ethylbenzene
Aqueous	EPA 624.1	GC/MS	Isopropylbenzene
Aqueous	EPA 624.1	GC/MS	m- + p-Xylene
Aqueous	EPA 624.1	GC/MS	Methylene Chloride
Aqueous	EPA 624.1	GC/MS	Methyl-tert-butyl ether (MTBE)
Aqueous	EPA 624.1	GC/MS	Naphthalene
Aqueous	EPA 624.1	GC/MS	N-butylbenzene
Aqueous	EPA 624.1	GC/MS	N-propylbenzene

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Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 624.1	GC/MS	o-Xylene
Aqueous	EPA 624.1	GC/MS	sec-butylbenzene
Aqueous	EPA 624.1	GC/MS	Styrene
Aqueous	EPA 624.1	GC/MS	tert-butylbenzene
Aqueous	EPA 624.1	GC/MS	Tetrachloroethene
Aqueous	EPA 624.1	GC/MS	Toluene
Aqueous	EPA 624.1	GC/MS	trans-1,2-Dichloroethene
Aqueous	EPA 624.1	GC/MS	trans-1,3-Dichloropropene
Aqueous	EPA 624.1	GC/MS	Trichloroethylene
Aqueous	EPA 624.1	GC/MS	Trichlorofluoromethane (Freon 11)
Aqueous	EPA 624.1	GC/MS	Vinyl chloride
Aqueous	EPA 624.1	GC/MS	Xylenes, total
Aqueous	EPA 625.1	GC/MS	1,2,4-Trichlorobenzene
Aqueous	EPA 625.1	GC/MS	1,2-Diphenylhydrazine
Aqueous	EPA 625.1	GC/MS	2,2-oxybis(1-chloropropane)
Aqueous	EPA 625.1	GC/MS	2,4,5-Trichlorophenol
Aqueous	EPA 625.1	GC/MS	2,4,6-Trichlorophenol
Aqueous	EPA 625.1	GC/MS	2,4-Dichlorophenol
Aqueous	EPA 625.1	GC/MS	2,4-Dimethylphenol
Aqueous	EPA 625.1	GC/MS	2,4-Dinitrophenol
Aqueous	EPA 625.1	GC/MS	2,4-Dinitrotoluene
Aqueous	EPA 625.1	GC/MS	2,6-Dinitrotoluene
Aqueous	EPA 625.1	GC/MS	2-Chloronaphthalene
Aqueous	EPA 625.1	GC/MS	2-Chlorophenol
Aqueous	EPA 625.1	GC/MS	2-Methylnaphthalene
Aqueous	EPA 625.1	GC/MS	2-Methylphenol
Aqueous	EPA 625.1	GC/MS	2-Nitrophenol
Aqueous	EPA 625.1	GC/MS	3,3'-Dichlorobenzidine
Aqueous	EPA 625.1	GC/MS	3+4-Methylphenol
Aqueous	EPA 625.1	GC/MS	4,6-Dinitro-2-methylphenol
Aqueous	EPA 625.1	GC/MS	4-Bromophenyl-phenylether
Aqueous	EPA 625.1	GC/MS	4-Chloro-3-methylphenol
Aqueous	EPA 625.1	GC/MS	4-Chlorophenyl-phenylether
Aqueous	EPA 625.1	GC/MS	4-Nitrophenol
Aqueous	EPA 625.1	GC/MS	Acenaphthene
Aqueous	EPA 625.1	GC/MS	Acenaphthylene

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Aqueous	EPA 625.1	GC/MS	Acetophenone
Aqueous	EPA 625.1	GC/MS	Anthracene
Aqueous	EPA 625.1	GC/MS	Benzidine
Aqueous	EPA 625.1	GC/MS	Benzo(a)anthracene
Aqueous	EPA 625.1	GC/MS	Benzo(a)pyrene
Aqueous	EPA 625.1	GC/MS	Benzo(b)fluoranthene
Aqueous	EPA 625.1	GC/MS	Benzo(g,h,i)perylene
Aqueous	EPA 625.1	GC/MS	Benzo(k)fluoranthene
Aqueous	EPA 625.1	GC/MS	Benzoic acid
Aqueous	EPA 625.1	GC/MS	Bis(-2-chloroethoxy)methane
Aqueous	EPA 625.1	GC/MS	Bis(2-chloroethyl)ether
Aqueous	EPA 625.1	GC/MS	Bis(2-ethylhexyl)phthalate
Aqueous	EPA 625.1	GC/MS	Butyl benzyl phthalate
Aqueous	EPA 625.1	GC/MS	Chrysene
Aqueous	EPA 625.1	GC/MS	Dibenzo(a,h)anthracene
Aqueous	EPA 625.1	GC/MS	Dibenzofuran
Aqueous	EPA 625.1	GC/MS	Diethylphthalate
Aqueous	EPA 625.1	GC/MS	Dimethyl phthalate
Aqueous	EPA 625.1	GC/MS	Di-n-butylphthalate
Aqueous	EPA 625.1	GC/MS	Di-n-octyl phthalate
Aqueous	EPA 625.1	GC/MS	Fluoranthene
Aqueous	EPA 625.1	GC/MS	Fluorene
Aqueous	EPA 625.1	GC/MS	Hexachlorobenzene
Aqueous	EPA 625.1	GC/MS	Hexachlorobutadiene
Aqueous	EPA 625.1	GC/MS	Hexachlorocyclopentadiene
Aqueous	EPA 625.1	GC/MS	Hexachloroethane
Aqueous	EPA 625.1	GC/MS	Indeno(1,2,3-cd)pyrene
Aqueous	EPA 625.1	GC/MS	Isophorone
Aqueous	EPA 625.1	GC/MS	Naphthalene
Aqueous	EPA 625.1	GC/MS	Nitrobenzene
Aqueous	EPA 625.1	GC/MS	N-nitrosodimethylamine
Aqueous	EPA 625.1	GC/MS	N-nitroso-di-n-propylamine
Aqueous	EPA 625.1	GC/MS	N-nitrosodiphenylamine
Aqueous	EPA 625.1	GC/MS	Pentachlorophenol
Aqueous	EPA 625.1	GC/MS	Phenanthrene
Aqueous	EPA 625.1	GC/MS	Phenol

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Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 625.1	GC/MS	Pyrene
Aqueous	EPA 625.1	GC/MS	Pyridine
Aqueous	EPA 6020A	ICP/MS	Aluminum
Aqueous	EPA 7470A	CVAA	Mercury
Aqueous	EPA 8081B	GC-ECD	Hexachlorobenzene
Aqueous	EPA 8151A	GC-ECD	Dinoseb
Aqueous	EPA 8270D	GC-MS-SIM	1,4-Dioxane
Aqueous	EPA 8270D	GC-MS	1-Methylnaphthalene
Aqueous	EPA 9012B	Colorimetric	Cyanide
Aqueous	EPA 9040C	pH Meter	pH
Aqueous	EPA 9040C	POT	pH
Aqueous	EPA 9056A	IC	Bromide
Aqueous	EPA 9056A	IC	Chloride
Aqueous	EPA 9056A	IC	Fluoride
Aqueous	EPA 9056A	IC	Nitrate
Aqueous	EPA 9056A	IC	Nitrite
Aqueous	EPA 9056A	IC	Sulfate
Aqueous	EPA 9060A	TOC Meter	TOC
Aqueous	EPA 9066	Colorimetric	Phenols
Aqueous	RSK-175	GC-FID	Acetylene
Aqueous	RSK-175	GC-FID	Ethane
Aqueous	RSK-175	GC-FID	Ethylene
Aqueous	RSK-175	GC-FID	Methane
Aqueous	RSK-175	GC-FID	Propane
Aqueous	SM 2320B	Titrimetric	Alkalinity
Aqueous	SM 2340B	ICP-AES	Hardness by calculation
Aqueous	SM 2540B	Gravimetric	TS
Aqueous	SM 2540C	Gravimetric	TDS
Aqueous	SM 2540D	Gravimetric	TSS
Aqueous	SM 3500 Fe D	Colormetric	Ferrous Iron
Aqueous	SM 4500 H + B	Electrochemical	pH
Aqueous	SM 4500 S2F	Titrimetric	Sulfide
Aqueous	SM 5210B	Electrochemical	BOD/CBOD
Aqueous	SM 5310C	TOC Meter	TOC
Aqueous	SM 2540E	Gravimetric	TVS
Solid	EPA 7471B	CVAA	Mercury

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Matrix	Standard/Method	Technology	Analyte
Solid	EPA 9045D	Electrochemical	pH
Solid	EPA Lloyd Kahn	TOC Meter	Total organic carbon
Solid	ALS SOP GEN-DWPS	Gravimetric	Percent Solid
Aqueous/Solid	EPA 6010C	ICP-AES	Aluminum
Aqueous/Solid	EPA 6010C	ICP-AES	Antimony
Aqueous/Solid	EPA 6010C	ICP-AES	Arsenic
Aqueous/Solid	EPA 6010C	ICP-AES	Barium
Aqueous/Solid	EPA 6010C	ICP-AES	Beryllium
Aqueous/Solid	EPA 6010C	ICP-AES	Boron
Aqueous/Solid	EPA 6010C	ICP-AES	Cadmium
Aqueous/Solid	EPA 6010C	ICP-AES	Calcium
Aqueous/Solid	EPA 6010C	ICP-AES	Chromium
Aqueous/Solid	EPA 6010C	ICP-AES	Cobalt
Aqueous/Solid	EPA 6010C	ICP-AES	Copper
Aqueous/Solid	EPA 6010C	ICP-AES	Iron
Aqueous/Solid	EPA 6010C	ICP-AES	Lead
Aqueous/Solid	EPA 6010C	ICP-AES	Magnesium
Aqueous/Solid	EPA 6010C	ICP-AES	Manganese
Aqueous/Solid	EPA 6010C	ICP-AES	Molybdenum
Aqueous/Solid	EPA 6010C	ICP-AES	Nickel
Aqueous/Solid	EPA 6010C	ICP-AES	Potassium
Aqueous/Solid	EPA 6010C	ICP-AES	Selenium
Aqueous/Solid	EPA 6010C	ICP-AES	Silver
Aqueous/Solid	EPA 6010C	ICP-AES	Sodium
Aqueous/Solid	EPA 6010C	ICP-AES	Strontium
Aqueous/Solid	EPA 6010C	ICP-AES	Thallium
Aqueous/Solid	EPA 6010C	ICP-AES	Tin
Aqueous/Solid	EPA 6010C	ICP-AES	Titanium
Aqueous/Solid	EPA 6010C	ICP-AES	Vanadium
Aqueous/Solid	EPA 6010C	ICP-AES	Zinc
Aqueous	EPA 6020A	ICP/MS	Aluminum
Aqueous/Solid	EPA 6020A	ICP-MS	Antimony
Aqueous/Solid	EPA 6020A	ICP-MS	Arsenic
Aqueous/Solid	EPA 6020A	ICP-MS	Barium
Aqueous/Solid	EPA 6020A	ICP-MS	Beryllium
Aqueous/Solid	EPA 6020A	ICP-MS	Cadmium

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 6020A	ICP-MS	Chromium
Aqueous/Solid	EPA 6020A	ICP-MS	Cobalt
Aqueous/Solid	EPA 6020A	ICP-MS	Copper
Aqueous/Solid	EPA 6020A	ICP-MS	Lead
Aqueous/Solid	EPA 6020A	ICP-MS	Manganese
Aqueous/Solid	EPA 6020A	ICP-MS	Nickel
Aqueous/Solid	EPA 6020A	ICP-MS	Selenium
Aqueous/Solid	EPA 6020A	ICP-MS	Silver
Aqueous/Solid	EPA 6020A	ICP-MS	Thallium
Aqueous/Solid	EPA 6020A	ICP-MS	Vanadium
Aqueous/Solid	EPA 6020A	ICP-MS	Zinc
Aqueous/Solid	EPA 7199	IC-UV	Chromium, hexavalent
Aqueous/Solid	EPA 8081B	GC-ECD	4,4'-DDD
Aqueous/Solid	EPA 8081B	GC-ECD	4,4'-DDE
Aqueous/Solid	EPA 8081B	GC-ECD	4,4'-DDT
Aqueous/Solid	EPA 8081B	GC-ECD	a-BHC
Aqueous/Solid	EPA 8081B	GC-ECD	Aldrin
Aqueous/Solid	EPA 8081B	GC-ECD	Alpha-chlordane
Aqueous/Solid	EPA 8081B	GC-ECD	b-BHC
Aqueous/Solid	EPA 8081B	GC-ECD	Chlordane, technical
Aqueous/Solid	EPA 8081B	GC-ECD	d-BHC
Aqueous/Solid	EPA 8081B	GC-ECD	Dieldrin
Aqueous/Solid	EPA 8081B	GC-ECD	Endosulfan I
Aqueous/Solid	EPA 8081B	GC-ECD	Endosulfan II
Aqueous/Solid	EPA 8081B	GC-ECD	Endosulfan sulfate
Aqueous/Solid	EPA 8081B	GC-ECD	Endrin
Aqueous/Solid	EPA 8081B	GC-ECD	Endrin aldehyde
Aqueous/Solid	EPA 8081B	GC-ECD	Endrin ketone
Aqueous/Solid	EPA 8081B	GC-ECD	Gamma-BHC (Lindane)
Aqueous/Solid	EPA 8081B	GC-ECD	Gamma-chlordane
Aqueous/Solid	EPA 8081B	GC-ECD	Heptachlor
Aqueous/Solid	EPA 8081B	GC-ECD	Heptachlor epoxide
Aqueous/Solid	EPA 8081B	GC-ECD	Methoxychlor
Aqueous/Solid	EPA 8081B	GC-ECD	Toxaphene
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1016
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1221

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1232
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1242
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1248
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1254
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1260
Aqueous/Solid	EPA 8082A	GC-ECD	PCB 1268
Aqueous/Solid	EPA 8151A	GC-ECD	2,4,5-T
Aqueous/Solid	EPA 8151A	GC-ECD	2,4,5-TP
Aqueous/Solid	EPA 8151A	GC-ECD	2,4-D
Aqueous/Solid	EPA 8151A	GC-ECD	Dicamba
Aqueous/Solid	EPA 8151A	GC-ECD	Pentachlorophenol (PCP)
Aqueous/Solid	EPA 8260C	GC-MS	1,1,1,2-Tetrachloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1,1-Trichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
Aqueous/Solid	EPA 8260C	GC-MS	1,1,2-Trichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1-Dichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1-Dichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	1,1-Dichloropropene
Aqueous/Solid	EPA 8260C	GC-MS	1,2,3-Trichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2,3-Trichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2,4-Trimethylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dibromo-3-chloropropane
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dibromoethane
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dichloroethene, total
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	1,3,5-Trimethylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,3-Dichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	1,4-Dichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	2,2-Dichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	2-Butanone (MEK)
Aqueous/Solid	EPA 8260C	GC-MS	2-Chloroethylvinyl ether

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623

Contact Name: Vicky Collom Phone: 585-288-5380

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260C	GC-MS	2-Chlorotoluene
Aqueous/Solid	EPA 8260C	GC-MS	2-Hexanone
Aqueous/Solid	EPA 8260C	GC-MS	4-Chlorotoluene
Aqueous/Solid	EPA 8260C	GC-MS	4-Isopropyltoluene
Aqueous/Solid	EPA 8260C	GC-MS	4-Methyl-2-pentanone (MIBK)
Aqueous/Solid	EPA 8260C	GC-MS	Acetone
Aqueous/Solid	EPA 8260C	GC-MS	Benzene
Aqueous/Solid	EPA 8260C	GC-MS	Bromobenzene
Aqueous/Solid	EPA 8260C	GC-MS	Bromochloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Bromodichloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Bromoform
Aqueous/Solid	EPA 8260C	GC-MS	Bromomethane
Aqueous/Solid	EPA 8260C	GC-MS	Carbon disulfide
Aqueous/Solid	EPA 8260C	GC-MS	Carbon tetrachloride
Aqueous/Solid	EPA 8260C	GC-MS	Chlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	Chloroethane
Aqueous/Solid	EPA 8260C	GC-MS	Chloroform
Aqueous/Solid	EPA 8260C	GC-MS	Chloromethane
Aqueous/Solid	EPA 8260C	GC-MS	cis-1,2-Dichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	cis-1,3-Dichloropropene
Aqueous/Solid	EPA 8260C	GC-MS	Cyclohexane
Aqueous/Solid	EPA 8260C	GC-MS	Dibromochloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Dibromomethane
Aqueous/Solid	EPA 8260C	GC-MS	Dichlorodifluoromethane (Freon 12)
Aqueous/Solid	EPA 8260C	GC-MS	Dichloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Ethylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8260C	GC-MS	Isopropylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	m- + p-Xylene
Aqueous/Solid	EPA 8260C	GC-MS	Methyl acetate
Aqueous/Solid	EPA 8260C	GC-MS	Methylcyclohexane
Aqueous/Solid	EPA 8260C	GC-MS	Methyl-tert-butyl ether (MTBE)
Aqueous/Solid	EPA 8260C	GC-MS	Naphthalene
Aqueous/Solid	EPA 8260C	GC-MS	N-butylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	N-propylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	o-Xylene

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ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623

Contact Name: Vicky Collom Phone: 585-288-5380

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260C	GC-MS	sec-butylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	Styrene
Aqueous/Solid	EPA 8260C	GC-MS	tert-butylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	Tetrachloroethene
Aqueous/Solid	EPA 8260C	GC-MS	Toluene
Aqueous/Solid	EPA 8260C	GC-MS	trans-1,2-Dichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	trans-1,3-Dichloropropene
Aqueous/Solid	EPA 8260C	GC-MS	Trichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	Trichlorofluoromethane (Freon 11)
Aqueous/Solid	EPA 8260C	GC-MS	Vinyl chloride
Aqueous/Solid	EPA 8260C	GC-MS	Xylenes, total
Aqueous/Solid	EPA 8270D	GC-MS	1,2,4,5-Tetrachlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,2-Diphenylhydrazine
Aqueous/Solid	EPA 8270D	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,4-Dichlorobenzene
Aqueous	EPA 8270D	GC-MS	1-Methylnaphthalene
Aqueous/Solid	EPA 8270D	GC-MS	2,3,4,6-Tetrachlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4,5-Trichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4,6-Trichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dimethylphenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dinitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dinitrotoluene
Aqueous/Solid	EPA 8270D	GC-MS	2,6-Dichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,6-Dinitrotoluene
Aqueous/Solid	EPA 8270D	GC-MS	2-Chloronaphthalene
Aqueous/Solid	EPA 8270D	GC-MS	2-Chlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2-Methylnaphthalene
Aqueous/Solid	EPA 8270D	GC-MS	2-Methylphenol
Aqueous/Solid	EPA 8270D	GC-MS	2-Nitroaniline
Aqueous/Solid	EPA 8270D	GC-MS	2-Nitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,2'-Oxybis(1-chloropropane)
Aqueous/Solid	EPA 8270D	GC-MS	3,3'-Dichlorobenzidine
Aqueous/Solid	EPA 8270D	GC-MS	3+4-Methylphenol

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623

Contact Name: Vicky Collom Phone: 585-288-5380

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270D	GC-MS	3-Nitroaniline
Aqueous/Solid	EPA 8270D	GC-MS	4,6-Dinitro-2-methylphenol
Aqueous/Solid	EPA 8270D	GC-MS	4-Bromophenyl-phenylether
Aqueous/Solid	EPA 8270D	GC-MS	4-Chloro-3-methylphenol
Aqueous/Solid	EPA 8270D	GC-MS	4-Chloroaniline
Aqueous/Solid	EPA 8270D	GC-MS	4-Chlorophenyl-phenylether
Aqueous/Solid	EPA 8270D	GC-MS	4-Nitroaniline
Aqueous/Solid	EPA 8270D	GC-MS	4-Nitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	Acenaphthene
Aqueous/Solid	EPA 8270D	GC-MS	Acenaphthylene
Aqueous/Solid	EPA 8270D	GC-MS	Acetophenone
Aqueous/Solid	EPA 8270D	GC-MS	Anthracene
Aqueous/Solid	EPA 8270D	GC-MS	Atrazine
Aqueous/Solid	EPA 8270D	GC-MS	Benzaldehyde
Aqueous/Solid	EPA 8270D	GC-MS	Benzidine
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(a)anthracene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(a)pyrene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(b)fluoranthene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(k)fluoranthene
Aqueous/Solid	EPA 8270D	GC-MS	Benzoic acid
Aqueous/Solid	EPA 8270D	GC-MS	Benzyl alcohol
Aqueous/Solid	EPA 8270D	GC-MS	Biphenyl
Aqueous/Solid	EPA 8270D	GC-MS	Bis(-2-chloroethoxy)methane
Aqueous/Solid	EPA 8270D	GC-MS	Bis(2-chloroethyl)ether
Aqueous/Solid	EPA 8270D	GC-MS	Bis(2-ethylhexyl)phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Butyl benzyl phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Caprolactam
Aqueous/Solid	EPA 8270D	GC-MS	Carbazole
Aqueous/Solid	EPA 8270D	GC-MS	Chrysene
Aqueous/Solid	EPA 8270D	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solid	EPA 8270D	GC-MS	Dibenzofuran
Aqueous/Solid	EPA 8270D	GC-MS	Diethylphthalate
Aqueous/Solid	EPA 8270D	GC-MS	Dimethyl phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Di-n-butylphthalate
Aqueous/Solid	EPA 8270D	GC-MS	Di-n-octyl phthalate

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Contact Name: Vicky Collom Phone: 585-288-5380

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270D	GC-MS	Fluoranthene
Aqueous/Solid	EPA 8270D	GC-MS	Fluorene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachlorocyclopentadiene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachloroethane
Aqueous/Solid	EPA 8270D	GC-MS	Indeno(1,2,3-cd)pyrene
Aqueous/Solid	EPA 8270D	GC-MS	Isophorone
Aqueous/Solid	EPA 8270D	GC-MS	Naphthalene
Aqueous/Solid	EPA 8270D	GC-MS	Nitrobenzene
Aqueous/Solid	EPA 8270D	GC-MS	N-nitrosodiethylamine
Aqueous/Solid	EPA 8270D	GC-MS	N-nitrosodimethylamine
Aqueous/Solid	EPA 8270D	GC-MS	N-nitroso-di-n-propylamine
Aqueous/Solid	EPA 8270D	GC-MS	N-nitrosodiphenylamine
Aqueous/Solid	EPA 8270D	GC-MS	Pentachlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	Phenanthrene
Aqueous/Solid	EPA 8270D	GC-MS	Phenol
Aqueous/Solid	EPA 8270D	GC-MS	Pyrene
Aqueous/Solid	EPA 9034	Titration	Sulfide, Acid Soluble
Aqueous/Solid	HPLC-METACID	HPLC	Acetic Acid
Aqueous/Solid	HPLC-METACID	HPLC	Butanoic Acid (Butyric Acid)
Aqueous/Solid	HPLC-METACID	HPLC	Lactic Acid
Aqueous/Solid	HPLC-METACID	HPLC	Propionic Acid
Aqueous/Solid	HPLC-METACID	HPLC	Pyruvic Acid

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Rochester

1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Contact Name: Vicky Collom Phone: 585-288-5380

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	(ALS SOP)	SPE Extraction	1,4-Dioxane
Aqueous	EPA 3005A	Acid Digestion	Hot Plate
Aqueous	EPA 3010A	Acid Digestion	Metals prep
Aqueous	EPA 3510C	SF Extraction	Semivolatiles, pesticides, PCBs, DRO
Aqueous	EPA 5030B	P&T	Volatiles
Solid	EPA 3050B	Acid Digestion	Metals prep
Solid	EPA 3060A	Digestion	Hexavalent chromium digestion
Solid	EPA 3541	SOX Extraction	Semivolatiles, pesticides, PCBs, DRO
Solid	EPA 5035	P&T closed	Volatiles
Aqueous/Solid	EPA 3620B	Florisil Cleanup	Semivolatiles, pesticides, PCBs
Aqueous/Solid	EPA 3660B	Sulfur Cleanup	Semivolatiles, pesticides, PCBs
Aqueous/Solid	EPA 3665A	Sulfuric Acid Cleanup	PCBs
Aqueous/Solid	EPA 9030B	Distillation	Sulfide, Acid Soluble

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

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

ISO/IEC 17025:2005 and DoD-ELAP

while demonstrating technical competence in the field of

TESTING

Refer to the accompanying Scope of Accreditation for information regarding the types of tests to which this accreditation applies.

L2223
Certificate Number

ANAB Approval

Certificate Valid: 02/22/2018-02/01/2019
Version No. 003 Issued: 02/22/2018

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 V5.1)

Katahdin Analytical Services, LLC

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
207-874-2400

TESTING

Valid to: **February 1, 2019** 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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 608; EPA 8081B	Endosulfan sulfate
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)

Non-Potable Water		
Technology	**Method**	**Analyte**
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)
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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 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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloropropene
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	1-Chlorohexane
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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C	Acrolein
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Acrylonitrile
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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
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; EPA 524.2	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; EPA 524.2	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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B/C; EPA 524.2	Tetrahydrofuran
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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	Methylcyclohexane
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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; EPA 8270C/D	Dibenz(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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Ethyl methacrylate
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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-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 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

Non-Potable Water		
Technology	**Method**	**Analyte**
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	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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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/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

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/AES	EPA 200.7; EPA 6010C/D	Beryllium
ICP/AES	EPA 200.7; EPA 6010C/D	Boron
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

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020A/B	Boron
ICP/MS	EPA 200.8; EPA 6020A/B	Cadmium
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

Non-Potable Water		
Technology	**Method**	**Analyte**
IC	EPA 300.0; EPA 9056A	Orthophosphate as P
IC	EPA 300.0; EPA 9056A	Sulfate
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

Non-Potable Water		
Technology	**Method**	**Analyte**
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 335.4; EPA 9012B; SM 4500-CN G	Amenable cyanide
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
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)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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	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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 3-Dichloropropane
GC/MS	EPA 8260B/C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	1-Chlorohexane
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	n-proplybenzene
GC/MS	EPA 8260B/C	o-Xylene
GC/MS	EPA 8260B/C	pentachloroethane
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 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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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	Methylcyclohexane
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	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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 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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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	Dibenz(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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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-2,3-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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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
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

Air		
Technology	**Method**	**Analyte**
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
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

Air		
Technology	**Method**	**Analyte**
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
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	Chlordane (tech.)
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	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)

Biological Tissue		
Technology	**Method**	**Analyte**
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)
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	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/D SIM	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D SIM	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dichlorobenzene
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

Biological Tissue		
Technology	**Method**	**Analyte**
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
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
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene

Biological Tissue		
Technology	**Method**	**Analyte**
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
ICP/AES	EPA 6010C/D	Arsenic

Biological Tissue		
Technology	**Method**	**Analyte**
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

State of Rhode Island and Providence Plantations
DEPARTMENT OF HEALTH
Certifies

LAO00333

ALS ENVIRONMENTAL ROCHESTER
BLDG 300 SUITE 360
1565 JEFFERSON RD
ROCHESTER NY 14623
Laboratory Director: JOE RIBAR

for the analysis of: **Potable Water Organic Chemistry - Potable Water Inorganic Chemistry - Non-potable Water Organic Chemistry - Non-potable Water Inorganic Chemistry -**

This certificate is issued, pursuant to Rhode Island General Laws 23-16.2 and supersedes all previous Rhode Island certificates issued to this laboratory. Certification is no guarantee of the validity of the laboratory results.

This certificate is valid only when accompanied by the certificate and list of analytes and methods for which certification has been granted based upon the following out of state certification(s):

Certifying Authority	*Certification Number*	*Expiration Date*
NY	*10145*	*04/01/2019*

ALS ENVIRONMENTAL ROCHESTER is responsible for maintaining each of the certifications listed above. Failure to notify the Laboratory Certification Officer of any change in the status of these certifications may result in the suspension or revocation of certification. Contact the Laboratory Certification Officer to verify the current certification status of this laboratory.

Nicole Alexander-Scott, MD, MPH
Director of Health

Expires: 12/30/2019

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY IN A LOCATION VISABLE TO THE PUBLIC

State of Rhode Island and Providence Plantations
DEPARTMENT OF HEALTH
Certifies

LAO00344

KATAHDIN ANALYTICAL SERVICES
600 TECHNOLOGY WAY
SCARBOROUGH ME 04074
Laboratory Director: LESLIE DIMOND

for the analysis of: **Potable Water Organic Chemistry - Potable Water Inorganic Chemistry - Non-potable Water Organic Chemistry - Non-potable Water Inorganic Chemistry -**

This certificate is issued, pursuant to Rhode Island General Laws 23-16.2 and supersedes all previous Rhode Island certificates issued to this laboratory. Certification is no guarantee of the validity of the laboratory results.

This certificate is valid only when accompanied by the certificate and list of analytes and methods for which certification has been granted based upon the following out of state certification(s):

Certifying Authority	*Certification Number*	*Expiration Date*
NELAC/NH	*200117*	*04/02/2018*
MAINE	*ME00019*	*06/01/2018*

KATAHDIN ANALYTICAL SERVICES is responsible for maintaining each of the certifications listed above. Failure to notify the Laboratory Certification Officer of any change in the status of these certifications may result in the suspension or revocation of certification. Contact the Laboratory Certification Officer to verify the current certification status of this laboratory.

Nicole Alexander-Scott, MD, MPH
Director of Health

Expires: 12/30/2018

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY IN A LOCATION VISABLE TO THE PUBLIC

Appendix F

Laboratory Standard Operating Procedures

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: Deborah J. Madeau 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 ~~5970~~ 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 noting 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 from 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

LIST OF TABLES AND FIGURES

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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

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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.

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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
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.

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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	

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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.

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0000007

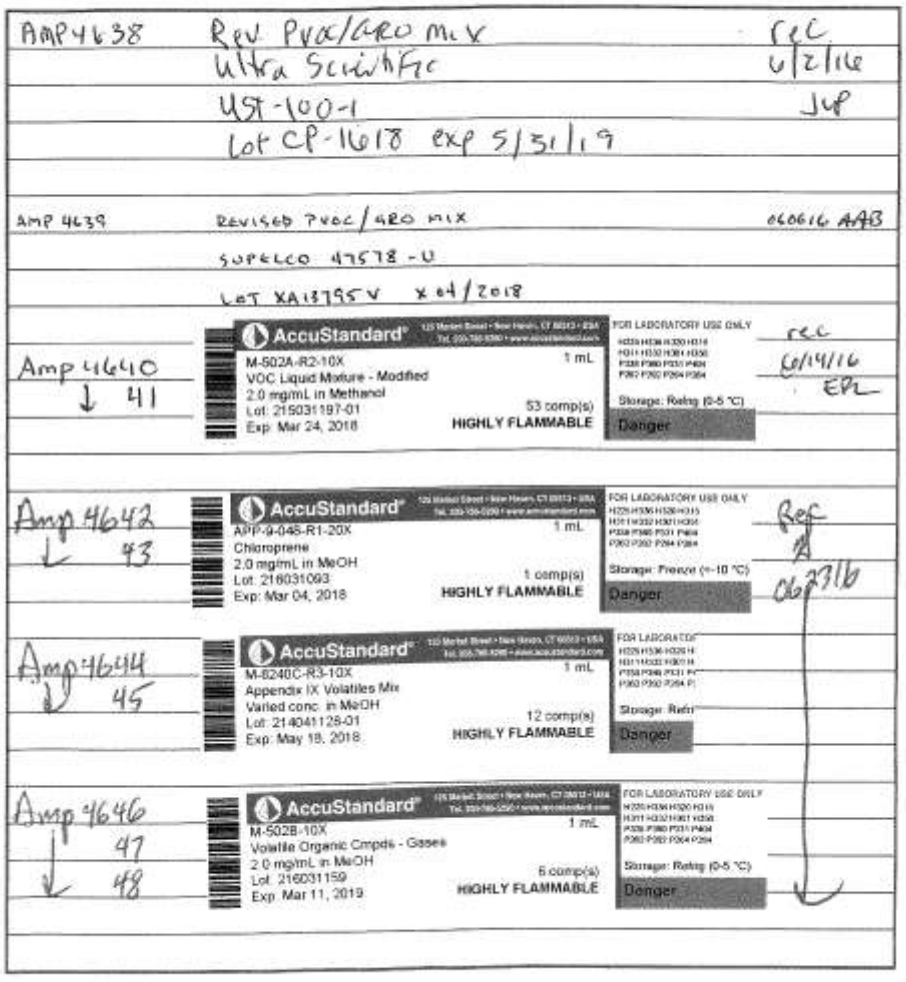
TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
STOCK STANDARDS RECEIVED
GCMS VOLATILES LABORATORY

REVIEWED BY/DATE:



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: 073116				
MEOH VOLUME (uL): 4500				
MEOH LOT #: DP303				
INITIALS: JH				
			JH 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: 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: [signature] Date: 1/31/01

QA Officer: [signature] Date: 1.31.01

General Manager: [signature] Date: 2/07/01

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.	EN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 & Table 1 to comply with Navy.	EN	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 ml minimum extract vol. & 1 ml IS is added for each 100 ml 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 1 ml to 2 ml. 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 Quickform 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 2.0 ng/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.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

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.

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 ≤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.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

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.		

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
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 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
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)

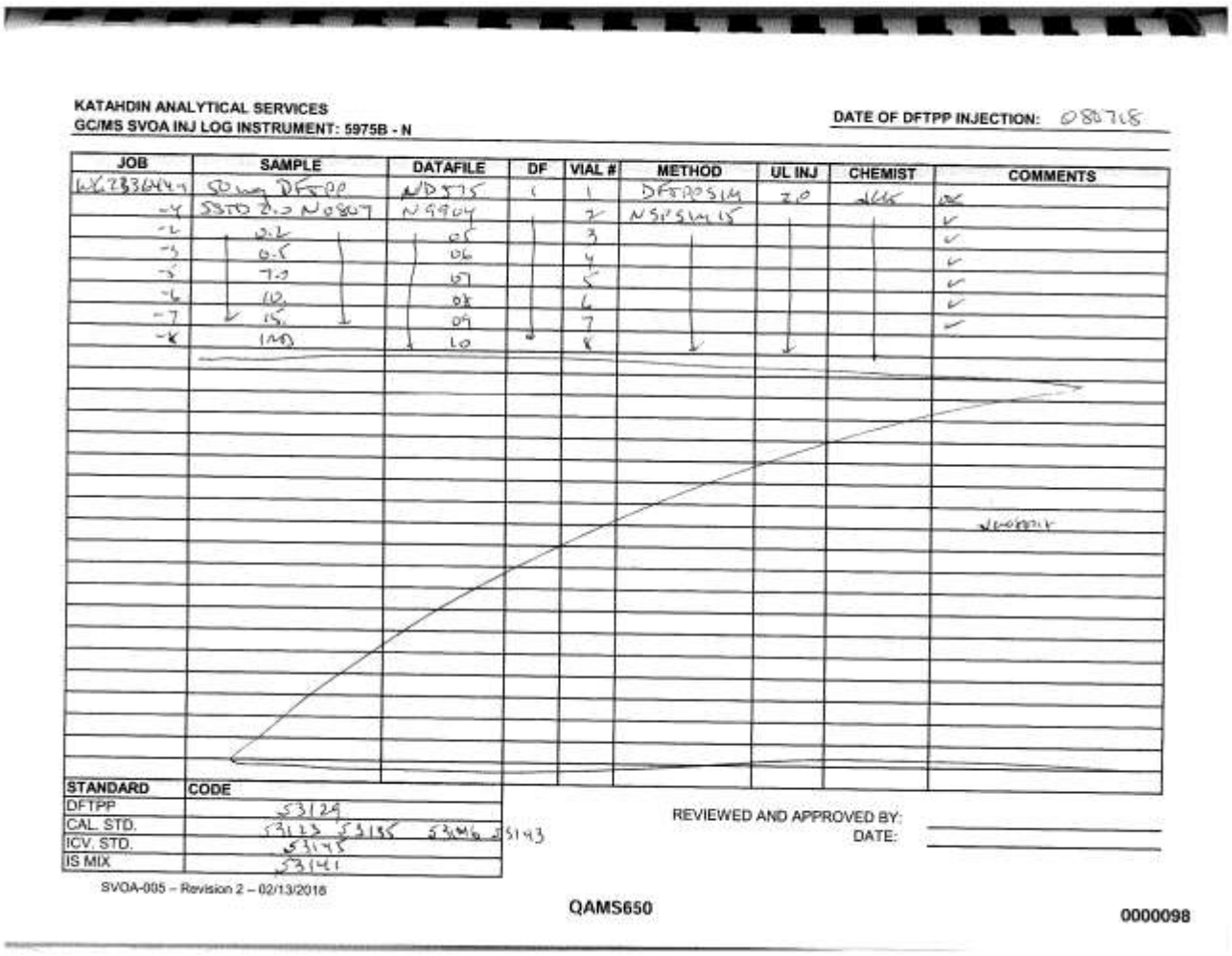
The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

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
LK233644-1	50ng DFTPP	ND575	1	1	DFTPPSIM	2.0	JLG	OK
-4	SSTD 2.0 ND809	N9904		2	NSIPSIM15			✓
-2	0.2	05		3				✓
-3	0.5	06		4				✓
-5	1.0	07		5				✓
-6	10.	08		6				✓
-7	15.	09		7				✓
-8	100	10		8				

STANDARD	CODE
DFTPP	53129
CAL. STD.	53133 53135 53136 53143
ICV. STD.	53145
IS MIX	53141

REVIEWED AND APPROVED BY:
DATE:

SVOA-005 – Revision 2 – 02/13/2018

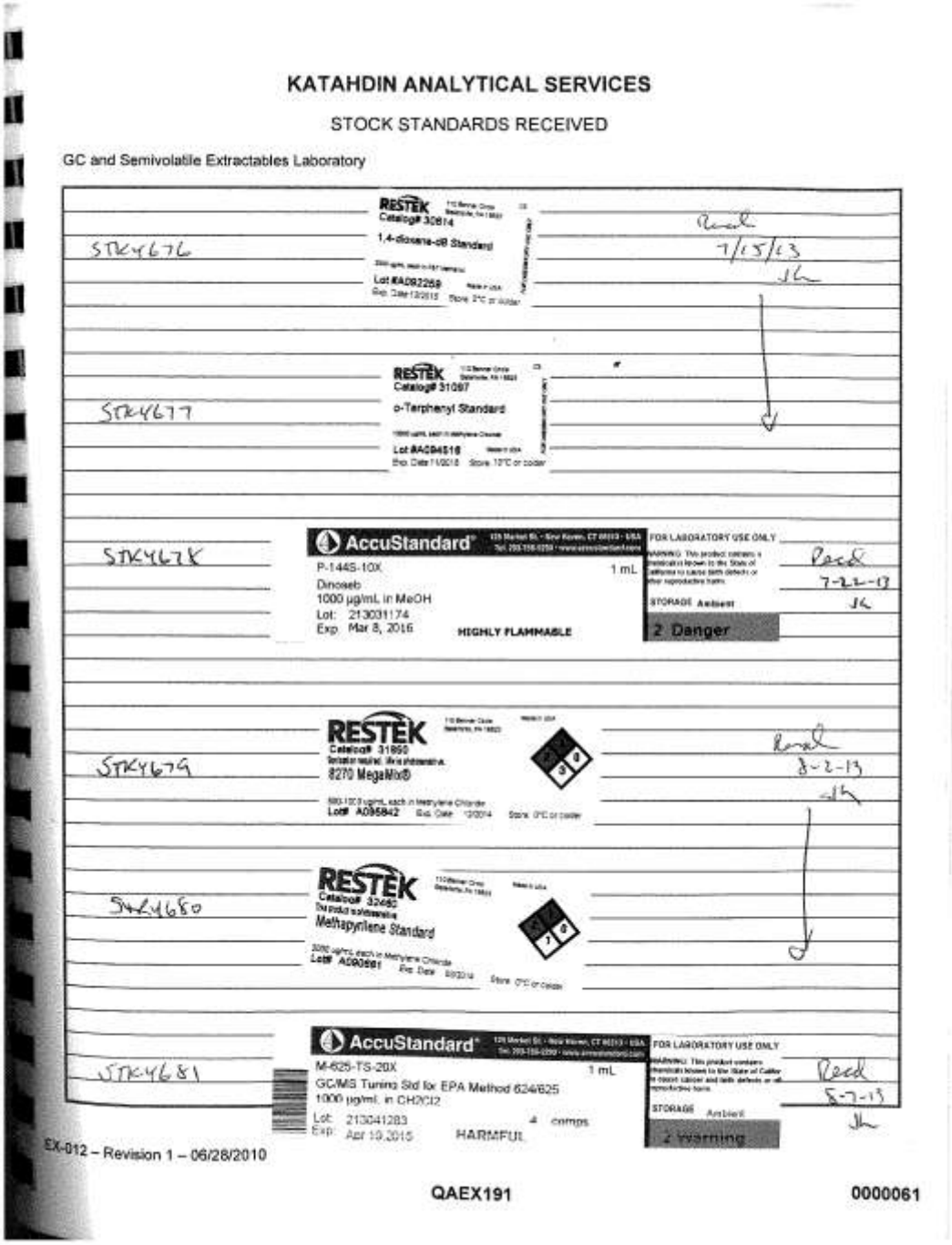
QAMS650

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# 30814, 1,4-dioxane-d8 Standard, Lot #AD82259, Exp. Date 12/2015, Store 2°C or colder — Rec'd 7/15/13 JK

STK4677 — RESTEK Catalog# 31097, o-Terphenyl Standard, Lot #AG84518, Exp. Date 1/2016, Store 10°C or colder

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 — Rec'd 7-22-13 JK

STK4679 — RESTEK Catalog# 31850, 8270 MegaMix®, Lot# A095842, Exp. Date 12/2014, Store 0°C or colder — Rec'd 8-2-13 JK

STK4680 — RESTEK Catalog# 32460, Methapyrilene Standard, Lot# A090691, Exp. Date 8/2014, Store 0°C or colder

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 10,2015, 4 comps, HARMFUL, FOR LABORATORY USE ONLY, STORAGE Ambient, 2 Warning — Rec'd 5-7-13 JK

EX-012 – Revision 1 – 06/28/2010

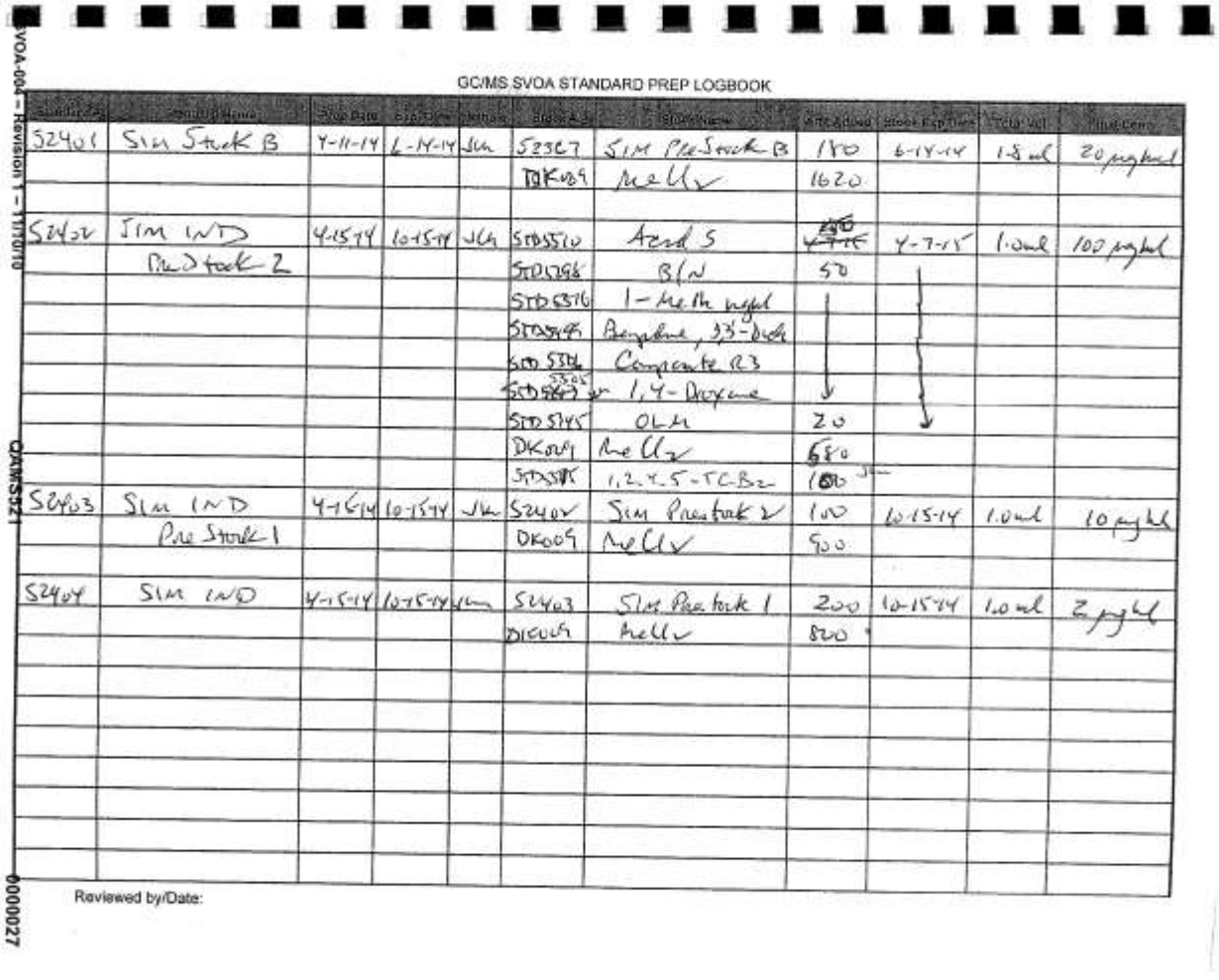
QAEX191

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

Standard ID	Standard Name	Prep Date	Expiration	Initials	Reagent ID	Reagent Name	Amt Added	Stock Expiration	Total Vol	Final Conc
S2401	SIM Stock B	4-11-14	6-14-14	JLH	53367	SIM PreStock B	180	6-14-14	1.8 ml	20 µg/ml
					DK089	MeCl2	1620			
S2402	SIM IND PreStock 2	4-15-14	10-15-14	JLH	STD5510	Acid 5	~~450~~ 400	4-7-15	1.0 ml	100 µg/ml
					STD5298	B/N	50			
					STD5316	1-Meth napht	↓	↓		
					STD5345	Acenaphthene, 33-Dich				
					STD5316	Corporate R3				
					STD5345	1,4-Dioxane				
					STD5345	OLM	20			
					DK089	MeCl2	680			
					STD5317	1,2,4,5-TCBz	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	800			

Reviewed by/Date:

CAMS5521

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: 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.	HRC	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**

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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

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 VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

Prepared By: GC/MS Group Date: 07/01

Approved By:

Group Supervisor: [signature] Date: 080307

Operations Manager: John C. Burton Date: 08/03/01

QA Officer: Deborah J. Nadeau Date: 8/6/01

General Manager: Donavan P. Kingham Date: 8/6/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8260B	Minor change to criteria in section 7.5.4. Updated calibration standard mixes.	EN	5.23.02	5.23.02
02 8260B	Reorganization of sections 4,5,6 and 7 and Tables and Figures. Added definitions and information for the new data processing system.	MRC	07.06.04	07.06.04
03 8260B	Corrected %RSD for Initial Cal. in Sect. 7.4.3 & Table 1 added wording to sec.s 6 and 8 minor changes throughout	LAD	020405	020405
04 8260B	Change Title to include all VOC's. Added instrumentation to sect. 4.4, 7.4.2 & 7.6.3 Update std. conc. sect's 5.3.2.1, 5.3.2.2, 5.3.2.3, 7.4.4, 7.5.1 & 7.5.9. Added "T" instr. and removed "O" instr. Updated cal. information, added compounds to Table 3. Updated ISand SS mixes	LAD	04/06	04/06
05	Sect. 4.7 - changed syringe sizes Sect. 5.2 - changed milli Q to Siemens Sect. 7.3 - changed reset time sect. 7.4.1 - Changed desorb time Added ICV criteria, MI references and RT window criteria.	LAD	06/07	06/07

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 4.7 - changed syringe size Sect. 5.2 - Changed Milli-Q to Siemens Sect. 7.3 - changed Run time Sect. 7.4.1 - Changed desorb time Added ICV criteria, MI references and RT window criteria	LAD	06/07	06/07
07	Updated sections 7.4.4, 7.4.6, 7.5.2, 8.1, 8.2, 8.3, 10.0 and Table 1 with DoD QSM Version 4.1 criteria.	LAD	08/09	08/09
08	Removed the "Z" instrument and added the "C" and "D" instruments. Added Table 2 - DoD QSM ver. 4.1 QC Requirements.	LAD	04/10	04/10
09	Removed Tekmar 2000 and 2016 throughout. Added heated purge and 1,4-Dioxane information. Update standard prep information, columns, instrument configuration, purge and desorb conditions to reflect current practices.	LAD	03/12	03/12
10	Throughout - minor edits for reporting through KIMS and the QSM version number.	LAD	04/13	04/13
11	~~~~	~~~~	LAD 09/13	~~~~
11	Sect. 9, Tables 4 & 5 - Updated to include Carbon Disulfide, m, p, o-Xylenes, Ethyl-benzene and methylcyclohexane. Updated Figures 1→3	LAD	09/13	09/13
12	Sect. 5 - added Standards to title. Sect. 7 - minor edits to reflect current practices. Added Table 3 - DoD QSM 5.0 QC Requirements, renumbered rest. Also fixed Table references	LAD	05/16	05/16

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
13	Added Methylene chloride and Toluene, Added new column information	LAD	09/16	09/16
14	Sect. 10 - Updated method references. Sect. 4 - added GC + MS models. Sect 5 + 7 - added soil parameters for initial calibration, IS, SS + LCS standard prep.	LAD	09/17	09/17
15 LAD 03/5/19	Removed all Tekmar references. Changed independent standard expiration to 14 days. Added 2 ICAL levels. Updated references. Removed Table 2, DoD QSM 4.2 QC requirements. Updated logbook example. Updated Table 4 and 5 w/ current analyte list	LAD	03/19	03/19

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – 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.

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Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-220-15**, titled **ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

Recipient: _____ Date: _____

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – 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 aqueous samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision, modified for selected ion monitoring to achieve lower detection levels for Volatile Organic Compounds (VOCs). This includes a heated purge procedure for the analysis of 1,4-Dioxane.

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

ANALYTICAL BATCH: 20 or fewer samples which 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. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

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.

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.

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. When analyzed directly after a calibration, the LCS doubles as the Independent Calibration Verification (ICV).

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.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

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.

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 modified for SIM. 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 volatiles organics by Method 8260, modified for SIM, 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 and 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.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. These volatile organics are then retained on a cooled trap (SP1000/tenax/silica gel medium, or equivalent) 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.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890, 6890 and 7890

4.2 Mass Spectrometers (MS): HP5972, HP5973 and HP5975.

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 components are not to be used.

4.4 Column: RTX-VMS, 30 meter, 0.25mm ID

4.5 Purge and Traps: Archon 5100 and Centurion auto samplers, and Encon concentrators.

4.6 Purge tube: 25 ml fritted purge vessel and 5ml fritted purge vessel for heated purge.

4.7 Hamilton Gastight and or SGE Gastight syringes: 5.00 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 Water Technologies. 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”.

5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

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 – Prepare a standard in purge and trap methanol containing the components listed below. The final concentration of each component is 5.0 ug/mL (25 ug/mL for 4-methyl2-pentanone and 2-Hexanone). 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. (For 1,4-dioxane heated purge, prepare a standard in purge and trap methanol containing 1,4-dioxane at a final concentration of 100ug/mL.)

Chloromethane	1,2-Dichloroethane	2-Hexanone
Vinyl Chloride	Trichloroethene	1,1,1,2-Tetrachloroethane
Trichlorofluoromethane	1,2-Dichloropropane	Isopropylbenzene
1,1-Dichloroethene	Bromodichloromethane	1,1,2,2-Tetrachloroethane
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,2,3-Trichloropropane
1,1-Dichloroethane	4-Methyl-2-pentanone	1,2,4-Trimethylbenzene
Cis-1,2-Dichloroethene	Tetrachloroethene	1,3-Dichlorobenzene
Chloroform	trans-1,3-Dichloropropene	1,4-Dichlorobenzene
Carbon Tetrachloride	Dibromochloromethane	1,2-Dichlorobenzene
1,1,1-Trichloroethane	1,3-Dichloropropane	1,2-Dibromo-3-chloropropane
Benzene	1,2-Dibromomethane	Hexachlorobutadiene
1,1,2-Trichloroethane	1,2,4-Trrichlorobenzene	Carbon Disulfide
Ethylbenzene	Xylenes	m,p-Xylene
o-Xylene	Methylcyclohexane	Methylene Chloride
	Toluene	

5.3.2.2 Laboratory Control Spike and MS/MSD Mixture - Prepare a standard independent from the calibration standards, as above containing the compounds listed below. The final concentration of each component is 5.0 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 14 days and stored in the VOA standards freezer between uses. (For 1,4-dioxane heated purge, prepare a independent standard in purge and trap methanol containing 1,4-dioxane at a final concentration of 100ug/mL.)

Chloromethane	1,2-Dichloroethane	2-Hexanone
Vinyl Chloride	Trichloroethene	1,1,1,2-Tetrachloroethane
Trichlorofluoromethane	1,2-Dichloropropane	Isopropylbenzene
1,1-Dichloroethene	Bromodichloromethane	1,1,2,2-Tetrachloroethane
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,2,3-Trichloropropane
1,1-Dichloroethane	4-Methyl-2-pentanone	1,2,4-Trimethylbenzene
Cis-1,2-Dichloroethene	Tetrachloroethene	1,3-Dichlorobenzene
Chloroform	trans-1,3-Dichloropropene	1,4-Dichlorobenzene
Carbon Tetrachloride	Dibromochloromethane	1,2-Dichlorobenzene
1,1,-Trichloroethane	1,3-Dichloropropane	1,2-Dibromo-3-chloropropane
Benzene	1,2-Dibromomethane	Hexachlorobutadiene
1,1,2-Trichloroethane	1,2,4-Trrichlorobenzene	Carbon Disulfide
Ethylbenzene	Xylenes	m,p-Xylene
o-Xylene	Methycyclohexane	Methylene chloride
	Toluene	

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5.3.2.3 Internal Standard and Surrogate spiking solution for 8260 SIM – Prepare a combined standard as above containing the compounds listed below. The final concentration of each component is 25 ug/mL. The standard must be prepared every 14 days and stored on the Auto Sampler in a pressurized vial or in the VOA standards freezer between uses.

IS	SS
Pentafluorobenzene	DBFM
1,4-Difluorobenzene	1,2-Dichloroethane-d4
Chlorobenzene-d5	Toluene-d8
1,4-Dichlorobenzene-d4	p-Bromofluorobenzene

For heated purge prepare a combined standard as above containing the compounds listed below. The final concentration of 5 and 25 ug/mL.

IS - Heated purge	SS - Heated purge
p-Bromofluorobenzene @ 5 ug/mL	Fluorobenzene @ 5 ug/mL
	1,4-Dioxane-d8 @ 25 ug/mL

For low level soils prepare a combined standard as above containing the compounds listed below. The final concentration of 5 ug/mL.

IS	SS
Pentafluorobenzene	DBFM
1,4-Difluorobenzene	1,2-Dichloroethane-d4
Chlorobenzene-d5	Toluene-d8
1,4-Dichlorobenzene-d4	p-Bromofluorobenzene

5.3.2.4 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.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are sampled into 40 mL glass VOA vials leaving no headspace. Samples are preserved with 2 drops 1:1 HCL and stored at 4°C (±2°C) until analysis.

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved.

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:

BFB Tune: VOABFBAQ.M

All other samples and standards:YSIMAXX.M

where: Y = instrument ID (Each instrument is given a unique identifier)
XX = the calibration number in chronological order

Data files:

BFB: IB_ _ _.D
where: I is the instrument ID
_ _ _ is a number in chronological order from 000 to 999

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 50ng 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-VMS, 40meter, 0.18mmID. or equivalent.	
Temperatures:	Injection port:	200°
	Transfer line:	150°
	Detector:	240°
Isothermal temperature:	150°	
Run time:	6-8 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:	100 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 8260 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB 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

7.4.1 Archon 5100, Setup/Operation: Please refer to the Archon Manual for more detailed operations for these instruments.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon, the settings for these features will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:	Purge Gas: Helium Purge Time: 11.0 ±0.1 minute Purge Flow Rate: appox. 40 mL/min Purge Temperature: Ambient (water) (80 ºC for 1,4-dioxane heated purge).
Desorb Conditions:	DesorbTemp: 250ºC Desorb Flow rate: 15mL/min Desorb Time: 1.0-2.0± 0.1 min Bake Time: 10 min Bake Temperature: 260º C

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.

The Centurion autosampler should be set up according to the specifications in the manual.

7.4.3 Initial Calibration for Method SIM 8260 and 1,4-Dioxane heated purge.

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

The GC/MS must be initially calibrated at six different levels (target compounds at concentrations listed below) to determine the linearity of response. See Section 5.3.2 for preparation of the calibration standards. Tables 4 and 5 contain a list of target compounds, internal standards, and surrogates with their defined primary quantitation ions.

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:

SIM 8260 AQ	
8260 Sim	**Cal mix @ 5.0/25 ug/mL**
VSTD0.05	1.0 uL
VSTD0.075	1.5 uL
VSTD0.10	2.0 uL
VSTD0.30	6.0 uL
VSTD0.50	10 uL
VSTD0.75	15 uL
VSTD1.00	20 uL
VSTD2.00	40 uL

1,4-Dioxane (heated purge)	
1,4-dioxane	**Cal mix @ 5.0/25 ug/mL**
VSTD2.0	2 uL
VSTD5.0	5 uL
VSTD10	10 uL
VSTD20	20 uL
VSTD50	50 uL
VSTD100	100 uL

SIM 8260 SL	
8260 Sim	**Cal mix @ 5.0/25 ug/mL**
VSTD0.50	10 uL
VSTD0.75	15 uL
VSTD1.00	20 uL
VSTD2.00	40 uL
VSTD5.00	100 uL
VSTD10.0	200 uL

The internal standard and surrogates are spiked by the autosampler.

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 4 and 5.

7.4.4. Initial Calibration Criteria

Refer to Tables 1 & 2, QC Requirements, for specific criteria that must be met for Method 8260. The percent (%) RSD for VOCs must be less than or equal to 15% (30% for heated purge).

For projects or clients requiring DoD QSM, current version, an independent calibration verification (ICV) sample must be run. The ICV must contain compounds from a different source than the ICAL. All project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met.

7.4.5. Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing the target compounds, internal standards and surrogates at the concentrations below must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB.

SIM 8260		**1,4-Dioxane (heated purge)**	
Standard	**Conc.**	**Standard**	**Conc.**
VOC Mix	0.5ug/mL	VOC Mix	20.ug/mL
SS Mix	1.0 ug/mL	SS Mix	1.0 ug/mL
IS Mix	1.0 ug/mL	IS Mix	1.0 ug/mL

The relative response factor from the continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extended ion current profile) area for the internal standard 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 the 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 VOCs is less than or equal to 15% (30% for heated purge), the continuing calibration is considered valid.

Continuing calibration check criteria must be met before sample analysis can proceed. The CV level standard analyzed as part of the initial calibration curve can be used as the continuing calibration standard, assuming all criteria are met and time is left in the twelve-hour window to analyze samples.

7.4.6. Retention Time Windows

Retention time windows for the internal standards 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 for the internal standards of the daily CV must be within 30 seconds of the midpoint

calibration standard of the most recent ICAL. The internal standard of 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.

For projects or clients requiring DoD QSM, current version, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

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 AQ LCS, 10 uL of the LCS standard mix at 5.0 ug/mL is spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 0.5 ug/L.

To prepare the SL LCS, 20 uL of the LCS standard mix at 5.0 ug/mL is spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 1.0 ug/Kg.

The autosampler adds the internal and surrogate standard to a 25mL aliquot of this preparation for analysis. The concentration of the IS/SS mix is dependant of which autosampler is being used.

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 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 1/10th the measured amount in any sample or 1/10th 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

For blanks, laboratory control samples, and client samples, the nominal limits must be met 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 response and retention time in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extended ion current profile) area for 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 the 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 by inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

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 25 mL aliquots (aqueous), 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 equal to the mid point calibration level. Acceptance criteria for MS/MSD pairs are outlined in Section 8.0.

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.

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 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume. 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.

7.6.2 Centurion/Encon unit

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 twelve-hour window. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

For All Units:

Record the sample pH in the injection logbook.

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, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

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 tables found within this SOP (Table 1 & 2). 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 internal standard 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. A "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.

7.7.1.2 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 I 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 must be completed within twelve hours of batch completion; in the majority of instances, the 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 GC/MS Department Manager, or his designee.

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 Tables 1 & 2 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 LCS Criteria

Nominal limits of 70-130% (60-140% for heated purge) are used. 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 projects or clients requiring DoD QSM, current version, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used. Laboratory limits may not be greater than $\pm$ 3 standard deviations from the mean recovery.

8.2 MS/MSD Criteria

Nominal limits of 70-130% (60-140% for heated purge) are used. 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 projects or clients requiring DoD QSM, current version, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used. Laboratory limits may not be greater than $\pm$ 3 standard deviations from the mean recovery. A MS/MSD must be analyzed with each analytical/preparatory batch per matrix. RPD must be $\leq$ 30% between the MS and MSD.

8.3 Surrogate Recovery Criteria

Surrogate Limits: (Nominal Limits) 70-130% (60-140% for heated purge).

For projects or clients requiring DoD QSM, current version, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used.

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.

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.

The 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 Method 8260 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 8260B.

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 8260C.

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 8260D.

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.

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.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260 SIM

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	Initial calibration prior to sample analysis	RSD <15% (30% for 1,4-Dioxane and surrogates).	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 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.
Calibration verification	Once per each 12 hours, prior to sample analysis	RF within 15% of average initial multi-point RF. (30% for 1,4-Dioxane and surrogates).	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	Immediately after or during data acquisition of calibration check standard	Retention time $\pm$ 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for 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 For projects requiring DoD QSM, one per preparatory batch.	No analytes of interest detected > PQL See section 7.5.2 of this SOP for additional DoD acceptance requirements.	(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.
Surrogate spike	Every sample, control, standard and method blank	Nominal 70-130% recovery (60-140% for heated purge) For projects requiring DoD QSM, DoD limits shall be used, if available. Otherwise lab 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 re-extract and analyze sample (4) If reanalysis is out, flag data

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS	One LCS per batch of 20 or fewer samples	70-130% Recovery (60-140% for heated purge)	(1) Evaluate the samples and associated QC: 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.
MS/MSD	One MS/MSD per every 20 samples	Nominal limits of 70-130% (60-140% for heated purge) are used as default limits. See also section 8.2 of this SOP.	(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 Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstration of Proficiency	Once per analyst initially; 4 reps of LCS and annually thereafter	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 VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – 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
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 VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – 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
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.

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – 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 (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 VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-220-15	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	(4) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) 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	

TABLE 4

CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,1-Trichloroethane	97	99, 61
1,1,2,2-Tetrachloroethane	83	131, 85
1,1,2-Trichloroethane	83	97, 85
1,2,3-Trichloropropane	75	77, 110
1,1-Dichloroethane	75	53, 77
1,1-Dichloroethene	96	61, 63
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-chlororpropane	75	155, 157
1,2-Dibromomethane	107	109, 188
1,2-Dichlorobenzene	146	111, 148
1,2-Dichloroethane	62	98
1,2-Dichloropropane	63	112
1,3-Dichlorobenzene	146	111, 148
1,3-Dichloropropane	76	78
1,4-Dichlorobenzene	146	111, 148
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58
Benzene	78	77, 51
Bromodichloromethane	83	85, 127
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chloroform	83	85
Chloromethane	50	52
cis-1,2-Dichloroethene	96	61, 98
cis-1,3-Dichloropropene	75	77, 39
Dibromochloromethane	129	127
Ethylbenzene	91	106
Hexachlorobutadiene	225	223, 227
Isopropylbenzene	105	120
Methylene chloride	84	86, 49
m,p-Xylene	91	106
Methylcyclohexane	83	55, 98
o-Xylene	91	106
Tetrachloroethene	164	129, 131
Toluene	92	91
Trans-1,2-Dichloroethene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
Trichloroethene	95	97
Trichlorofluoromethane	101	103
Vinyl Chloride	62	64
Xylenes	91	106
Bromomethane	94	96
Chloroethane	64	66
Methyl tert-butyl ether	73	57, 41
Acrylonitrile	53	52, 51
Dibromomethane	93	95, 174
Chlorobenzene	112	77, 114
Bromoform	173	175, 254
Naphthalene	128	-

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 5

INTERNAL STANDARD

INTERNAL STANDARD	TARGET ANALYTE
Pentafluorobenzene	Chloromethane
	Vinyl Chloride
	Bromomethane
	Chloroethane
	Trichlorofluoromethane
	1,1-Dichloroethene
	Methylene chloride
	trans-1,2-Dicholoethene
	Methyl tert-butyl ether
	1,1-Dichloroethane
	Acrylonitrile
	Cis-1,2-Dichloroethene
	Chloroform
	1,1,1-Trichloroethane
	Carbon disulfide
	Methylcyclohexane
INTERNAL STANDARD	**TARGET ANALYTE**
1,4-Difluorobenzene	Carbon Tetrachloride
	Benzene
	1,2-Dichloroethane
	Trichloroethene
	Dibromomethane
	1,2-Dichloropropane
	Bromodichloromethane
	cis-1,3-Dichloropropene
	4-Methyl-2-pentanone
	Trans-1,3-Dichloropropene
	1,2-Dibromomethane
	1,1,2-Trichloroethene
	Toluene
INTERNAL STANDARD	**TARGET ANALYTE**
Chlorobenzene-d5	Tetrachloroethene
	Dibromochloromethane
	1,3-Dichloropropane
	2-Hexanone
	1,1,1,2-Tetrachloroethane
	Chlorobenzene
	Ethylbenzene
	m+p-Xylene
	o-Xylene
INTERNAL STANDARD	**TARGET ANALYTE**
1,4-Dichlorobenzene-d4	1,1,2,2-Tetrachloroethane
	1,2,3-Trichloropropane
	Isopropylbenzene
	1,2,4-Trimethylbenzene
	Bromoform
	1,3-Dichlorobenzene
	1,4-Dichlorobenzene
	1,2-Dichlorobenzene
	1,2-Dibromo-3-chlororpropane

	Hexachlorobutadiene
	1,2,4-Trichlorobenzene
	Naphthalene

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

W6

KATAHDIN ANALYTICAL SERVICES

GCMS-D INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 3-11-19 9:35

Method - Check One:	8260	8260 - 10 mL	8260 SIM ✓	624	524
STD IDs: * BFB STD: V1412	CAL STD: V1401	GAS STD: —	XTRAs STD: —	LCS/MS STD: V1401	
SURR STD: V1390	IS STD: V1390				

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	Prep Method 5030	Prep Method 5035	Prep Method 1311	Criteria KAS	Criteria DoD	Criteria QAPP	pH Paper Lot #: Y/N	pH Paper Lot #: ANALYST	pH	KI Paper Lot #: TRC	COMMENTS
WG248398 -3	DB436	—	—	VOABFBAQ	✓						Y	JP/HC	N/A	N/A	
Vstd0.5 -4	D5856	1	1	DSZSIMDMH1							Y				
LCS -1	57		2								Y				
VBLKA	58		3								N				6.2 Llc
VBLK13 -2	59		4								Y		↓		
SM1849 -2RA C	60		5						5.1		Y		<2		
-1ORA C	61		6								Y		<2		
SM1905 -1RA H	62		7								Y		<2		
SM1849 -8MSRA H	63		8								Y		<2		
-8MSDRA G	64		9						↓		Y		<2		
CCV	65		10								Y		N/A		1.0 ppb ✓
CCV	66		11								N				matrix 8
CCV	67		12								N				↓
Rinse	↓ 68	↓	13	✓	↓						—	↓	↓	↓	

3-12-19

* Refer to page 1 for standard preparation instructions.

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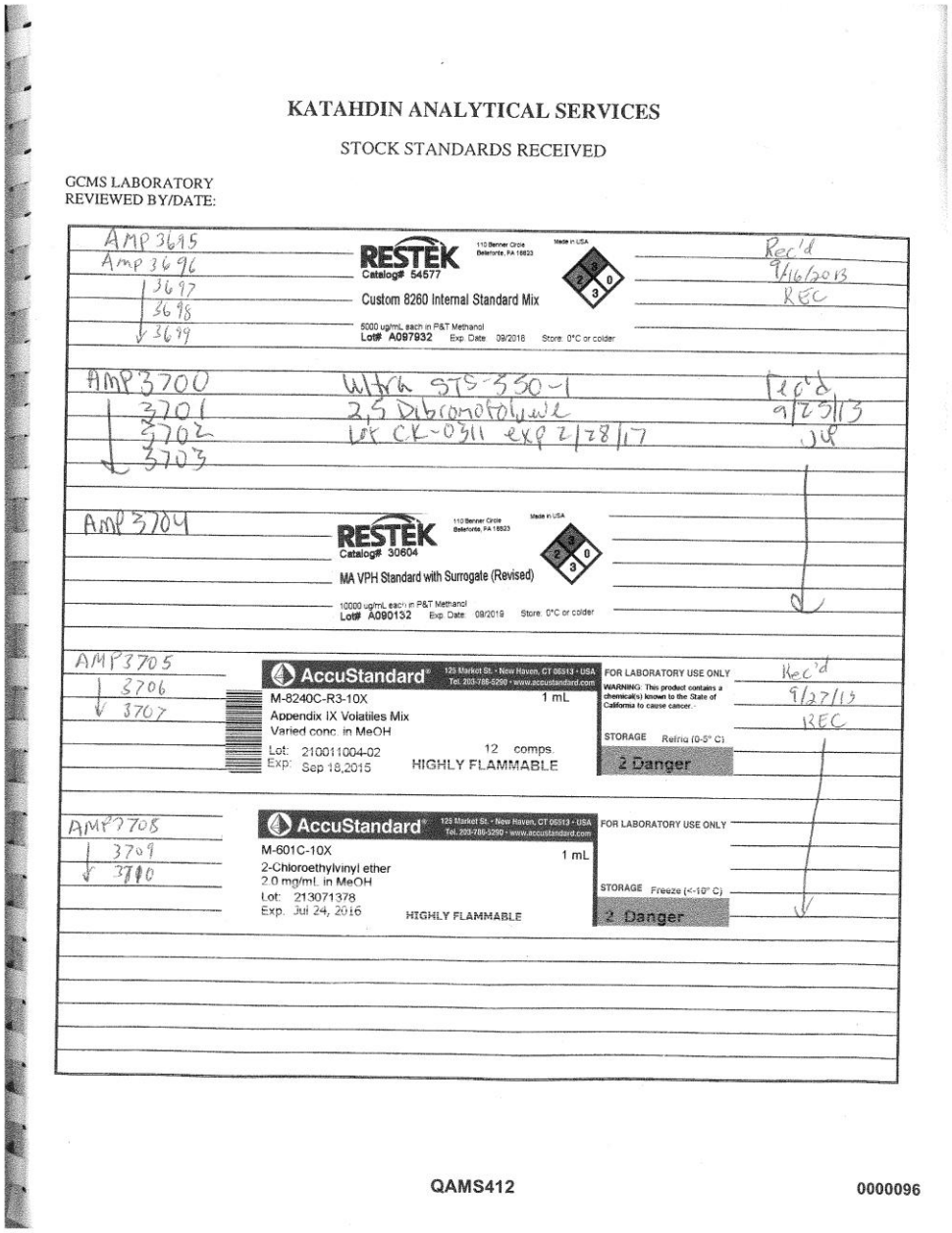
QAMS669

0000090

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP3695
AMP3696
3697
3698
3699

RESTEK 110 Benner Circle Bellefonte, PA 16823 Made in USA
Catalog# 54577
Custom 8260 Internal Standard Mix
5000 ug/mL each in P&T Methanol
Lot# A097932 Exp. Date: 09/2016 Store: 0°C or colder

Rec'd 9/16/2013 REC

AMP3700
3701
3702
3703

Ultra STS-330-1
2,5 Dibromotoluene
Lot CK-0311 exp 2/28/17

Rec'd 9/23/13 JG

AMP3704

RESTEK 110 Benner Circle Bellefonte, PA 16823 Made in USA
Catalog# 30604
MA VPH Standard with Surrogate (Revised)
10000 ug/mL each in P&T Methanol
Lot# A090132 Exp. Date: 09/2019 Store: 0°C or colder

AMP3705
3706
3707

AccuStandard® 125 Market St. - New Haven, CT 06513 - USA Tel. 203-786-5290 - www.accustandard.com
M-8240C-R3-10X 1 mL
Appendix IX Volatiles Mix
Varied conc. in MeOH
Lot: 210011004-02 12 comps.
Exp: Sep 18,2015 HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.
STORAGE Refrig (0-5° C)
2 Danger

Rec'd 9/27/13 REC

AMP3708
3709
3710

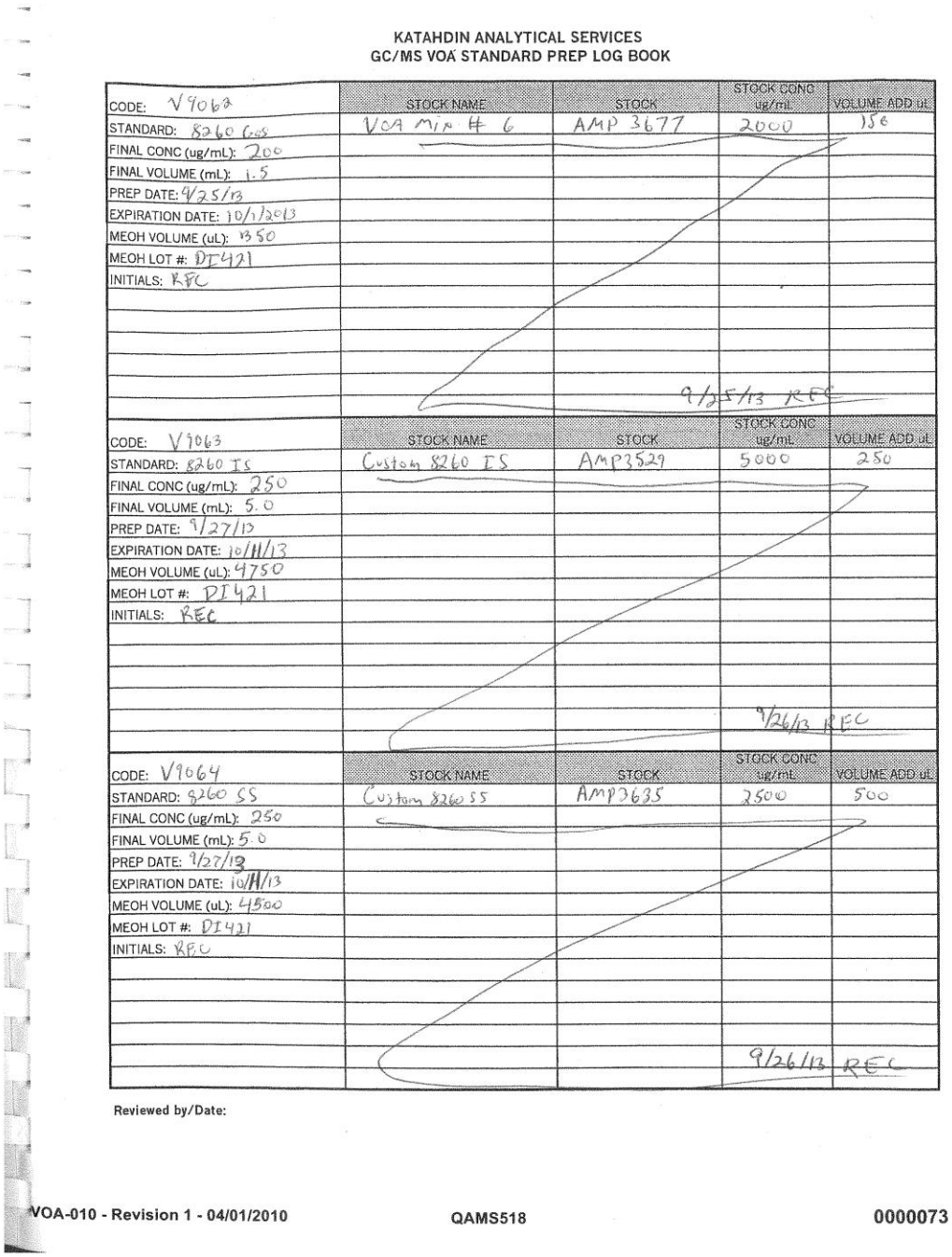
AccuStandard® 125 Market St. - New Haven, CT 06513 - USA Tel. 203-786-5290 - www.accustandard.com
M-601C-10X 1 mL
2-Chloroethylvinyl ether
2.0 mg/mL in MeOH
Lot: 213071378
Exp. Jul 24, 2016 HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
STORAGE Freeze (<-10° C)
2 Danger

QAMS412 0000096

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V9063	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 GCS	VOA Mix # 6	AMP 3677	2000	150
FINAL CONC (ug/mL): 200				
FINAL VOLUME (mL): 1.5				
PREP DATE: 9/25/13				
EXPIRATION DATE: 10/1/2013				
MEOH VOLUME (uL): 1350				
MEOH LOT #: DI421				
INITIALS: RFC				
		9/25/13 REC		
CODE: V9063	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 IS	Custom 8260 IS	AMP3529	5000	250
FINAL CONC (ug/mL): 250				
FINAL VOLUME (mL): 5.0				
PREP DATE: 9/27/13				
EXPIRATION DATE: 10/11/13				
MEOH VOLUME (uL): 4750				
MEOH LOT #: DI421				
INITIALS: REC				
			9/26/13 REC	
CODE: V9064	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 SS	Custom 8260 SS	AMP3635	2500	500
FINAL CONC (ug/mL): 250				
FINAL VOLUME (mL): 5.0				
PREP DATE: 9/27/13				
EXPIRATION DATE: 10/11/13				
MEOH VOLUME (uL): 4500				
MEOH LOT #: DI421				
INITIALS: RFC				
			9/26/13 REC	

Reviewed by/Date:

VOA-010 - Revision 1 - 04/01/2010 QAMS518 0000073

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.	EN	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.	LAN	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.	LAN	01/12	01/12
04	Sect. 1 & 7 - Changed reporting through quickform to reporting through KIMS. Sect. 8 - Added Marginal Exceedance criteria.	LAN	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.	LAN	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.

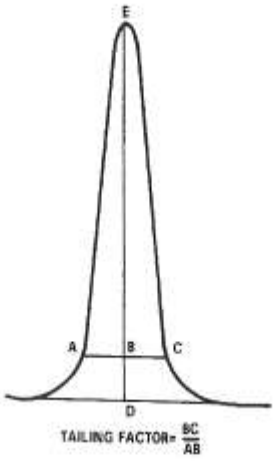
If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should 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:



Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

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

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
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.

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
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

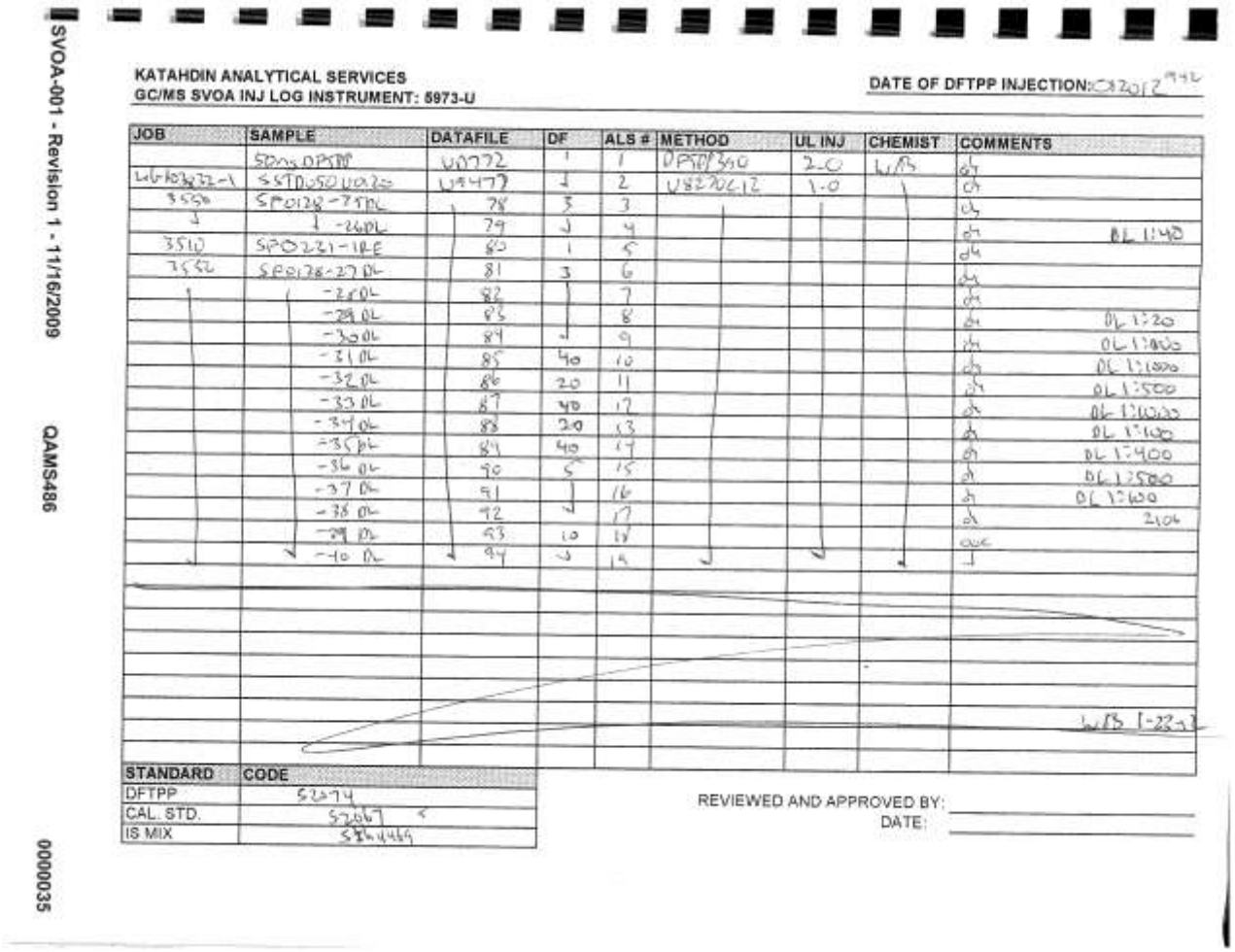
RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION (CONT.)

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: 3/20/12

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	SDG DFTPP	U0772	1	1	DFTPP350	2.0	WB	
L1603872-1	SSTD050 U0773	U0777	1	2	U8270C-12	1.0		
3550	SP0128-7 5DL	78	5	3				
1	1 -2 5DL	79	1	4				RL 1:40
3510	SP0231-1RE	80	1	5				
3552	SP0128-2 7 DL	81	3	6				
	-2 5DL	82		7				
	-28 DL	83		8				DL 1:20
	-30 DL	84		9				DL 1:100
	-31 DL	85	40	10				DL 1:1000
	-32 DL	86	20	11				DL 1:500
	-33 DL	87	40	12				DL 1:1000
	-34 DL	88	20	13				DL 1:100
	-35 DL	89	40	14				DL 1:400
	-36 DL	90	5	15				DL 1:500
	-37 DL	91	1	16				DL 1:600
	-38 DL	92	1	17				2.04
	-39 DL	93	10	18				DUP
	-40 DL	94	1	19				
								WB 1-23-12

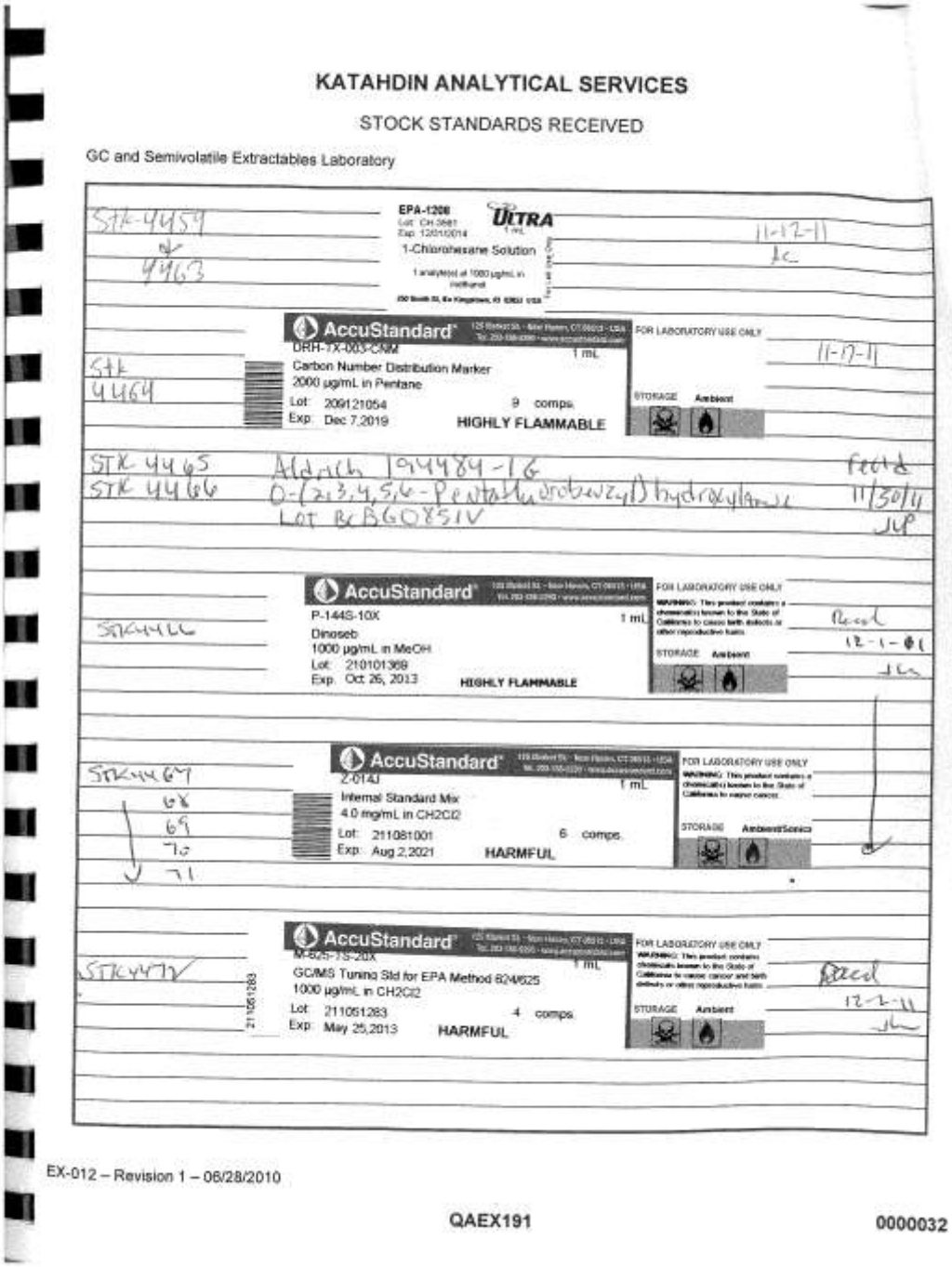
STANDARD	CODE
DFTPP	52674
CAL. STD.	52667
IS MIX	52669

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



EX-012 – Revision 1 – 06/28/2010

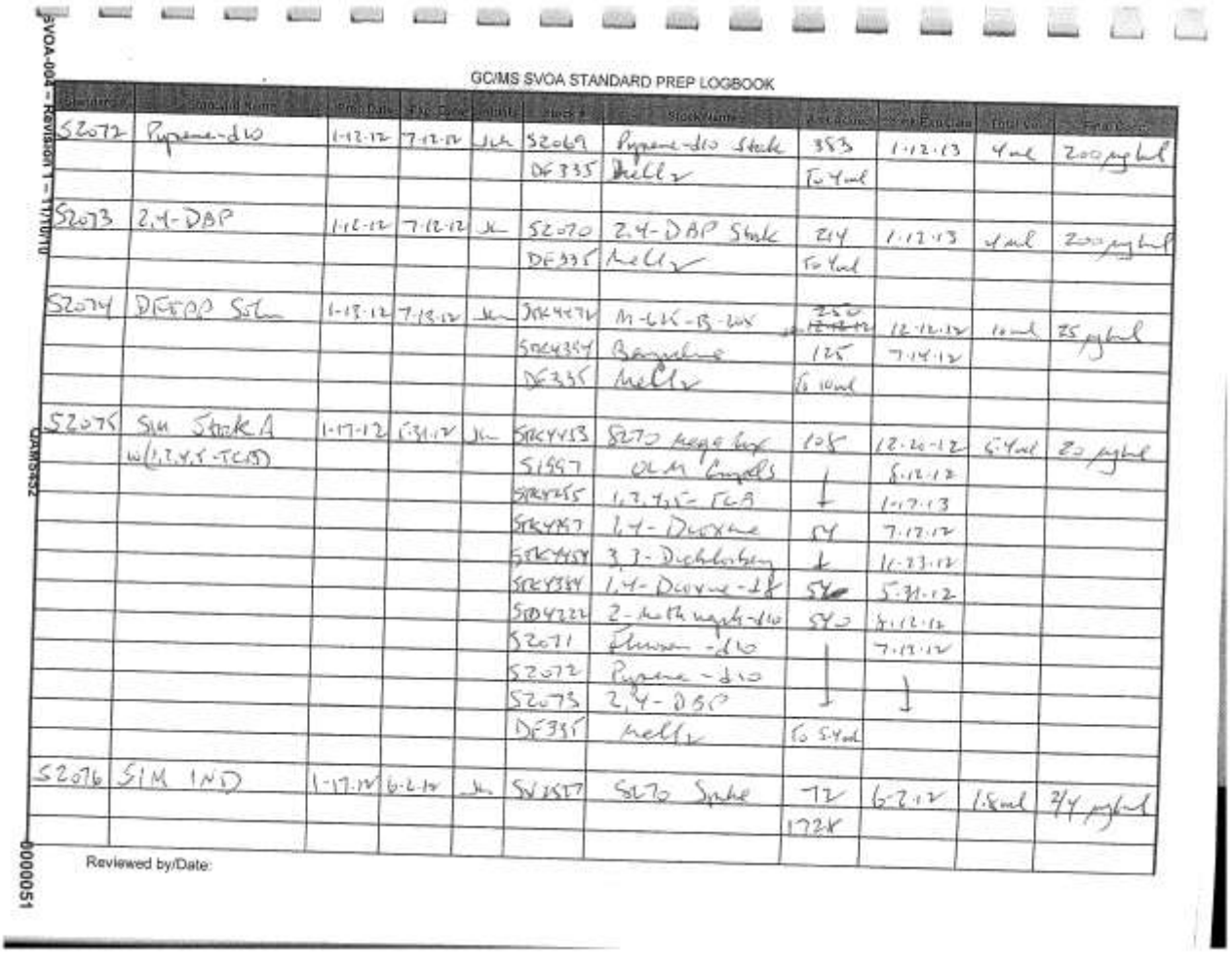
QAEX191

0000032

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 ID	Standard Name	Prep Date	Exp Date	Initials	Stock #	Stock Name	# of Drops	Used Previously	Final Vol	Final Conc
S2072	Pyrene-d10	1-12-12	7-12-12	JLH	S2069	Pyrene-d10 Stock	353	1-12-13	4 mL	200 µg/mL
					DF335	MeCl2	To 4 mL			
S2073	2,4-DBP	1-12-12	7-12-12	JLH	S2070	2,4-DBP Stock	214	1-12-13	4 mL	200 µg/mL
					DF335	MeCl2	To 4 mL			
S2074	DFTPP Stock	1-13-12	7-13-12	JLH	SRK4472	M-6K-B-2K	~~250~~ 200	12-12-12	10 mL	25 µg/mL
					SRK4354	Benzidine	125	7-14-12		
					DF335	MeCl2	To 10 mL			
S2075	SIM Stock A w/1,2,4,5-TCB	1-17-12	7-31-12	JLH	SRK4413	S270 Acid Mix	125	12-20-12	5 mL	20 µg/mL
					S1597	ULM Cmpds	↓	8-12-13		
					SRK4455	1,2,4,5-TCB	↓	1-17-13		
					SRK4457	1,4-Dioxane	5K	7-17-12		
					SRK4458	3,3-Dichlorobenz	↓	1-23-13		
					SRK4384	1,4-Dioxane-d8	5K	5-31-12		
					SRK4224	2-Methylnaph-d10	5K	8-12-12		
					S2071	Fluoren-d10	↓	7-17-12		
					S2072	Pyrene-d10	↓	↓		
					S2073	2,4-DBP	↓	↓		
					DF335	MeCl2	To 5 mL			
S2076	SIM IND	1-17-12	6-2-12	JLH	SV1517	S270 Spike	72	6-7-12	1.8 mL	2/4 µg/mL
							1722			

Reviewed by/Date:

00000051

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

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

Prepared By: _____ Date: 2/2/06

Approved By:

Department Manager: _____ Date: 2/2/06

Operations Manager: Deborah J. Nadeau Date: 2/2/06

QA Officer: Leslie Dimond Date: 2-2-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Section 7.9 removed surrogate reference. Added information to Section 1.4 for headspace vial disposal. Minor changes throughout.	LAD	03/07	03/07
02	Added LCS to definitions and Sect. 8.2 Added N_2 Purging to sect.s 7.1 and 7.2	LAD	01/08	01/08
03	Removed all references to method SW846 3810. Updated Figure 1 - Logbook Page.	LAD	06/09	06/09
04	Added Independant calibration verification to sections 1.0, 7.4, 7.4.2, 8.2 and Table 1. Section 7.4 and 7.4.1 - Corrected tables and formula. Changed CAR to NCR. Updated References.	LAD	09/10	09/10
05	Removed Surrogate reference in Section 7.9. Added references to Section 10.	LAD	07/11	07/11

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 1 and 7 – Removed Quickform reference and added reporting through KIMS. Sect. 8 – Removed annual MDL requirement. Sect. 9 – Added MDL. LOD and LOQ information.	LAD	05/13	05/13
07	Sect. 9- Updated calibration high point. Updated references and logbook example.	LAD	03/19	03/19

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

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-336-07, titled “Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Technique EPA SOP RSK-175”.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-336-07, titled “Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Technique EPA SOP RSK-175”.

Recipient: _____ Date: _____

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of aqueous samples for Methane, Ethane and Ethene by RSK-175, as performed by Katahdin Analytical Services, Inc. including sample preparation, sample analysis, data review, standard preparation and instrument calibration.

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.

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.

CALIBRATION STANDARD (WORKING STANDARD): A standard prepared from the stock standard that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION VERIFICATION (ICV): The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. A reagent water blank is spiked with the ICV Standard and analyzed immediately following a calibration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) of interest 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 LCS is obtained from a source different from the source of the calibration standards.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock standards of certain analytes are added to a sample matrix prior to sample 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. MS/MSD's are spiked with the same standard as the LCS.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

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: A data acquisition system that is used to collect chromatographic data. The system 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.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of Methane Ethane, Ethene by method RSK-175. 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 the Methane Ethane, Ethene by method RSK-175 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

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 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. Used headspace vials are disposed of in the “P” waste satellite accumulation area located under GC05.

2.0 SUMMARY OF METHOD

A water sample is collected in a 40 mL VOA bottle using a Teflon faced septum and cap. In the laboratory, a 5 mL aliquot of a sample is injected into a vial and placed onto a headspace autosampler where each sample is shaken and heated prior to injection. The analyte(s) present in the samples will partition between the water and the gas phase according to Henry’s Law. The autosampler pressurizes the sample in order to inject 1 mL of headspace onto a gas chromatographic column where the gaseous compounds are separated and detected by a flame ionization detector (FID).

3.0 INTERFERENCES

The sample integrity is compromised if the sample vial contains headspace prior to sample preparation. The presence of headspace in the sample vial is notated in the laboratory narrative.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph: GC Hewlett Packard 5890 series I or II connected to the Turbochrom data system, or equivalent

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4.2 Headspace Analyzer: Agilent Technologies G1888 Network Headspace Analyzer

4.3 Column: 80/100 mesh Poropak Column 6ft x 1/8”

4.4 Detector: Flame Ionization Detector (FID)

4.5 Data System: A data system which allows the continuous acquisition of data throughout the duration of the chromatographic program must be interfaced to the GC. The data system must be capable of storing and re-integrating chromatographic data and must be capable of determining peak areas using a forced baseline projection. All data editing will be reviewed by the Department Manager or qualified designee before samples are reported.

4.6 Headspace Syringes: various sizes for preparing standards and injecting samples

4.7 5 mL Leur Lock gas-tight syringe with liquid needle

4.8 10 mL headspace vials

4.9 40 mL VOA vials

4.10 Refrigerator for storage of samples

4.11 pH strips (pH 1 – 14 range)

4.12 Tedlar Bags

4.13 Septum cap and crimper

4.14 Brinkmann Pipetter, volume up to 5 mL

5.0 REAGENTS

5.1 Ultra high purity Nitrogen

5.2 Ultra high purity Hydrogen

5.3 Laboratory Reagent Grade Water: Milli-Q, or equivalent

5.4 Certified Gas Standards, Scotty or equivalent

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected into 40 mL VOA vials. The vials have been preserved with 1:1 HCL prior to collection. Care should be taken so there are no air bubbles in the vials.

Samples are stored at 4 (±2) °C until time of analysis. Samples must be analyzed within 14 days of sampling. Unpreserved samples must be analyzed within 7 days of sampling.

7.0 PROCEDURES

7.1 Preparing Samples for Analysis:

Allow samples to warm to room temperature prior to preparation. Inspect all VOA vials for bubbles and notate any bubbles in the logbook.

Purge all headspace vials with nitrogen for approximately ten seconds prior to injecting them with sample. The empty headspace vials are purged and then capped. The nitrogen line is located between the headspace sampler and the GC05 oven.

Using a 5 mL Leur lock syringe, pull up 5 mL of Nitrogen from a Tedlar bag. While inverting a VOA vial push the syringe through the vial septa. Insert a second 5 mL syringe into the VOA vial. By injecting the 5 mL of nitrogen, 5 mL of sample will be displaced into the second syringe. Take the syringe containing the sample aliquot out of the VOA vial and immediately inject the aliquot through the headspace vial septa. The sample is now ready to be loaded onto the autosampler.

If a dilution is required in order to bring the sample within range of the calibration curve, the sample is prepared as above, but less than 5 mL of sample is injected into the vial. An aliquot of laboratory reagent grade water is used to bring the liquid volume to 5 mL. The laboratory reagent grade water is purged with the nitrogen line for approximately ten seconds before capping the headspace vials and adding sample. The amounts of sample and water are based on the factor needed to bring the sample within range of the upper half of the calibration curve. The amount of sample and water are notated in the logbook and the proper dilution factor is applied to the final result.

7.2 Standards Preparation

Using a pipetter, inject 5 mL DI water into a headspace vial, purge with nitrogen for approximately ten seconds and crimp the top. A gas standard is then injected into the vial. The standards are calibrated as µg/mL of water.

7.3 GC Conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Nitrogen Carrier:	20 mL/min.
Ultra Zero Air:	400 mL/min.
Hydrogen:	40 mL/min.
Injector Temp.:	200°
Detector Temp.:	250°
Oven Ramp:	40 hold 1 min; 10 degrees/min to 100°
Run time:	7 min
Injection size:	1 mL
Column head pressure:	14 psi

7.4 Calibration

The GC system is calibrated using the external standard calibration procedure. A six-point (5-point minimum) calibration is prepared according to the concentrations listed below. When the calibration curve is run an independent check standard should also be run to validate the curve.

Methane MW=16

Vol of Std inj. (µL)	Std Conc (ppm)	Std. Injected into Vial (µg)	Water Volume (mL)	Water Conc. (µg/mL)
38	1000	0.025	5.0	0.005
200	1000	0.133	5.0	0.027
500	1000	0.333	5.0	0.067
1000	1000	0.665	5.0	0.133
5000	1000	3.326	5.0	0.665
9000	1000	5.986	5.0	1.197

Ethene MW=28

Vol of Std inj. (µL)	Std Conc (ppm)	Std. Injected into Vial (µg)	Water Volume (mL)	Water Conc. (µg/mL)
38	1000	0.044	5.0	0.009
200	1000	0.233	5.0	0.047
500	1000	0.582	5.0	0.116
1000	1000	1.164	5.0	0.233
5000	1000	5.819	5.0	1.164
9000	1000	10.476	5.0	2.095

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Ethane MW=30

Vol of Std inj. (µL)	Std Conc (ppm)	Std. Injected into Vial (µg)	Water Volume (mL)	Water Conc. (µg/mL)
38	1000	0.047	5.0	0.009
200	1000	0.249	5.0	0.050
500	1000	0.624	5.0	0.125
1000	1000	1.247	5.0	0.249
5000	1000	6.236	5.0	1.247
9000	1000	11.224	5.0	2.245

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height or area for each compound. A calibration curve can be prepared in Target using the peak height or area against the concentration of the standard. An average calibration applying a first order polynomial equation is used to prepare the curve.

7.4.1 Calculating the concentration of the calibration standard (x)

$$\text{µg std. injected into vial} = \frac{(\text{ppmv of std.})(\text{MW of gas})(\text{mL injected})}{24055}$$

$$\text{conc.} = \frac{\text{µg std. injected into vial}}{\text{amount of water in vial (mL)}}$$

7.4.2 An Independent Calibration Verification Standard (ICV) is analyzed immediately after calibration, before any samples are analyzed.

7.5 Retention Time Study

Three injections are made of all the analytes throughout the course of a 72-hour period.
A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

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 weight heavily in the interpretation of chromatograms. Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

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7.6 Sample Analysis

Each vial may only be analyzed once. If a second analysis is required, the sample must be re-prepped. The samples and standards are loaded onto the autosampler, which heats and shakes the vials. The autosampler then pressurizes the sample to fill the 1 mL sample loop. The 1 mL headspace sample is then injected into the GC instrument.

Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with instrument calibration as listed in section 7.4 followed by sample aliquots interspersed with mid-concentration calibration standards.

Before any samples are analyzed the instrument must be calibrated by analyzing a five-point (minimum) calibration or a mid-concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 30%. Each sample analysis must be bracketed with an acceptable initial calibration and a closing CV, or an opening CV and a closing CV. The calibration standard must also be injected at intervals of not less than once every twenty samples (or every 12 hours), whichever is more frequent, and at the end of the analysis sequence.

If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. All samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >30%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the analyses for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample analysis, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 30% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

Absolute retention time windows are established using the mid-point of the window of that day if after analyzing the mid-point it is determined that one or more of the analytes fall outside of the previously established absolute retention time window. The daily retention time window equals the mid-point ±three times the standard deviations.

The identification of methane, ethane and ethene 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.

If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

If the amount recovered is not detectable or below the PQL, then the compound is not considered to be present in the sample and is reported as <PQL.

When a GC system is determined to be out of control because either a CV can not 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, 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 FID or an electronic board, this information is written in the instrument maintenance logbook.

7.7 Calculations

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 when the file is processed through the appropriate calibrated method.

Concentration (µg/L) = $[(C)\ (0.005L)/(V_s)]\ (1000)$

Where: C = Concentration calculated by Target in µg/mL
V_s = Volume of sample purged in L

7.8 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 in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or reextracted.
These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.

- Chromatography: cleanups, manual integration.

- Target compound detection: quantitation and 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 7.10.

7.9 Chromatography

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.

In Target Review, 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), manual integration is performed in Target Review. An “M” qualifier will automatically be printed on the quantitation report summary indicating that a manual integration was performed. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, “Manual Integration,” current revision.

7.9.1 Target Compound Detection

The chromatogram is evaluated to determine if a target analyte is indicated. The concentration of the analyte(s) is then evaluated to determine if it is above the PQL and within the calibration range.

7.10 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.

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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 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 Nonconformance Report (NCR) describing the problem, suspected cause and final resolution. A NCR must be initiated as soon as possible.

8.1 Continuing Calibration Verification (CV)

A mid-level concentration standard is analyzed daily prior to sample analysis. The calibration standard must also be injected at intervals of not less than once every twenty samples (or every 12 hours), whichever is more frequent, and at the end of the analysis sequence. The acceptance criterion is ±30% of the expected value. If response of a compound, in the opening CV, fails to met the criterion, the system is checked, the standard reprepped and analyzed. In the event the criterion cannot be met, the instrument is recalibrated.

8.2 Independent Calibration Verification (ICV)

An ICV is a mid-level concentration standard using a source different from the source of the calibration standards. This can include a different lot from the same manufacturer. An ICV is analyzed immediately following a curve. The acceptance criterion is ±30% of the expected value. If the ICV fails to meet this criterion, the

system is checked, and another ICV is analyzed. In the event the criterion cannot be met, the instrument is recalibrated.

8.3 Laboratory Control Sample (LCS)

An LCS is a mid-level concentration standard using a source different from the source of the calibration standards. This can include a different lot from the same manufacturer. An LCS is analyzed prior to sample analysis. The acceptance criterion is ±30% of the expected value. If the compound recovery fails to meet this criterion, the system is checked, and another LCS is prepped and analyzed. In the event the criterion cannot be met, the instrument is recalibrated.

8.4 Laboratory Blank

The Laboratory Blank is prepared by injecting 5 mL of DI water into a 10 mL headspace vial. A Laboratory Blank is analyzed between analysis of standards and project samples. If analytes are detected above the detection limit, the blank is reprepped and analyzed. If analytes are still detected above the detection limit, the possibility exists that all the vials in the batch contain contamination. In this case all samples and QC are reprepped in new vials.

8.5 Sample Duplicates

Sample duplicates are analyzed as required for certain clients. The duplicate is prepared using a second VOA sample using the procedures in section 7.1.

8.6 Detection Limits

An Method Detection Limit (MDL) study is preformed using a minimum of seven replicates at 1-2 times the Practical Quantitation Limit (PQL) or Reporting Limit (RL) described in 40 CFR Pt. 136 App. B. The MDL must be less than or equal to the detection limit.

Compound	PQL or RL (µg/L)
Methane	10
Ethane	10
Ethene	10

8.7 Matrix Spikes and Matrix Spike Duplicates (MS/MSD)

For projects requiring MS/MSD sets, aliquots of the sample are prepared using the procedures in section 7.1. The MS/MSD's are then spiked in the same fashion as

the LCS. After analysis, the original sample amount is subtracted out and the % recovery is calculated. The acceptance criterion for MS/MSD sets are 70-130% recovery and 30% RPD. If the criteria are not met, the data is flagged and the incident is narrated. Since samples are analyzed using an autosampler, it is not possible to know in advance the concentration of the sample. Consequently, the concentration of analytes in the unspiked analysis may be greater than four times the concentration of the added spike, making the spike amount insignificant to the original concentration. In these situations, recoveries and RPD may not meet the acceptance criterion. In addition, as MS/MSD's are typically taken from separate vials, sample heterogeneity may contribute to failed criteria.

8.8 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 Method RSK-175 for other method performance parameters and requirements.

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10.0 APPLICABLE DOCUMENTS/REFERENCES

"Analysis of Dissolved Methane, Ethane and Ethylene in Ground Water by a Standard Gas Chromatographic Technique", EPA SOP RSK-175, Revision No. 0, 8/11/94

Katahdin SOP CA-101, "Equipment Maintenance and Troubleshooting," 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, 2009.

Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," current revision.

LIST OF TABLES AND FIGURES

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
ICAL	Initial calibration prior to sample analysis	RSD ≤ 30%	Investigate and repeat initial calibration
ICV	Immediately following initial calibration	Recovery must be between 70% and 130%	Investigate; reprep. Repeat initial calibration if criteria cannot be met.
CV	If initial calibration analyzed, daily and after 20 samples, and at end of sequence.	%D for all analytes within 30%	(1) Evaluate the samples: (2) If the %RPD >30% and sample results are < PQL, narrate. (3) If %RPD >30% and is likely a result of matrix interference, narrate. (4) Otherwise, reanalyze all samples after last acceptable CV.
LCS	One LCS per 20 samples	Recovery must be between 70% and 130%	(1) Evaluate the samples and associated QC. (2) If an MS/MSD was performed and acceptable, narrate. (3) If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep.
Method Blank	One per batch of 20 or fewer samples	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 results which are < PQL >10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
Matrix Spike/Matrix Spike Duplicate	One MS/MSD as requested by clients.	Recovery must be between 70% and 130%, RPD ≤30.	(1) Evaluate the samples and associated QC. (2) If the LCS is acceptable, narrate. (3) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate	If requested by the client	%RPD of duplicate must be less than 30%.	(1) Check calculations for errors (2) Evaluate QC
Demonstration of capability - four replicate analyses of a QC check sample	One time per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Investigate; reprep
MDL and/or LOD/LOQ Verification	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

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-336-07	Method: EPA SOP RSK-175
Apparatus/Materials		
Reagents		
Sample preservation/ handling	(1) Collect sample in 40 mL VOA vial. (2) HCL added in field; hold time is 14 days, un-preserved is 7 days.	(1) Collect sample in 60 mL crimp top vial. (2) HCL is added in field; hold time is 14 days.
Procedures	(1) 5 mL of sample is displaced with 5 mL of nitrogen and transferred to a capped autosampler vial. Headspace is then generated in the autosampler vial. (2) Prior to injection, autosampler shakes sample for 15 min while heating to 40°C. (3) Autosampler pressurizes sample to fill 1 mL loop with headspace sample. (4) Calibration is obtained by spiking headspace samples with gas phase analyte and analyzing using the same procedure as the samples. Quantitation of samples is directly obtained using the calibration curve that relates µg analyte/mL water sample to peak area. (5) ICAL using average response factor	(1) Headspace is generated in 60 mL vials by displacing volume of liquid with helium. The amount of liquid should be 10% of sample volume in bottle, up to 10mL. (2) Sample is shaken 5 min to equilibrate analyte between headspace and liquid phase. (3) Syringe injections of 300 µL headspace into GC. (4) Direct injections of gas phase standards are used to obtain a calibration curve. Henry's law is used to calculate mg of gas per L of water. Calculation requires recording total volume of serum bottle and headspace, and sample temperature. (5) ICAL using linear regression
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL	See Section 9 of this SOP.	No information.

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FIGURE 1

EXAMPLE OF RUNLOG

Katahdin Analytical Services

GC Laboratory Instrument Runlog — Date: 2-21-19

Instrument: GC05 — Methods: RSK SOP-175 / EPA Rerion 1

Sample Name	Data File	Sample Amt.	DF	Method	Y/N	pH	Analyst	Comments
Prime	SMB2010	5mL	1	MEEB31A	N	-	JHR	
CV	11				Y	-		W6247064-3
W6247064-1	12				Y	-		
W6247064-2	13				Y	-		
SM1491-14	14				Y	4.2		
-15	15				Y			
-16	16				Y			
-17	17				Y			
-1	18				Y			
-2	19				Y			
-3	20				Y			
-4	21				Y			
-5	22				Y			
-6	23				Y			
-7	24				Y			Needs DL
-8	25				Y			
-9	26				Y			Needs DL
-10	27				Y			
-11	28				Y			
-12	29				Y			
-13	30				Y			
CV	31				N	-		Instrument fault
CV	32				Y	-		W6247064-4
								JHR 2-21-19

STANDARD	STOCK #	CONC.	AMOUNT
ICAL		1000 ug/mL	see comments
CV	AMP 5568	1000 ug/mL	500 uL
LCS	AMP 5098	10 000 ug/mL	100 uL

pH Lot #: HC857466

GC-002 - Revision 2 - 06/06/2018

QAGC377

0000051

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: _____

KATAHDIN ANALYTICAL SERVICES
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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.

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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

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 #: DV249-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 # RE5341	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/MS	Initials	Comments
12-26-18	AC	WG243501-1 WG243502-1	1000	1mL	NR	✓	✓	1mL	12-26-18	A1	JMS	SV- R497250
		WG243501-2	↓		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
		WG243502-2	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/MS	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		
		-5J	1060							6		
		-6K	1060							7		s.de 4e/a
		TL2691-27A	1030				✓			8		

Reviewed By _____ Date _____

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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: 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.**

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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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.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

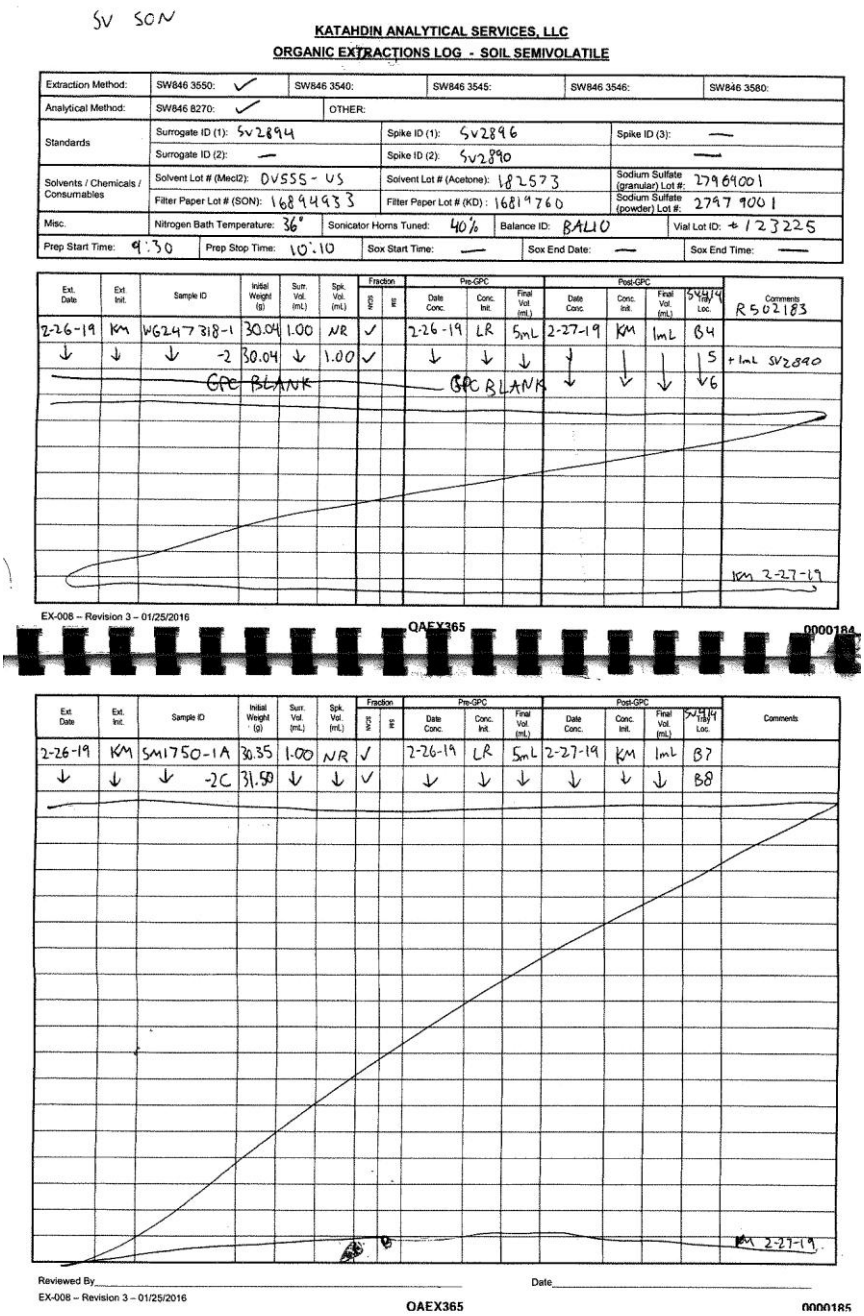
SUMMARY OF METHOD MODIFICATIONS

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 #: 27989001
	Filter Paper Lot # (SON): 16894933		Filter Peper 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 WDS	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)	SVHH Trib/1 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

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WDS	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)	SVHH Trib/1 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_____

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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 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 LMD 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 weigh 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**

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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”.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

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.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

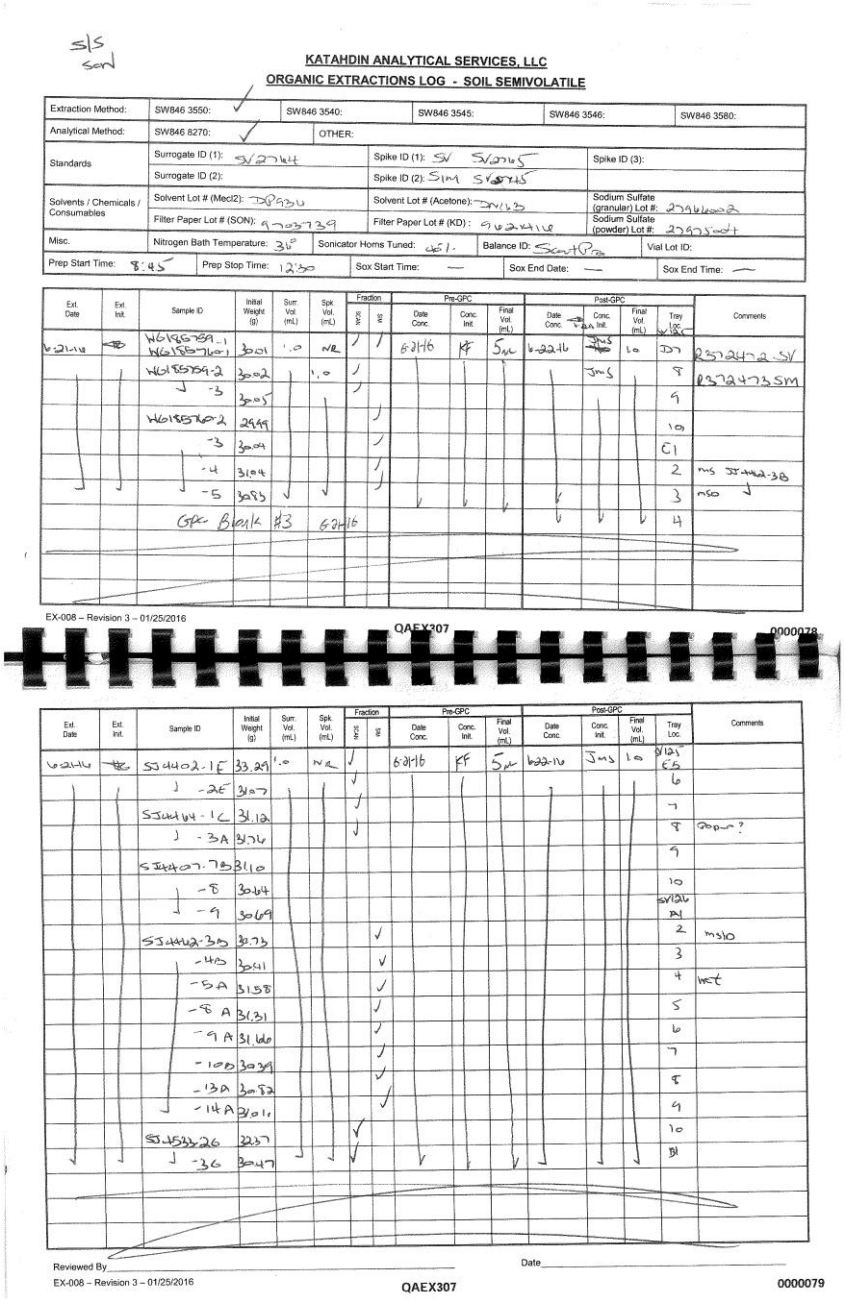
SUMMARY OF METHOD MODIFICATIONS

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		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



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): SV 27164	Spike ID (1): SV SV 27165	Spike ID (3):		
	Surrogate ID (2):	Spike ID (2): SIM SV 27145			
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): DP9314	Solvent Lot # (Acetone): DN163	Sodium Sulfate (granular) Lot #: 279616 w2		
	Filter Paper Lot # (SON): 97037139	Filter Paper Lot # (KD): 96244168	Sodium Sulfate (powder) Lot #: 279515 w1		
Misc.	Nitrogen Bath Temperature: 35°	Sonicator Horns Tuned: ✓	Balance ID: Scout Pro	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 WS	Fraction WB	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)	Post-GPC Tray Loc.	Comments
6-21-16	[illegible]	W61855759-1 W61855760-1	30.01	1.0	NA	✓	✓	6-21-16	KF	5mL	6-22-16	JMS	1.0	DD7	2357247-2 SV
		W61855759-2	30.02	1.0		✓						JMS		8	2357247-3 SIM
		-3	30.05				✓							9	
		W61855760-2	29.99				✓							10	
		-3	30.04				✓							C1	
		-4	31.04				✓							2	ms SJ4402-3B
		-5	30.83	✓	✓		✓	✓	✓	✓	✓	✓	✓	3	msd
		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 WS	Fraction WB	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)	Post-GPC Tray Loc.	Comments
6-21-16	[illegible]	SJ4402-1E	33.29	1.0	NA	✓		6-21-16	KF	5mL	6-22-16	JMS	1.0	SV12b E5	
		-2E	31.07			✓								6	
		SJ4464-1C	31.12			✓								7	
		-3A	31.16											8	90pm?
		SJ4407-7B	31.10											9	
		-8	30.64											10	
		-9	30.69											SV12b B4	
		SJ4442-3B	30.73				✓							2	msd
		-4B	30.41				✓							3	
		-5A	31.58				✓							4	wet
		-8A	31.31				✓							5	
		-9A	31.66				✓							6	
		-10B	30.39				✓							7	
		-13A	30.52				✓							8	
		-14A	31.01				✓							9	
		SJ4533-26	22.37			✓	✓							10	
		-36	30.47	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	B1	

Reviewed By_____ Date_____

EX-008 – Revision 3 – 01/25/2016 QAEX307 0000079

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

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-551
Revision History
Cover Page
Page 1

TITLE: GRAIN SIZE ANALYSIS

Prepared By: Jessica Quinn Date: 10-1-15

Approved By:

Department Manager: [signature] Date: 10-1-15

Operations Manager: Deborah J. Nadeau Date: 10.1.15

QA Officer: Lisa Dimond 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

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
	25	50	75	100	200
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	50	75	100	200
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
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	SM2456-4	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		

% Moisture	71.105

Sample Split (Oven Dried)	Sample (g)
Sample >=#10	0
Sample <=#10	25.98

Sieve Only:	
Sieve Only, Wet Wash:	

Hydrometer Data	
Serial Number	152303
Cal Date:	3/25/19: 11:32
Low Temp C	16.4
Low Temp Reading	1.0035
High Temp	17.6
High Temp Reading	1.0035
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	276.0	270.16
#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	340.43 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)	17.43	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

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. Buxton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Hexham 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^{o}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

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

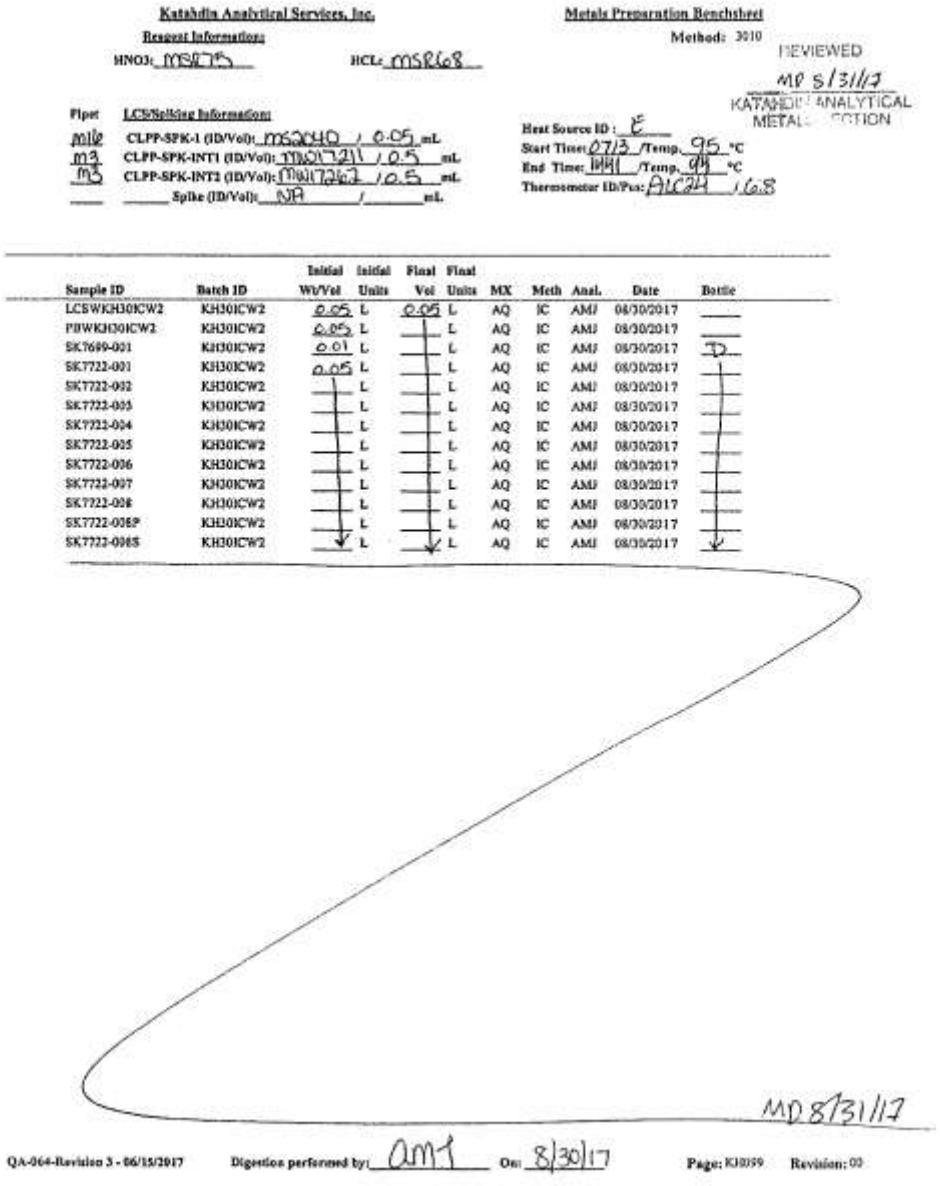
PROCEDURE CONDENSATION: EPA METHOD 3010

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: MSB75 HCL: MSB68

Method: 3010

REVIEWED
MP 8/31/17
KATAHDIN ANALYTICAL
METALS SECTION

Pipet LCS/Spiking Information:

m1b CLPP-SPK-1 (ID/Vol): MS2040 / 0.05 mL
m3 CLPP-SPK-INT1 (ID/Vol): MW17211 / 0.5 mL
m5 CLPP-SPK-INT2 (ID/Vol): MW17267 / 0.5 mL
___ _____ Spike (ID/Vol): NA / _____ mL

Heat Source ID: E
Start Time: 0713 /Temp. 95 °C
End Time: 1111 /Temp. 94 °C
Thermometer ID/Pos: A1C24 / 6.8

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	
SK7689-001	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	

MP 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

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: Joel C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Hayden 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.	DH	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.	DH	8.29.02	8.29.02
03 3050B	New Title to include ICP-MS. 3. Use of digestion block and polyethylene digestion tubes added to sections 9.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 5.0ml, 2.0ml then 7.0ml. 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	UAD	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.	UAD	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	UAD	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.**

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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^{o}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^{o}C ± 5^{o}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^{o}C ± 5^{o}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^{o}C ± 5^{o}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

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:
MLD	☒	☒	CLPP-SPK-1 (ID/Vol): MS20640 / 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: AIC24 / 4.1

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METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOKJ01ICS1	KJ01ICS1	1.0	g	0.1	L	SL	IC	AMJ	09/01/2017	—
PBSKJ01ICS1	KJ01ICS1	1.0	g		L	SL	IC	AMJ	09/01/2017	—
SK7769-001	KJ01ICS1	1.88	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001P	KJ01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001S	KJ01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7789-001	KJ01ICS1	1.20	g		L	SL	IC	AMJ	09/01/2017	G
SK7794-001	KJ01ICS1	1.95	g		L	SL	IC	AMJ	09/01/2017	A
SK7794-002	KJ01ICS1	1.98	g		L	SL	IC	AMJ	09/01/2017	〃
SK7794-003	KJ01ICS1	1.92	g		L	SL	IC	AMJ	09/01/2017	〃
SK7837-001	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-002	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-003	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-004	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-005	KJ01ICS1	1.0	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-006	KJ01ICS1	1.0	g	↓	L	AR	IC	AMJ	09/01/2017	↓

MD 9/1/17

QA-065-Revision 2 - 06/16/2017 Digestion performed by: AMJ On: 9/1/17 Page: KJ001 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	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	94.0	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. - 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 - 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	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	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 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: 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).

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-608
Revision History
Cover Page
Page 1

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 8 to reflect this. Made changes to element conc's. 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 Chk std. 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~~ 200.7ICS-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, DoDQSM 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.

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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.

List of Tables & Figures

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-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: Jeb C. Bustin Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1-29-01

General Manager: Dennis F. Murphy 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 M6100 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 LCS0 - 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 MDL, 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 ~~KASNHG~~ KAS throughout. KASINC to LAD 10/17	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^{o} to 110^{o} 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.

List of Tables and Figures

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

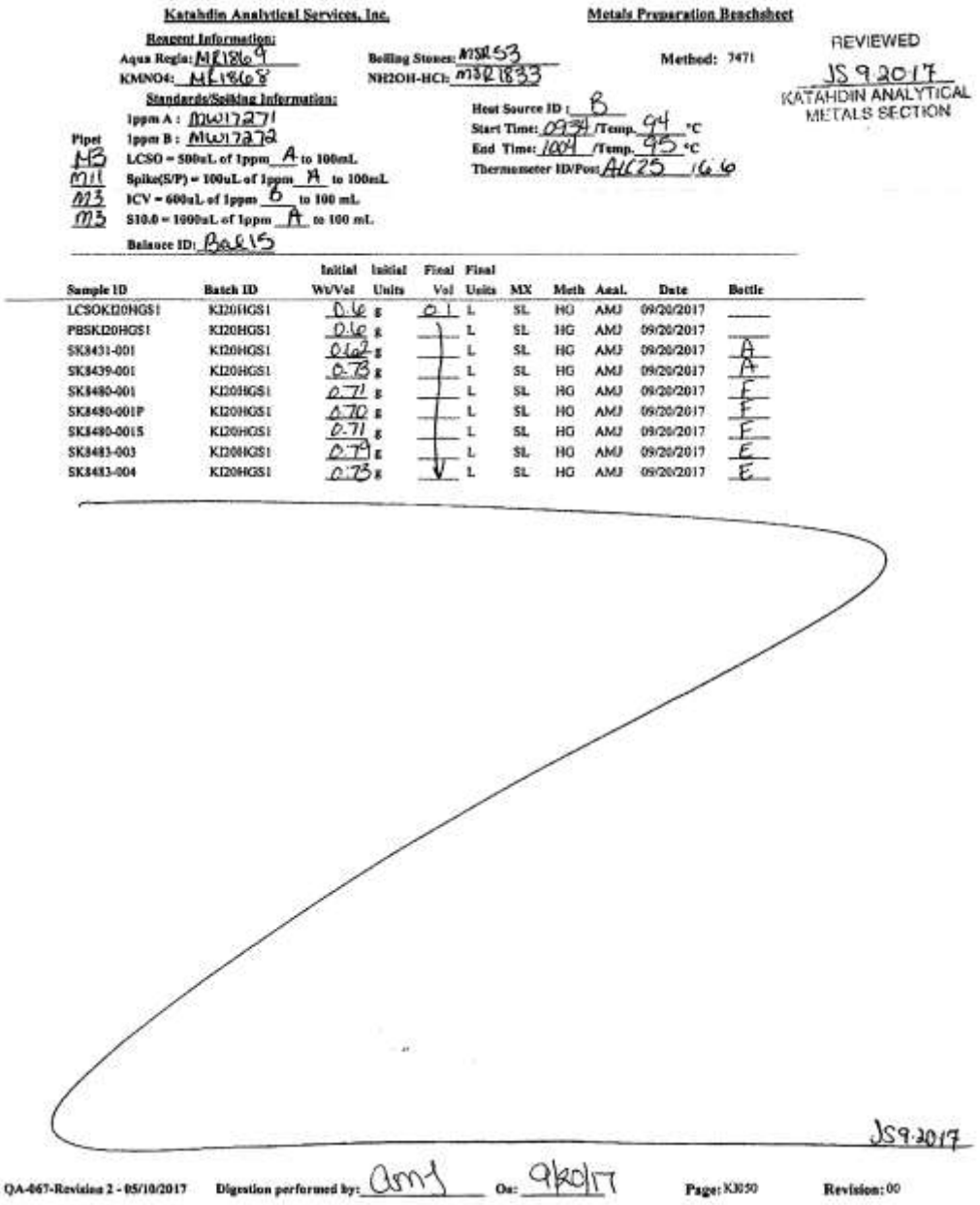
SUMMARY OF METHOD MODIFICATIONS

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**

REVIEWED JS 9.2017 KATAHDIN ANALYTICAL METALS SECTION

Reagent Information:
Aqua Regia: ME1869 Boiling Stones: M3R53 Method: 7471
KMNO4: ME1868 NH2OH-HCl: M3R1833

Standards/Spiking Information:
1ppm A: MW17271
1ppm B: MW17272

Pipet
M5 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
M5 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: AHC25 16.6

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
SK8480-001P	KI20HGS1	0.70	g		L	SL	HG	AMJ	09/20/2017	F
SK8480-001S	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		L	SL	HG	AMJ	09/20/2017	E

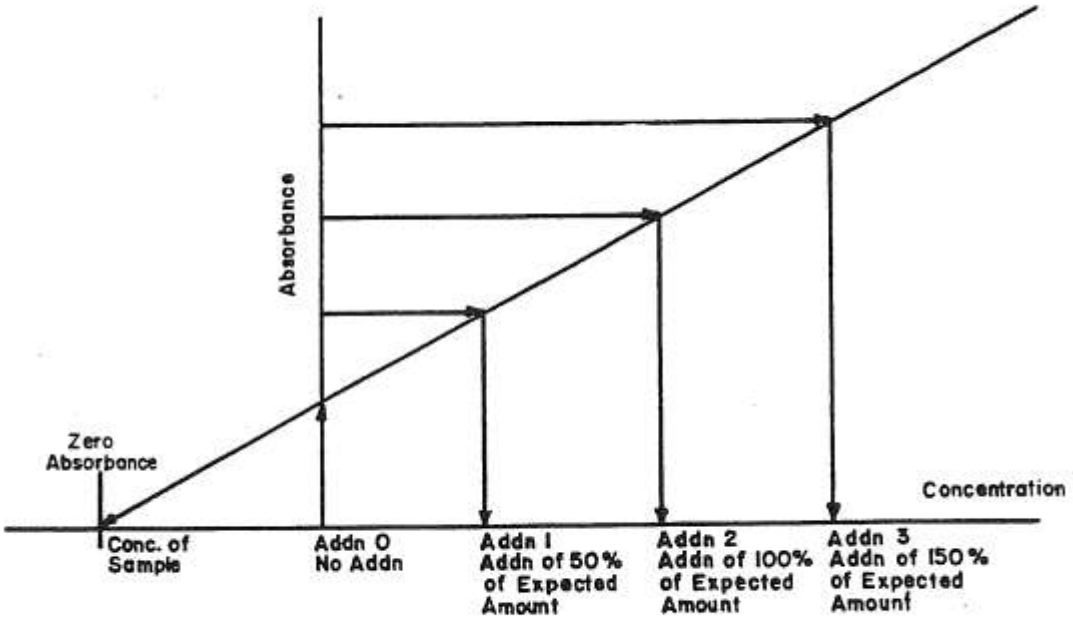
JS 9.2017

QA-067-Revision 2 - 05/10/2017 Digestion performed by: AMJ On: 9/20/17 Page: KI050 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	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	860	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	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	58.9 - 139
sodium	15600*	1080	602 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	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	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 - 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	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	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 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	$SnCl_2$	MSR160/MSR760		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	MSR97/MSR98	480.01	FV= 4000mL w/ Reag H_2O
↓	↓	↓	↓	↓	NaCL	MSR26	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	↓
MR867	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.**

Recipient: _____ Date: _____

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^{o} 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^{o} to 750^{o} 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^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 90-95^{o} C, continue heating the samples at 90-95^{o} 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^{o} 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.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

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

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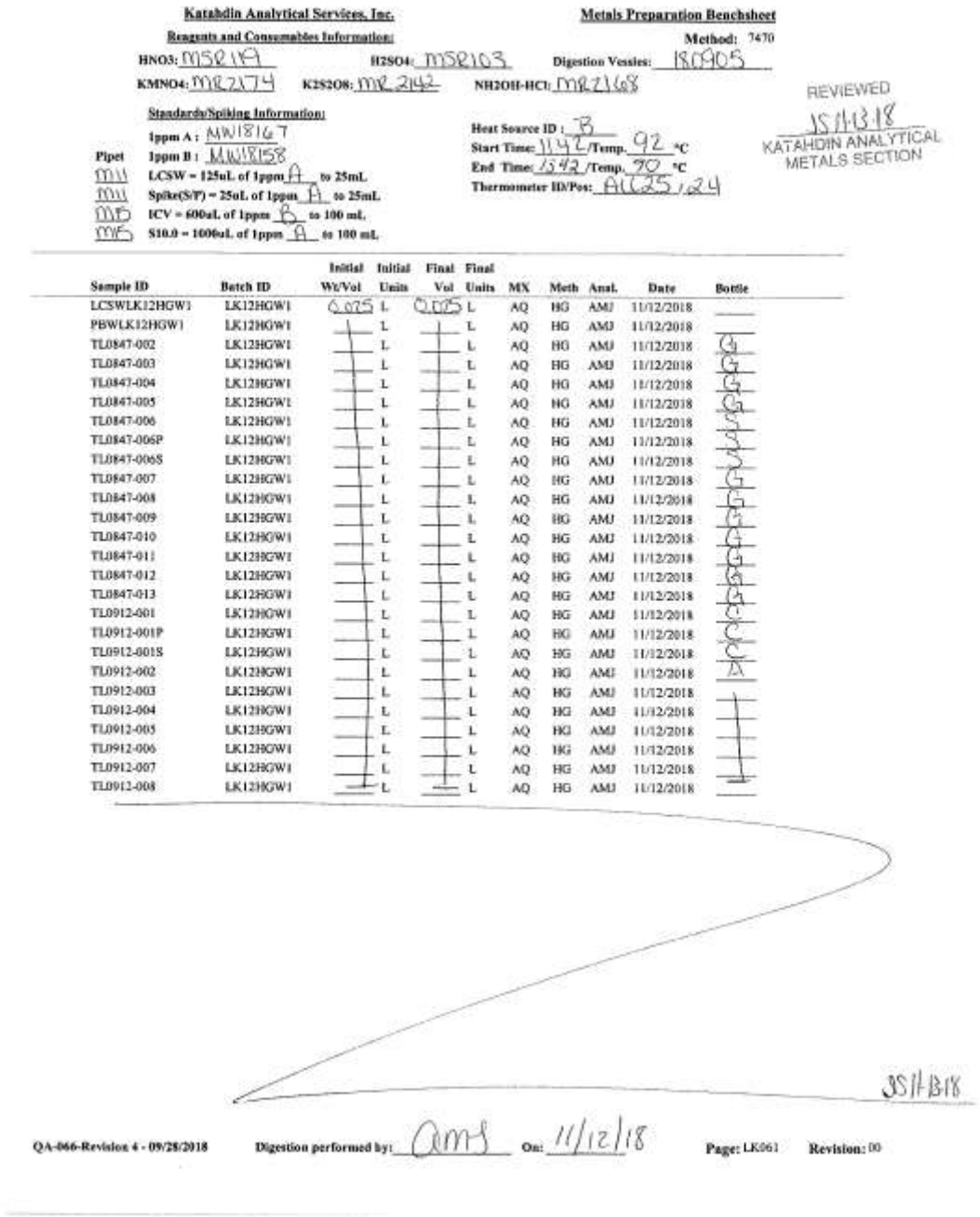
FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagents and Consumables Information: Method: 7470

HNO3: MS0149 H2SO4: MS2103 Digestion Vessles: 180905

KMNO4: MR2174 K2S2O8: MR2142 NH2OH-HCl: MR2168

REVIEWED 1/3/18 KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:

1ppm A : MW18167

1ppm B : MW18158

Pipet

MM1 LCSW = 125uL of 1ppm A to 25mL

MM1 Spike(S/P) = 25uL of 1ppm A to 25mL

MP5 ICV = 600uL of 1ppm B to 100 mL

MM5 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: AC25,24

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	
TL0847-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-006P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-006S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-009	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-010	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-011	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-012	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-013	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-001	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-001P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-001S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
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	

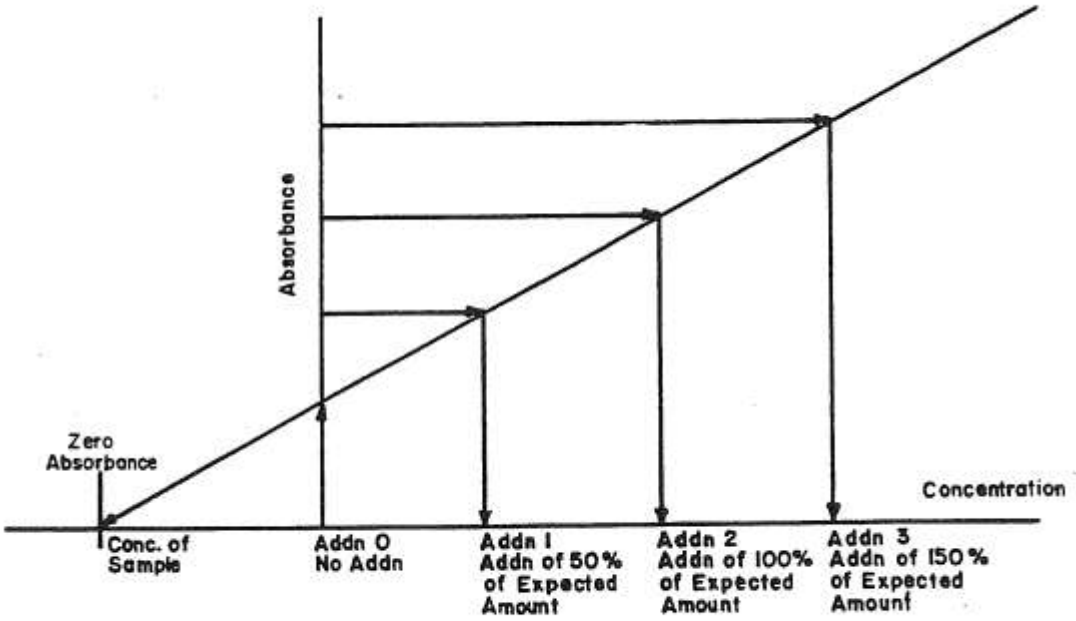
JS 11-13-18

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



TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Prepared By: George Brewer Date: 03/14/05

Approved By:

Department Manager: George Brewer Date: 03/14/05

Operations Manager: Deborah J. Nadeau Date: 3.14.05

QA Officer: Leslie D. Monod Date: 031405

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added expirations dates (1yr) for SPLP fluid 3 Added DOC requirements Revised TCLP/SPLP logbooks - making it usable for both TCLP/SPLP and record pH, expiration date	LAD	01/07	01/07
02	Updated figures 6 & 8 with current logbook pages.	LAD	04/08	04/08
03	Updated or added references to sections 1.3, 9 and 10. Updated logbook page.	LAD	06/10	06/10
04	Figure 8 - Updated with new logbook. Revised text + references to logbook throughout. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added, removed and edited references	LAD	04/12	04/12
05	Changed KAS INC. to KAS throughout. Updated Figures 6 & 7. Updated DoD references	LAD	03/15	03/15

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sec. 4 – added thermometer. Sec. 5.4 - Changed HCl to HNO_3. Moved table in Sec 6.4 to end (Table 4). Updated logbook pages. Changed wording in Secs. 6, 7, and 8 for clarity and to match CA-510. Updated SPLP matrix spiking (per PBT).	UAD	03/18	03/18

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

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-620-06**, titled **Synthetic Precipitation Leaching Procedure (SPLP) For Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-620-06** titled **Synthetic Precipitation Leaching Procedure (SPLP) For Inorganic and Non-Volatile Organic Analytes.**

Recipient: _____Date:_____

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to define the procedures used by Katahdin Analytical Services, personnel for SPLP extraction of samples for inorganic and non-volatile organic components using US EPA Method 1312 (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA SW846), with the modifications discussed in Table 2.

The SPLP (Synthetic Precipitation Leaching Procedure) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.1 Definitions - None.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in SPLP extractions. Each analyst must demonstrate the 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 SPLP extractions 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

Users of this procedure must be aware 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 may not be precisely known; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDS) 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 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

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes from SPLP extraction may contain acids, heavy metals, toxic organics, and other toxic components and should be disposed of in a manner appropriate to the hazards they present. Further information regarding waste classification and disposal may be obtained by consulting the Katahdin Hazardous Waste Plan and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the initial liquid phase is first separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary, and the solid phase is extracted with an amount of extraction fluid equal to 20 times its weight. If the sample is a soil, the composition of the extraction fluid employed depends on the region of the country where the sample site is located. If the sample is a waste or a wastewater, the extraction fluid used is a pH 4.2 solution. After extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If they are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these are analyzed together. If they are incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Because the dissolved solids contents of SPLP extracts are typically high, analyses of these extracts are often troubled by matrix interferences. Methods to detect and overcome matrix interferences are integral to the SPLP procedure and are discussed in detail in Section 8.0: Quality Control and Acceptance Criteria.

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus (rotary extractor) - The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 revolutions per minute (rpm) – see Figure 1. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the results are recorded in the Non-Volatile TCLP/SPLP Extraction Logbook (Figure 7). If the measured rotation rate of an extractor is outside the range 30 ± 2 rpm, it must be taken out of service until it can be repaired.

4.2 Extraction vessels - must fit the rotary extractor and have sufficient capacity to hold the sample and the extraction fluid (jars with capacities of 2.2 L are normally used). The vessel must be made of borosilicate glass or fluorinated plastic (e.g. Teflon) if the extract is to be analyzed for organics. If the extract is to be analyzed only for inorganics, polyethylene or polypropylene containers may be used.

4.3 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.

4.4 Filters - Borosilicate glass fiber filters containing no binder materials and having an effective pore size of 0.6 to 0.8 µm, 142 mm diameter or equivalent. Prefilters must not be used. Glass fiber filters are fragile and should be handled with care. Filters should be acid-washed with 1N HNO_3 and triple rinsed with laboratory reagent grade water (minimum 500 mL/ rinse) prior to use.

4.5 pH meter accurate to ± 0.05 units at 25 °C. The pH meter must be calibrated on each day of use.

4.6 Laboratory balance accurate to within ± 0.01 g

4.7 Beakers, glass, 500 mL

4.8 Watch glasses, appropriate diameter to cover beakers

4.9 Magnetic stirrer

4.10 Room thermometer capable of recording min/max temperatures over a 24 hour cycle

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

5.0 REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1 Laboratory reagent grade water - Reagent water should be monitored periodically for impurities.

5.2 Sulfuric acid, concentrated (H_2SO_4) – reagent grade

5.3 Nitric acid, concentrated (HNO_3) – reagent grade

5.4 Nitric acid, 1N, for acid-washing filters. Dilute 63 mL reagent grade HNO_3 to 1000 mL with laboratory reagent grade water.

5.5 Sulfuric acid (0.6 weight percent) / nitric acid (0.4 weight percent) mixture – Add 0.60 ± 0.05 g concentrated H_2SO_4 and 0.40 ± 0.05 g HNO_3 to a 100 mL volumetric flask half-filled with laboratory reagent grade water. Swirl to mix and bring to a final volume of 100 mL with laboratory reagent grade water.

5.6 SPLP Fluid #1 – Add approximately 15 L of laboratory reagent grade water to a clean graduated 20 L polyethylene carboy reserved for this fluid. Add approximately 5 mL of 0.6% H_2SO_4 / 0.4% HNO_3 solution to the carboy, and fill with laboratory reagent grade water to the 20 L graduation. Cap the carboy tightly and agitate it until the fluid is well mixed. Dispense approximately 30 mL of fluid from the carboy's spigot into a disposable cup and measure the pH of the fluid. The pH of the fluid must be 4.20 ± 0.05. If necessary, add more acid solution to lower the pH, or remove some of the fluid and replace it with laboratory reagent grade water to raise the pH, until the correct pH is obtained. SPLP Fluid #1 is used to determine the leachability of soils from sites east of the Mississippi River, and the leachability of wastes and wastewaters. The fluid may be used for up to one year from the preparation date.

5.7 SPLP Fluid #2 - Add approximately 4 L of laboratory reagent grade water to a clean graduated 5 L polyethylene carboy reserved for this fluid. Add approximately 1.5 mL of 0.6% H_2SO_4 / 0.4% HNO_3 solution to the carboy, and fill with laboratory reagent grade water to the 5 L graduation. Cap the carboy tightly and agitate it until the fluid is well mixed. Dispense approximately 30 mL of fluid from the carboy's spigot into a disposable cup and measure the pH of the fluid. The pH of the fluid must be 5.00 ± 0.05. If necessary, add more acid solution to lower the pH, or dump out some of the fluid and replace it with laboratory reagent grade water to raise the pH, until the correct pH is obtained. SPLP Fluid #1 is used to determine the leachability of soils from sites west of the Mississippi River. The fluid may be used for up to one year from the preparation date.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

5.8 SPLP Fluid #3 – This fluid is laboratory reagent grade water (Section 5.1) and is used to determine cyanide leachability.

NOTE: The pH of each extraction fluid must be checked prior to each use to ensure that it has been prepared accurately, and the measured pH is recorded in the Non-Volatile TCLP/SPLP Extraction Logbook (Figure 7) for each sample extracted. Details of the preparation of these fluids (reagent lot numbers, volumes, and masses; measured pH; etc.) are recorded in the SPLP Fluid Preparation and Use Logbook (Figure 6). Upon preparation, each new batch of extraction fluid is assigned a batch number by the analyst (batches are numbered consecutively), and the Katahdin Sample Number of each client sample extracted with a particular fluid batch is recorded in the SPLP Fluid Preparation and Use Logbook. Extraction fluids are monitored for impurities as described in Section 8.0 of this SOP.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for all analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 250 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4 °C and opened immediately prior to SPLP extraction.

6.3 SPLP extracts should be prepared for analyses and analyzed as soon as possible following SPLP extraction. Extracts for metals analysis must be acidified to a pH < 2 with nitric acid. Extracts for other analyses should be preserved according to the guidance given in the individual analytical methods. Extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

6.4 See Table 4 for sample holding times for non-volatile SPLP extraction and analysis.

7.0 PROCEDURES

The procedure consists of a series of preliminary evaluations of the waste, followed by the actual extraction. Flow charts summarizing the procedure appear as Figures 2 and 3. Preliminary evaluations are to be performed on a minimum 100 g aliquot of the waste. This aliquot may not actually undergo SPLP extraction. These preliminary evaluations include: (1) determination of the percent solids, Section 7.1; (2) determination of whether the waste

contains insignificant solids and is, therefore, its own extract after filtration, Section 7.2; (3) particle size evaluation, Section 7.3; and (4) determination of the appropriate extraction fluid to be used for the SPLP extraction, Section 7.4.

All information and measurements pertaining to SPLP extractions are recorded in the Non-Volatile SPLP Extraction Logbook (Figure 7). In the following procedure, the section or column of the Non-Volatile SPLP Extraction Logbook page in which the pertinent information should be recorded is indicated in bold, e.g. **Section II** or **Column C**.

PRELIMINARY EVALUATIONS

7.1 Determination of Percent Solids (**Section II**) - Percent solids is defined for SPLP as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) the percent solids determination may be omitted. Proceed to Section 7.3, Particle Size Evaluation.

If the sample is liquid or multiphasic, liquid/solid separation by filtration is required to make a preliminary determination of percent solids. This involves the filtration device. The procedure is as follows, Sections 7.1.1 through 7.1.9:

7.1.1 Pre-weigh the filter (**Column A**) and the container that will receive the filtrate (filtrate vessel) (**Column B**).

7.1.2 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.2 Weigh out a subsample of the waste (100 g minimum) and record the combined weight of the weigh boat and waste (**Column C**).

7.1.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged, prior to filtration. Centrifugation is to be used only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.5 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder, spreading the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.1.6 Weigh the weigh boat and any residue clinging to it (**Column D**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Column E**).

7.1.7 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.1.8 Weigh the filtrate vessel and its contents (**Column F**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Column G**).

7.1.9 Calculate the percent wet solids as follows (**Column H**):

$$\text{Percent wet solids} = \frac{\text{(Total weight of waste)-(Weight of liquid phase)}}{\text{Total weight of waste}}$$

7.2 If the percent solids determined in Section 7.1.9 above is equal to or greater than 0.5% and the weight of water entrained in the filter is small in comparison with the weight of the solid phase, then proceed to Section 7.3 to determine whether the solid material requires particle size reduction. Continue with Section 7.2 if it is noticed that the amount of the filtrate entrained in wetting the filter is significant in proportion to the weight of the solid phase. If the percent solids determined in Section 7.1.9 is less than 0.5%, then proceed to Section 7.5.4 using a fresh portion of the waste.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighings yield the same value within ± 1%. Record the weight of the filter and dry solids (**Column I**).

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the weight of dry solids by subtracting the weight of the filter from the weight of the filter and dry solids (**Column J**).

7.2.4 Calculate the percent dry solids as follows (**Column K**):

$$\text{Percent dry solids} = \frac{\text{Weight of dry solids}}{\text{Total weight of waste}} \times 100$$

Note: Non-aqueous liquid samples (e.g. oils) may be entrained in the filter, and may remain in the filter after drying, contributing weight to the dried filter. If this is the case, the surface of the filter should be examined for apparent solids or particulate material. If none are found, a comment to that effect should be made in the Comments section of the Non-Volatile SPLP Extraction Logbook (e.g. "No apparent solids present – dry solid weight is due to entrained non-volatile liquid"), and the sample should be treated as if it contains less than 0.5% dry solids.

7.2.5 If the percent dry solids is less than 0.5%, then proceed to Section 7.5.4. If the percent dry solids is greater than or equal to 0.5%, proceed to Section 7.3.

7.3 Particle Size Evaluation - Visually evaluate the particle size of the solid phase of the waste. Filamentous material (cloth, paper, etc.) will require particle size reduction if it has a surface area per gram of less than 3.1 cm^3. Other solid materials require particle size reduction if the particles are greater than 1 cm in their narrowest dimension (i.e. if they will not pass through a 9.5 mm standard sieve). Particle size reduction may be accomplished by cutting, crushing, or grinding the waste to a surface area or particle size as described above. Perform particle size reduction on the solid material that will actually undergo extraction, not on that used for the preliminary determinations.

7.4 Determination of Appropriate Extraction Fluid - If the solid content of the waste is greater than or equal to 0.5%, determine the appropriate fluid for the non-volatiles extraction as follows:

7.4.1 For soils, if the sample is from a site that is east of the Mississippi River, SPLP Fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, SPLP Fluid #2 should be used.

7.4.2 For wastes and wastewaters, SPLP Fluid #2 should be used.

7.4.3 For cyanide containing wastes or soils, SPLP Fluid #3 must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

SPLP EXTRACTION FOR NON-VOLATILES

7.5 A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of SPLP extract will be sufficient to perform all of the required analyses. If necessary, multiple extractions may be performed and the extracts combined and aliquoted for analysis.

7.5.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e. is 100% solid), weigh out a subsample of the waste (100 gram minimum), record the weight (**Section II**), and proceed to Section 7.5.11. If the sample is liquid or multiphasic, liquid/solid separation is required - proceed to Section 7.5.2.

7.5.2 Pre-weigh the container that will receive the filtrate (filtrate vessel) (**Column L**).

7.5.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if extracting for metals components. Acid-washed filters may be used for non-volatile extractions even when metals are not of concern.

7.5.4 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the waste and weigh boat (**Column M**). If the waste contains < 0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the SPLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the required analyses. For wastes containing > 0.5% dry solids, information is obtained in Section 7.1 (Percent Solids Determination) to calculate the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the SPLP extract.

7.5.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the sample filtration system.

7.5.6 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.5.7 Weigh the weigh boat and any residue clinging to it (**Column N**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Column O**).

7.5.8. Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through he filter in any 2-minute interval, slowly increase in pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.5.9 Weigh the filtrate vessel and its contents (**Column P**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Column Q**). Decant the liquid phase into a graduated cylinder and measure and record its volume (**Column R**). Pour the liquid phase back into the filtrate vessel for storage. The liquid phase may now either be analyzed or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, as outlined in Section 7.5.8, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.5.10 Calculate the weight of wet solids by subtracting the weight of the liquid phase from the total weight of waste (**Column S**).

7.5.11 If necessary, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.3. Describe the particle size reduction process in **Section IV** of the

logbook. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

7.5.12 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{(20)\,(\text{Weight of wet solids})}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Record the fluid batch ID and the pH (measured on day of use) in the Comments section **(Section V)** of the logbook. Record the amount used in **Section II** of the logbook. Close the extractor bottle tightly (Teflon tape may be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 RPM during the extraction period of 18 ± 2 hours at 23 ± 2 °C. Record the extraction start and end times and the room temperatures (min/max throughout extraction process) in **Section I** of the logbook. In order to maintain the required temperature range throughout the extraction, a temperature controlled extraction case is used that has temperature maintained by an individual heating and cooling system.

NOTE: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.5.13 Following the extraction, separate the contents of the vessel into its component liquid and solid phases by filtering through a new acid-washed glass fiber filter, as outlined in Sections 7.5.6 and 7.5.8. For final filtration of the SPLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: If the waste contained no initial liquid phase, it is only necessary to filter enough extract to support the required analyses. However, if the waste contained an initial liquid phase, the entire contents of the extraction vessel must be filtered.

7.5.14 Prepare the SPLP extract as follows:

7.5.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.5.13 is defined as the SPLP extract. Proceed to Section 7.5.15.

7.5.14.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.5.13 with the initial liquid phase of the waste obtained in Section 7.5.8. This combined liquid is defined as the SPLP extract. Proceed to Section 7.5.15.

7.5.14.3 If the initial liquid phase of the waste, as obtained prior to extraction from Section 7.5.8, is not or may not be compatible with the filtered liquid resulting from Section 7.5.13, do not combine these liquids. Measure the volume of filtrate obtained in Section 7.5.13 and record in **Section V** of the logbook. Individually analyze these two liquids, collectively defined as the SPLP extract, and combine the results mathematically, as described in Section 7.6.

7.5.15 Following collection of the SPLP extract, the pH of the extract should be measured and recorded (**Section II**). Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH < 2. All other aliquots must be stored under refrigeration (4 °C) until analyzed.

7.6 The SPLP extract shall be prepared and analyzed according to appropriate analytical methods. SPLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.6 Compare the analyte concentrations in the SPLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality control requirements.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 1312 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 listed 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.

8.1 A minimum of one method blank for every 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel must be extracted and analyzed for the same contaminants as all associated samples. The method blanks are analyzed to check for laboratory contamination. A count of extractions performed in each extraction vessel is maintained in order to monitor the frequency of method blanks (1 per 20 extractions per vessel) required for each extraction vessel.

8.1.1 After SPLP extraction, SPLP method blanks must undergo preparative extraction and analysis within method holding times (refer to Table 4). For this reason it may be necessary to extract more than one method blank using a particular batch of extraction fluid. For example, suppose that a sample requiring analysis for SPLP metals and semivolatiles is extracted using freshly prepared fluid from Batch 1391. Because the fluid is new, a method blank is extracted with the sample and analyzed for the same components as the sample. Eight days later, a different sample requiring full SPLP analysis (metals, semivolatiles, pesticides, and herbicides) is extracted using fluid from Batch 1391. Because the holding time for the previous SPLP method blank

for pesticides and herbicides has expired, a new SPLP method blank must be extracted and analyzed for pesticides and herbicides. The new method blank need not be analyzed for metals and semivolatiles, because the first method blank that was prepared with fluid from Batch 1391 has already been analyzed for these constituents.

8.1.2 Each SPLP method blank is identified in the SPLP extraction logbooks by code. The first three characters are "PBT", which stands for "Preparation Blank – TCLP/SPLP". Following this is the preparation number of the extraction fluid (e.g., 1391), which is unique to the extraction date for a particular batch of fluid. The last character is a letter, starting with "A" and proceeding alphabetically, which is unique to the extraction date for a particular batch of fluid. For example, "PBT1391A" refers to the first SPLP method blank extracted using fluid from Batch 1391; "PBT1391B" refers to the second SPLP method blank extracted using the same fluid. The extraction date of each SPLP method blank is recorded in the Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook. For every SPLP method blank prepared, at least one matrix-spiked aliquot must be prepared with a sample associated with that SPLP method blank. Record the sample chosen and spiking amounts in the logbook. See Section 8.2 for matrix spiking procedure.

8.2 The laboratory recommends that a matrix spike be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. EPA Method 1312 requires one MS/MSD per analytical batch. Because the laboratory charges for the preparation and analysis of SPLP matrix spikes, selection of samples for SPLP matrix spiking is left to the discretion of the client. Follow the matrix spike addition guidance provided in each analytical method. Additional matrix spiking directions and guidance are provided in Table 3 and Figures 4 and 5.

8.2.1 Matrix spikes are to be added after filtration of the SPLP extract and before any preservation. Matrix spikes should not be added prior to SPLP extraction of the sample.

8.2.2 Instructions for preparing SPLP matrix spikes for metals analysis are contained in Table 3. Instructions for preparing SPLP matrix spikes for organics analyses are contained in Figures 4 and 5. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of SPLP extract as that which was analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

Recovery (%) = 100 $(X_s-X_u)/K$

where: X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample

8.2.4 The purpose of the matrix spike is to monitor the performance of the sample preparation and analytical methods used and to determine whether matrix interferences exist. Use of internal calibration methods (e.g. the method of standard additions [MSA]), modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the SPLP extract when the recovery of the matrix spike is below the expected analytical method performance. Metallic analytes must be quantitated by the method of standard additions if the SPLP matrix spike recovery for the analyte is less than 50% and the measured concentration of the analyte in the unspiked aliquot is within 20% of the regulatory level.

8.3 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

8.4 All quality control measures described in the appropriate analytical methods shall 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.

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The Limit of Detection (LOQ) is the minimum level, concentration, or quantity 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 1312 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Third Edition, Final Update I (7/92), Method 1312

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.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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List of Tables and Figures

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Synthetic Precipitation Leaching Procedure (SPLP) / EPA 1312	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid. One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods. Refer to individual analytical methods	Prepare fresh extraction fluid and repeat SPLP extraction of all associated samples. Remove extraction vessel from service.
	Matrix Spike	One per analytical batch (required by method 1312). One per waste type (suggested, left to discretion of client).	For metallic analytes, >50% if native analyte concentration is within ± 20% of regulatory level. For other analytes, refer to appropriate analytical methods.	For metallic analytes, quantitate by method of standard additions. For other analytes, refer to appropriate analytical methods.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-620-06	EPA METHOD 1312
QC - Method Blanks	Frequency of one method blank per 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC - Spikes	Matrix spike **recommended** for each waste type and analytical batch.	Matrix spike **required** for each waste type and analytical batch.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 3

SPLP MATRIX SPIKING FOR METALLIC ANALYTES

SPIKING INSTRUCTIONS			
Sample or Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 50 mL Final Volume (mL)**
SPLP Matrix Spike (ICP)	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
SPLP Matrix Spike (Mercury)	1000 ug/L Hg Standard	Prepared from 1000 mg/L stock standard	0.10

Note: Spiking must be performed after SPLP extraction and before preservation.

PREPARATION OF INTERMEDIATE SPIKING SOLUTIONS			
Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 100 mL Final Volume (mL)**
CLPP-SPK-INT1	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	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
1000 ug/L Hg Standard	1000 mg/L Hg	Inorganic Ventures	0.10

ELEMENT CONCENTRATIONS IN MATRIX SPIKES AND SPIKING SOLUTIONS				
Element	**CONCENTRATION IN SOLUTION, mg/L**			
	SPLP Matrix Spike	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**1000 ug/L Hg Std.**
Arsenic	2.000		200	
Barium	2.000	2000		
Cadmium	0.050		5	
Chromium	0.200	200		
Lead	0.500		50	
Selenium	2.000		200	
Silver	0.050	50		
Mercury	0.0020			1000

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 4

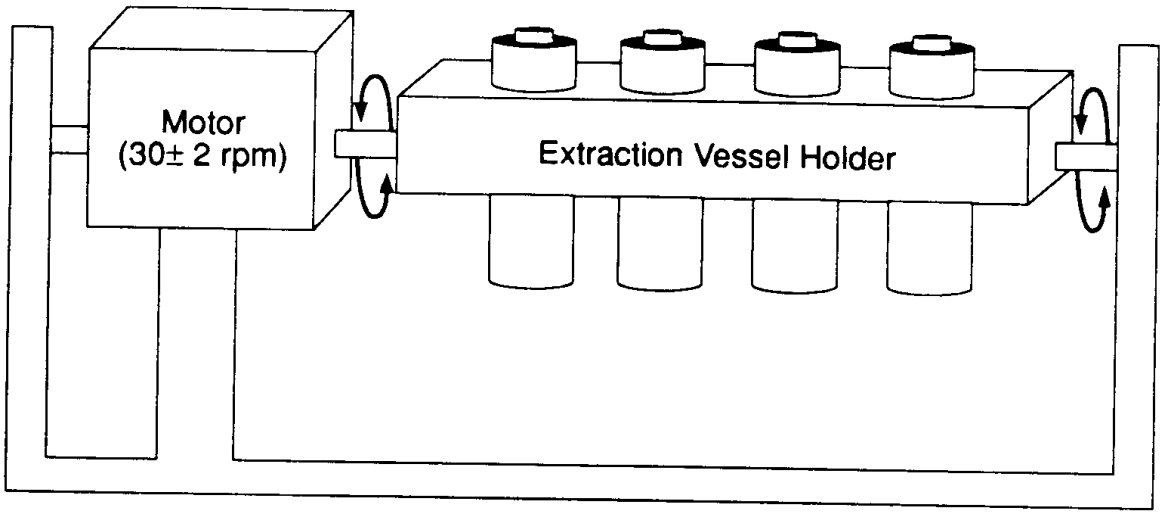
SPLP HOLDING TIMES SUMMARY

SPLP PARAMETER	FROM COLLECTION TO SPLP EXTRACTION	FROM SPLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
PEST/HERBS	14	7	40
SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 1

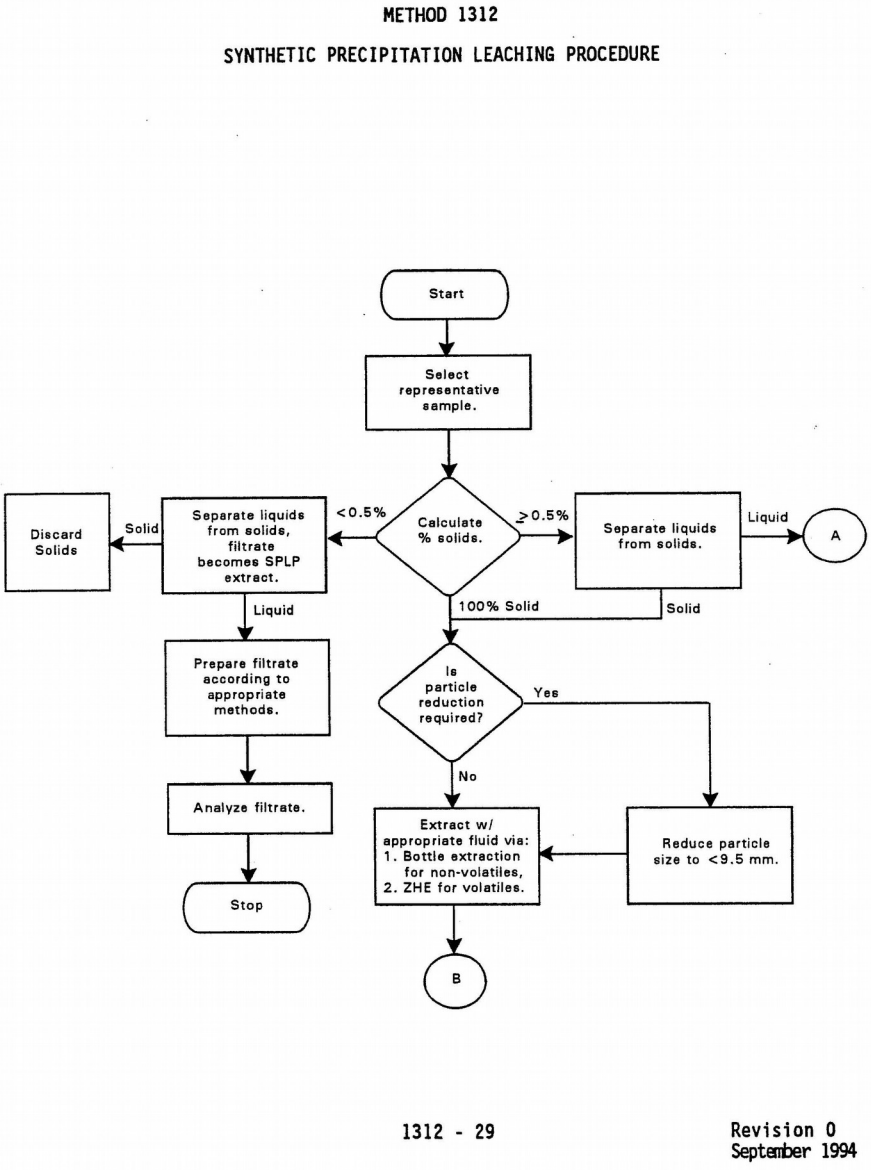
ROTARY AGITATION APPARATUS



TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 2

SPLP FLOW CHARTS



TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 3

SPLP FLOW CHARTS

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE (continued)

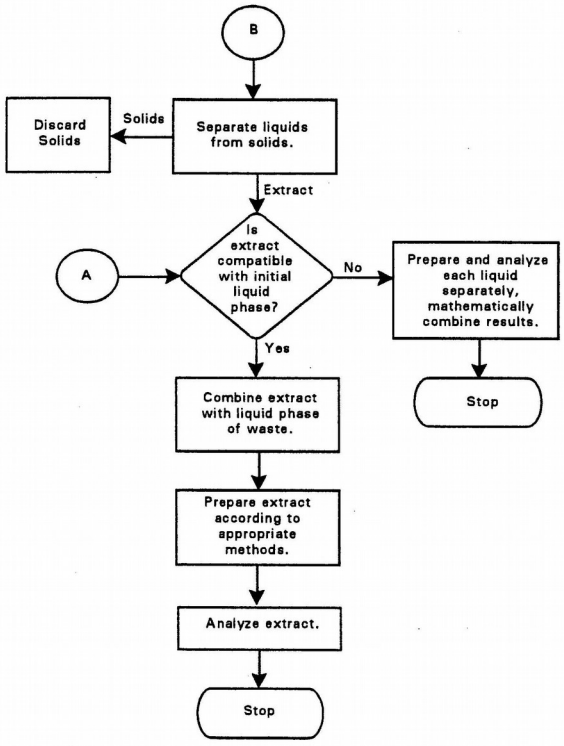


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FIGURE 4

SVOA SPLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for SPLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-502, current revision). Acid extractable compounds are at 100 ug/mL and base/neutral extractable compounds are at 50 ug/mL. 1.0 mL of this mix is added to the sample designated for the SPLP matrix spike.

Pyridine
1,4-Dichlorobenzene
2-Methylphenol
3-,4-Methylphenol*
Hexachloroethane
Nitrobenzene
Hexachlorobutadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorphenol
2,4-Dinitrotoluene
Hexachlorobenzene
Pentachlorophenol

***** Due to coelution on the GC/MS, 3-methylphenol and 4-methylphenol are reported as the combined concentration for the two isomers; the matrix spike solution contains 4-methylphenol at 100 ug/mL.

SURROGATE

The following surrogate compounds are reported for SPLP samples, although the surrogate mix also includes one additional surrogate (refer to SOP CA-502, current revision). Acid extractable surrogates are at 100 ug/mL and base/neutral extractable surrogates are at 50 ug/mL. 1.0 mL of this mix is added to all samples.

2-Fluorophenol	100 ug/mL
Phenol-d5	100 ug/mL
Nitrobenzene-d5	50 ug/mL
2-Fluorobiphenyl	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 5

PESTICIDE SPLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for SPLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-515, current revision). All compounds are at 0.5 ug/mL. 1.0 mL of this mix is added to the sample designated for the SPLP matrix spike.

Endrin
Heptachlor
Methoxychlor
Lindane
Heptachlor Epoxide

SURROGATE

Surrogates are at 1.0 ug/mL. 1.0 mL of this mix is added to all samples.

Decachlorobiphenyl (DCB)	1.0 ug/mL
Tetrachloro-m-xylene (TCMX)	1.0 ug/mL

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FIGURE 6

EXAMPLE PAGE FROM SPLP FLUID USE LOGBOOK

Katahdin Analytical Services, LLC

Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook

FLUID PREPARATION

TCLP ☐ SPLP ☑	~~TCLP~~ Fluid #:	Fluid Batch #:	Prep Date:	Prepared by:	Measured pH:
	~~1391~~ 1	1391	5/22/17	MD	4.15

SPLP MD 5/22/17

Reagent	Manufacturer's Lot Number	Reagent Volume (mL)	Reagent Mass (g)	Fluid Final Volume (L)
Glacial Acetic Acid			N.A.	
Sodium Hydroxide		N.A.		
0.6% Sulfuric Acid / 0.4% nitric acid	MR1793	2.5	N/A	20

MD 5/22/17

Matrix Spike: 10mL (MS) MW17051 into 100mL Extract

FLUID USE LOG

Katahdin Sample Number	TCLP Extraction Start Date	Extract to be Analyzed for: Metals	SVOA	Pest	Herb
Blank: PBTF391A	5/22/17	✓			
Matrix Spike: SK4095-4		✓			
SK4095-2A		✓			
SK4095-4A		✓			
SK4095-6A		✓			
SK4095-8B		✓			
SK6120-M	7-17-17	✓			

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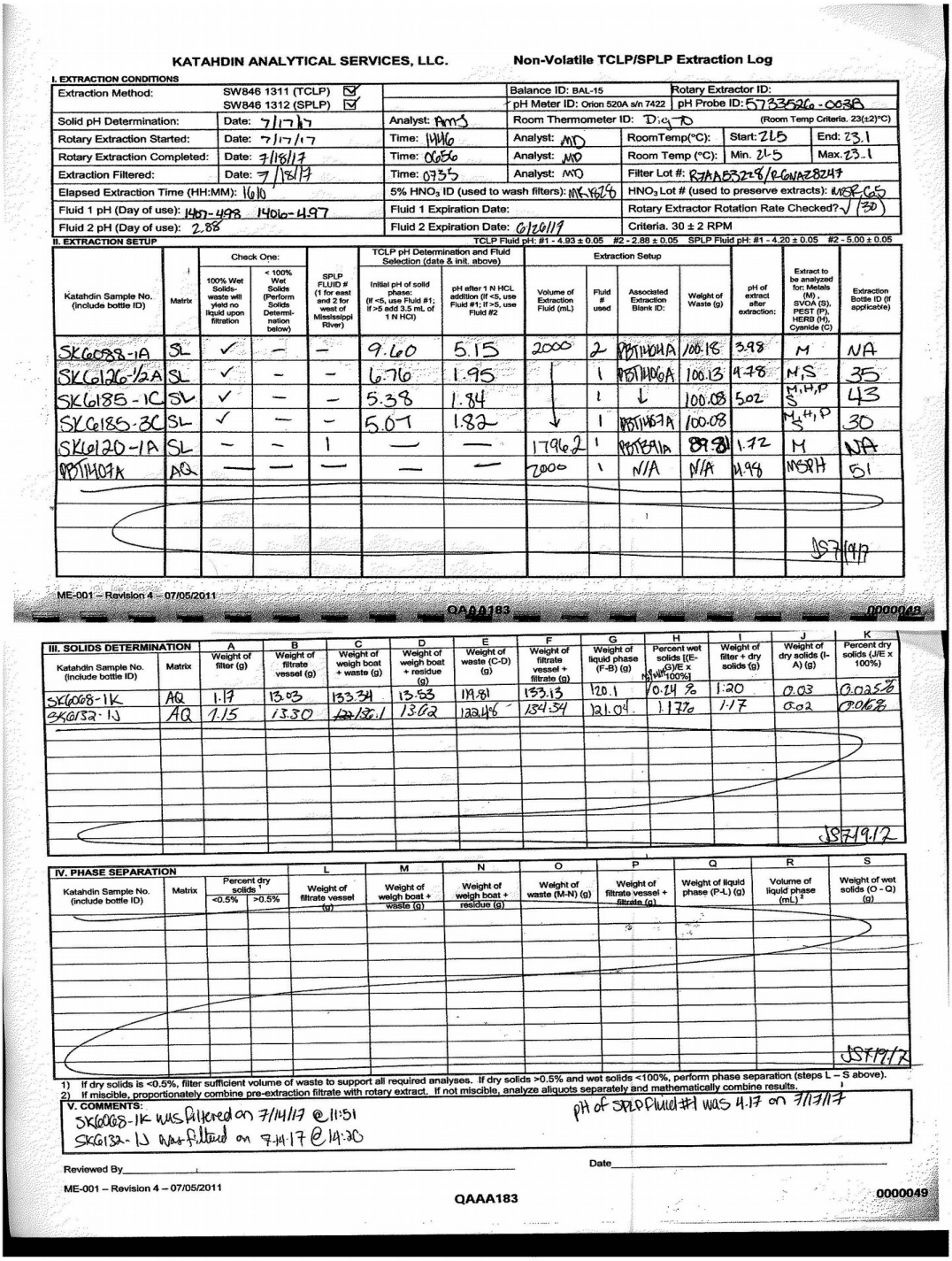
FIGURE 7

EXAMPLE PAGE FROM NON-VOLATILE TCLP/SPLP EXTRACTION LOGBOOK



KATAHDIN ANALYTICAL SERVICES, LLC. **Non-Volatile TCLP/SPLP Extraction Log**

I. EXTRACTION CONDITIONS

Extraction Method:	SW846 1311 (TCLP) ☑ SW846 1312 (SPLP) ☑		Balance ID: BAL-15	Rotary Extractor ID:		
			pH Meter ID: Orion 520A s/n 7422	pH Probe ID: 5733526a-0038		
Solid pH Determination:	Date: 7/17/17	Analyst: AMS	Room Thermometer ID: D:g-10	(Room Temp Criteria. 23(±2)°C)		
Rotary Extraction Started:	Date: 7/17/17	Time: 1446	Analyst: MD	RoomTemp(°C):	Start: 21.5	End: 23.1
Rotary Extraction Completed:	Date: 7/18/17	Time: 0656	Analyst: MD	Room Temp (°C):	Min. 21.5	Max. 23.1
Extraction Filtered:	Date: 7/18/17	Time: 0735	Analyst: MD	Filter Lot #: RJAA83278/R6NAZ8247		
Elapsed Extraction Time (HH:MM): 1610		5% HNO_3 ID (used to wash filters): MK-1625		HNO_3 Lot # (used to preserve extracts): MKBP665		
Fluid 1 pH (Day of use): 1407-498 1406-497		Fluid 1 Expiration Date:		Rotary Extractor Rotation Rate Checked? ✓ (30)		
Fluid 2 pH (Day of use): 2.88		Fluid 2 Expiration Date: 6/26/17		Criteria. 30 ± 2 RPM		

II. EXTRACTION SETUP TCLP Fluid pH: #1 - 4.93 ± 0.05 #2 - 2.88 ± 0.05 SPLP Fluid pH: #1 - 4.20 ± 0.05 #2 - 5.00 ± 0.05

Katahdin Sample No. (include bottle ID)	Matrix	Check One: 100% Wet Solids- waste will yield no liquid upon filtration	Check One: < 100% Wet Solids (Perform Solids Determination below)	SPLP FLUID # (1 for east and 2 for west of Mississippi River)	TCLP pH Determination and Fluid Selection (date & init. above): Initial pH of solid phase: (If <5, use Fluid #1; If >5 add 3.5 mL of 1 N HCl)	pH after 1 N HCL addition (If <5, use Fluid #1; If >5, use Fluid #2	Extraction Setup: Volume of Extraction Fluid (mL)	Fluid # used	Associated Extraction Blank ID:	Weight of Waste (g)	pH of extract after extraction:	Extract to be analyzed for: Metals (M), SVOA (S), PEST (P), HERB (H), Cyanide (C)	Extraction Bottle ID (if applicable)
SK60088-1A	SL	✓	–	–	9.60	5.15	2000	2	PBTHH04A	100.18	3.98	M	NA
SK60126-½A	SL	✓	–	–	6.76	1.95		1	PBTHH06A	100.13	4.78	M,S	35
SK6185-1C	SL	✓	–	–	5.38	1.84		1	↓	100.08	5.02	M,H,P S	43
SK6185-3C	SL	✓	–	–	5.07	1.82	↓	1	PBTHH07A	100.08		M,S,H,P	30
SK6120-1A	SL	–	–	1	—	—	17962	1	PBTHH01A	89.31	1.72	M	NA
PBTHH07A	AQ	—	—	—	—	—	2000	1	N/A	N/A	4.98	MSPH	51

JS 7/19/17

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III. SOLIDS DETERMINATION

Katahdin Sample No. (include bottle ID)	Matrix	A Weight of filter (g)	B Weight of filtrate vessel (g)	C Weight of weigh boat + waste (g)	D Weight of weigh boat + residue (g)	E Weight of waste (C-D) (g)	F Weight of filtrate vessel + filtrate (g)	G Weight of liquid phase (F-B) (g)	H Percent wet solids [(E-G)/E x 100%]	I Weight of filter + dry solids (g)	J Weight of dry solids (I-A) (g)	K Percent dry solids (J/E x 100%)
SK60088-1K	AQ	1.17	13.03	133.34	13.53	119.81	133.13	120.1	0.24 %	1.20	0.03	0.025%
SK60132-1J	AQ	1.15	13.30	~~132.1~~ 133.0	13.62	119.48	134.34	121.04	1.17%	1.17	0.02	0.017%

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IV. PHASE SEPARATION

Katahdin Sample No. (include bottle ID)	Matrix	Percent dry solids [1] <0.5%	Percent dry solids [1] >0.5%	L Weight of filtrate vessel (g)	M Weight of weigh boat + waste (g)	N Weight of weigh boat + residue (g)	O Weight of waste (M-N) (g)	P Weight of filtrate vessel + filtrate (g)	Q Weight of liquid phase (P-L) (g)	R Volume of liquid phase (mL) [2]	S Weight of wet solids (O - Q) (g)

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1) If dry solids is <0.5%, filter sufficient volume of waste to support all required analyses. If dry solids >0.5% and wet solids <100%, perform phase separation (steps L – S above).
2) If miscible, proportionately combine pre-extraction filtrate with rotary extract. If not miscible, analyze aliquots separately and mathematically combine results.

V. COMMENTS:
SK60088-1K was filtered on 7/14/17 @ 11:51
SK6132-1J was filtered on 7.14.17 @ 14:30
pH of SPLP Fluid #1 was 4.17 on 7/13/17

Reviewed By_____ Date_____

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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: Jol C. Burton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03.27.01

General Manager: Dennis P. Hufford 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 tebing 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 minimum 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 for 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 LLOC 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.

I acknowledge receipt of copy ___ of document **SOP CA-627-13**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
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Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-13**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

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.

List of Tables

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^{o} 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-701
Revision History
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Page 1

TITLE: BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES

Prepared By: Patty Gomez Date: 11/00

Approved By:

Group Supervisor: Keith Tangney Date: 02/60/1

Operations Manager: Jol C. Butler Date: 3/26/01

QA Officer: Deborah J. Nadeau Date: 2/15/01

General Manager: Duncan F. Shaffer Date: 2/15/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Removed references to HACH nutrient pillows. Made other minor edits to reflect current practices. Removed Attachment A.	DN	04/06	04/06
02	Made changes to Sections 5 and 7 to reflect current practices. Changed acceptance criteria for MS to 80-120% in Table 1. Changed Table 2 to refer to NELAC Interpretation instead of EPA Standards for QC Accuracy. Updated Attachment B and Figure 7.	LAD	04/08	04/08
03	Added alternate method for determination of total Residual chlorine. Added Na_2SO_3, replacing $Na_2S_2O_3 \cdot 5H_2O$. Also removed this from Table 2. Added reagents for this change to Sect. 5	LAD	06/08	06/08
04	Added information to sections 5.20, 7.17.2, 7.22 to reference effluent seed. Added EHSM, subsampling, QA506, DoD, NELAC and CA-101 references.	DN	08/09	08/09
05	Sect. 5.6 - Added that dilution water must stand in incubator for 24 hours prior to use. Sect. 7 - Replaced CAR with nonconformance report. Table 1 - changed TV of LCS to 164.7	LAD	06/10	06/10

TITLE: BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 4 – Added new meter and probe. Sect. 7 – Edited for new instrument throughout. Added correct seeded dilutions and removed references to polyseed. Corrected true value for LCS and MS. Replaced CAR with NCR. Other minor changes to reflect current practices. Sect. 9 – Added MDL, LOD & LOQ Information. Sect. 10 – Added and edited references. Table 1 – corrected CBOD true value. Updated logbook and BOD qualifier examples.	LAD	05/12	05/12
07	Sect. 5 - Removed heating dry reagents, added to store in dessicator. Sect. 7 - Removed Winkler Titration Calibration, corrected pH range, added 3rd LCS requirement, added Sample dup. requirement, corrected typos and removed redundant info. Sect. 10 - Added and updated references. Updated/added figures 1, 2, 5, 6, 7 (new), 8 and 9.	LAD	07/14	07/14
08	Sect. 1 - Removed Winkler & Azide definitions. Sect. 3 & 7 - Added 7.0 → 7.2 pH requirement. Sect. 5 - Removed obsolete reagents. Sect. 7 - Changed DO limit to 9.5, added # of blanks and LCS's to 3. Sect 2 & 7 - Added +/- 6 hours to 5 day incubation. Sect. 10 - Added SM4500 O G reference. Updated Fig. 3 & 6. Fixed typos. Changed KAS INC to KAS throughout.	LAD	07/15	07/15
09	Sect. 7 - Changed target pH range to 6.5-7.5, moved TRC neutralization by titration before sodium thiosulfate. Removed daily drift preparation and read. Added BOD Duplicate preparation and criteria, updated temperature verification. Updated References, Updated/Removed Figures	LAD	05/18	05/18
10	Added DO Table information and example. Updated references. Updated logbook example.	LAD	03/19	03/19

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE 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 **SOP CA-701-10**, titled **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-701-10**, titled **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES.**

Recipient: _____ Date: _____

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel for the analysis of biochemical oxygen demand (BOD), total and carbonaceous, in aqueous matrices in accordance with EPA Method 405.1 and SM 5210B. This SOP is applicable to the analysis of water, wastewater and seawater for total and carbonaceous BOD. A Practical Quantitation Level (PQL) of 2 mg/L is attainable using an initial sample volume of 300 mL.

1.1 Definitions

Carbonaceous Demand – A measure of the oxygen utilized during a specified incubation period for the biochemical degradation of organic material.

Nitrogenous Demand – A measure of the oxygen used to oxidize reduced forms of nitrogen.

BOD-5 - Carbonaceous demand plus nitrogenous demand

CBOD – Carbonaceous demand only

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the analysis of BOD by EPA Method 405.1 and SM 5210B. 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 BOD by EPA 405.1 and SM 5210B 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 follows 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 (MSDS) 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 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 Plan for further details on pollution prevention techniques.

Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and 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 lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

A sample of waste, or an appropriate dilution, is incubated for 5 days +/- 6 hours at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

3.0 INTERFERENCES

All samples are checked for residual chlorine before analysis and treated if residual chlorine is present.

Samples for BOD analysis may degrade significantly during storage between collection and analysis, resulting in low BOD values. Analyze 24-48 hours after collection See section 6 for additional information).

Samples with a pH of < 6 or > 8 need to be neutralized to pH 7.0 - 7.2

Headspace in the sample collection bottle may result in erroneous results.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

4.0 APPARATUS AND MATERIALS

4.1 Dissolved Oxygen Meter, Orion Star Plus or equivalent

4.2 BOD probe, Thermo Scientific BOD Auto-Stir or equivalent

4.3 Replacement membranes and probe filling solutions

4.4 Incubator, capable of maintaining temperature of 20° ± 1°C

4.5 300 mL Wheaton BOD bottles, clear, with stoppers and caps. All BOD bottles must be cleaned as described in the current revision of Katahdin SOP, CA-100, Labware Cleaning.

4.6 Volumetric flasks, Class A, 1 L and 100 mL

4.7 Graduated cylinders, 100 mL, 50 mL and 25 mL

4.8 Glass Beakers, 400 mL and 1 L

4.9 Burette, 10 mL, 0.01 mL graduations

4.10 Eppendorf calibrated adjustable pipettors or equivalent, 5 and 1 mL

4.11 Disposable pipette tips, 5 and 1 mL

4.12 Potassium Iodide Starch Test Paper

4.13 Teflon magnetic stir bars

4.14 Stir plate

4.15 Wide-range pH paper

4.16 Narrow-range pH paper

4.17 Small scoop

4.18 20 L plastic carboys

4.19 Analytical balance capable of weighing to ± 0.0001 g

4.20 Hach Pocket Colorimeter II.

5.0 REAGENTS AND STANDARDS

5.1 DI water, laboratory reagent grade water

Dilution Water Reagents

5.2 Phosphate buffer solution, Banco or equivalent

5.3 Magnesium sulfate solution - 22.5 g $MgSO_4 \cdot 7H_2O$ diluted to 1 L with laboratory DI water, or purchased equivalent

5.4 Calcium chloride solution - 27.5 g $CaCl_2$ diluted to 1 L using laboratory DI water, or purchased equivalent

5.5 Ferric chloride solution - 0.25 g $FeCl_2 \cdot 6H_2O$ diluted to 1 L using laboratory DI water, or purchased equivalent

5.6 Dilution water – Dilution water must be prepared with Laboratory DI water that has been incubated at 20 °C for at least 24hrs before use. The water should be incubated in 20 L plastic carboys that have been precleaned and dedicated exclusively to BOD dilution water. At the time of analysis, prepare the dilution water by adding 20 mL of calcium chloride solution (5.4), 20 mL of ferric chloride solution (5.5), 20 mL of the magnesium sulfate solution (5.3), and 20 mL of phosphate buffer (5.2) to the incubated laboratory DI water. The carboy must be full to the 20 L mark with the incubated laboratory DI water. This mixture should be stirred completely.

Sample Preparation Reagents

5.7 Sulfuric acid, 1 N, Baker analyzed high purity acid or equivalent

5.8 Sodium Hydroxide, 1 N, certified ACS or equivalent

5.9 Sodium thiosulfate solution, 0.025 N, HACH or equivalent

Analysis Reagents

5.10 Glucose-Glutamic Acid solution (also known as L-Dextrose) – In a 1 L volumetric flask, combine 150 mg dried glucose and 150 mg dried glutamic acid diluted to 1 L with laboratory DI water (5.7). Store dry reagent in dessicator prior to use. Prepare fresh daily.

5.11 Nitrification Inhibitor (2-chloro-6-(trichloromethyl) pyridine or, TCMP) - HACH or equivalent. (Refer to Attachment A for Nitrification Inhibitor Specifications.)

5.12 “Seeding” solution – Primary effluent from wastewater facility or PolySeed® – BOD Inoculum (Interlab) –Please refer to Attachment B for PolySeed® specifications. The prepared seeding solution is good for up to 6 hours. When using the primary effluent, an aliquot should be poured off into a beaker and spinning on a stir plate while in use.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample bottles should be filled leaving no headspace. Store samples at or below 4°C from collection until analysis. Warm chilled samples to room temperature before analysis. Start analysis no later than 24 hours after sampling (Standard Methods 5210B) or 48 hours after sampling (40 CFR 136). Please note that for compliance samples originating in the State of Maine, the Maine DEP only recognizes the Standard Method 5210B 24-hour holding time.

7.0 PROCEDURES

SAMPLE PRETREATMENT - pH AND INTERFERENCE CHECKS

7.1 Bring samples to 20 ± 1 °C before making dilutions

7.2 When sample volumes of 25 mL or more are used, the sample should be warmed as in 7.1 and shaken vigorously to decrease the dissolved oxygen (DO) present in the sample. If this is not done, the initial DO reading for that sample may exceed the limit of 9.5 mg/L of DO.

7.3 Pour small aliquot of sample in dose cup. Check pH of sample using multi-range pH test strips. The target range is 6.5 to 7.5. If the sample is outside of this range, adjust a pH between 7.0 and 7.2, with a solution of sulfuric acid or sodium hydroxide of sufficient strength so that the quantity of solution added does not dilute the sample aliquot by more than 0.5% (1 N is suggested).

7.4 Prior to analysis of the samples test for the presence of residual chlorine.

7.4.1 Test a small aliquot of sample using the Hach Pocket Colorimeter II. Please refer to the current revision of KAS SOP CA-765, “Spectrophotometric Analysis of Total Residual Chlorine Using DPD” for instructions. Record result in the BOD logbook.

7.4.2 If chlorine is present, remove a 100 mL aliquot. To this sample add 1 ml 1+50 H_2SO_4 and 1 mL potassium iodide solution.

7.4.3 Titrate with Na_2SO_3 solution to the starch-iodine end point for residual chlorine.

7.4.4 Determine the amount of Na_2SO_3 solution required to neutralize the chlorine in the sample and any sample dilutions.

7.4.5 Add Na_2SO_3 solution to the sample. Retest for residual chlorine after 10 – 20 minutes.

7.4.6 Alternatively, If there is not enough sample to titrate

7.4.6.1 Use the same aliquot of sample in dose cup as pH check.

7.4.6.2 Wet KI test strip with sample. Record test result (+/-) in the BOD logbook (Figure 1).

7.4.6.3 If the test strip turns blue-black (+), residual chlorine is present. No blue/black color indicates a negative result.

7.4.6.4 If residual chlorine is present, aliquot enough sample for analysis (enough for the appropriate dilutions) into a disposable cup or vial.

7.4.6.5 Add one to two drops of 1.0 N sodium thiosulfate to the vial or cup and mix using a stir bar and stir plate. Repeat step 7.4.6.2. If residual chlorine is still present, repeat the addition of 1.0 N sodium thiosulfate until no blue/black color is present on the KI test strip.

7.4.6.6 Record the required number of sodium thiosulfate drops in the logbook.

DISSOLVED OXYGEN (DO) METER CALIBRATION AND MAINTENANCE

7.5 Turn the meter on and using the line select button move the cursor on the meter to last line marked DO

7.6 Visually inspect the probe membrane for tears, oily residues, or fingerprints. Inspect for air bubbles under the membrane. If any of the above are present, replace the membrane cap.

7.6.1 Disconnect the probe from the meter. Remove the stir paddle from the probe by pulling it straight out.

7.6.2 Unscrew the old membrane cap from the probe. Before installing a new membrane, clean the probe tip with deionized water in order to remove any contaminants.

7.6.3 Hold the membrane cap and fill it about three quarters full with the electrolyte solution provided. Screw the membrane cap onto the probe moderately loose. A small amount of the electrolyte should overflow.

7.6.4 Rinse off excess electrolyte from the probe with deionized water.

7.6.5 Reinstall the stir paddle. The probe must be polarized before use. To polarize a probe, attach the probe to the meter, connect the meter to a power supply and wait 30 to 60 minutes. The probe is continuously polarized when it is connected to the meter, so this process does not need to be repeated unless probe maintenance is preformed or the probe is disconnected from the meter for more than an hour

7.6.6 The membrane cap and electrolyte solution should be replaced once a month.

7.6.7 Store the probe in the calibration chamber or a BOD bottle with about 50ml of distilled water in the bottle.

7.6.8 Record any maintenance including probe replacement in the BOD Probe Maintenance Log (Figure 2).

7.7 To prepare the calibration sleeve remove the cap from the sleeve and remove the sponge from the cap. Saturate the sponge with distilled water and then squeeze all the excess water out of sponge. Wipe off any excess water from the sleeve, the cap, and or the probe itself. Reassemble the calibration sleeve an insert the DO probe.

7.8 Allow five minutes for the probe to equilibrate.

7.9 Press the **calibrate** key. When the reading stabilizes the meter will display 102.3% saturation and then proceed to the measurement mode.

7.10 Immediately after calibration of the meter, place the DO probe in a BOD bottle containing approximately 50 mL of deionized water, making sure that any droplets of water have been blotted off the probe membrane. This bottle should be recapped when not in use to maintain equilibrium. Press the measurement button on the probe and record the DO reading.

7.11 Using the measured temperature and the measured barometric pressure in the building find the solubility of oxygen in the USGS Dissolved Oxygen tables found at tps://water.usgs.gov/software/DOTABLES/. The measured dissolved oxygen must be within 0.2 mg/L of the value obtained from the USGS DO Tables. Refer to Figure 8 for a copy of the USGS Dissolved Oxygen table ranging from 15.0 to 24.9 ^{o}C and 750 to 809 mm Hg.

7.12 If the measured DO in the bottle is outside this range, recalibrate the meter and reverify the calibration. If the calibration check again falls outside the acceptance range, perform corrective action (such as changing the probe membrane) before proceeding.

7.13 Perform the calibration check procedure at least once per hour while the meter is in use.

PREPARATION OF SAMPLE DILUTIONS

7.14 The following quality control samples must be prepared for each analytical batch of twenty or fewer samples:

7.14.1 (3) Unseeded Dilution Waters - 300 mL of prepared dilution water.

7.14.2 (4) Seeded Dilution Waters (for seed correction) –1 mL, 2 mLs, 4mLs, and 6 mLs of primary effluent in four different BOD bottles diluted to 300 mL with the prepared dilution water. Volume adjustments will need to be made when using the PolySeed®as the seeding solution.

7.14.3 (3) Laboratory Control Samples (LCS) - True value = 198 mg/L – 6.0 mLs of the Glucose-Glutamic Acid solution prepared in section 5.18 brought up to 300 mL with dilution water.

7.14.4 (1) Matrix Spike Sample per ten samples - True value = 198 mg/L – 6.0 mLs of the Glucose-Glutamic Acid solution prepared in section 5.18 added to the appropriate sample dilution and brought up to 300 mL with the prepared dilution water.

7.14.5 (1) Sample Duplicate per twenty samples – The Laboratory shall analyze a duplicate sample of a mid-range dilution. This will be compared to the final result of the sample and the relative percent difference will be calculated

RPD Criteria is $\leq$30. If the duplicate RPD is out of criteria narrate or flag appropriately.

7.15 The remainder of the batch consists of sample dilutions (field samples). Refer to Figure 3 for an example analytical run.

7.16 For samples with unknown analytical history, the sample dilution scheme may consist of up to ten dilutions: 0.050, 0.10, 0.50, 1.0, 2.0, 5.0, 10, 50, 100, and 300 mL of sample. The type of sample may help in determining the dilution scheme.

7.16.1 Wastewater samples would probably need the lower dilutions while surface water and ground water samples may not need to be diluted as much.

7.17 If analytical history is available for the site, or if an expected BOD range has been provided, prepare the sample dilution range expected with one extra dilution above and one extra below the anticipated range. Refer to Figure 4 for guidance on preparing the appropriate sample dilutions for known BOD concentration ranges.

7.18 When making sample dilutions, volumetric glassware (volumetric pipettes and volumetric flasks) and calibrated adjustable pipettes should be used. Sample dilutions should be made so as to maximize the amount of native sample used. This will provide for a more representative portion of sample. If a large dilution is required it is better to do a serial dilution rather than using a non-representative aliquot of sample. (Example: A sample requires a 1/1000 dilution. Make a 1/100 dilution of the native sample and then a 1/10 dilution of the initial dilution). Incubated dilution water must be used to make sample dilutions.

Note: Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.19 Once the BOD logbook has been filled out and the appropriate dilution scheme determined, the samples can be prepared.

7.20 All samples, LCSs and "seeded" dilution water blanks (4) are typically seeded with primary effluent (seed). Alternative seed may be used at client request. In this case, guidance on the appropriate volume of seed to add should be obtained from the client (via the Katahdin project manager).

7.20.1 In the case where an alternative seeding material is used, seed controls (7.10) must also be prepared using this seeding material.

7.21 First, the "seeded dilution water" bottles are seeded with 1, 2, 4 and 6 mLs of primary effluent respectively, as described in section 7.18. The samples and LCSs are seeded with 1 mL of primary effluent seed. Seed is not added to the two "unseeded dilution water" bottles. These are used to evaluate the quality of the unseeded dilution water.

7.22 The 6 mLs of glucose-glutamic acid spiking solution should be added to the three LCS bottles and the matrix spike bottle.

7.23 The appropriate amounts of sample and dilution water are then added to each BOD bottle. This should be done one BOD bottle at a time with the initial DO measured after each bottle is prepared (refer to steps 7.20 to 7.23 for initial DO measurement).

MEASUREMENT OF INITIAL DISSOLVED OXYGEN (DO)

7.24 Starting with the "Unseeded dilution Water", fill the bottle, carefully, to the midpoint of the bottleneck. Fill bottles with enough dilution water so that insertion of the stopper will displace all air, creating a water-seal. The total volume in the bottle will be approximately 300 mL. While filling the bottle, it is very important that the rate of filling does not aerate the sample, i.e. there should not be any bubbles formed during the filling process.

7.25 Insert the probe into the bottle. Press the measurement button and record the stabilized readings in the BOD logbook. All measurements must be documented in the BOD logbook as they are taken. The DO electrode must be rinsed in between readings to prevent cross-contamination.

7.26 If the sample is being analyzed for carbonaceous BOD, add four scoops of the Nitrification Inhibitor then stopper and cap the bottle. Fill bottles with enough dilution water so that insertion of the stopper will displace all air, creating a water-seal.

Note: Carbonaceous BOD and total BOD are always analyzed in separate batches. One or the other must be indicated in the BOD logbook at the top of the page (Figure 1). The nitrification inhibitor must be added to all samples in a batch of CBODs (i.e. unseeded dilution water, seeded dilution water, LCSs and client samples).

7.27 If the sample is being analyzed for total BOD, replace any displaced contents with dilution water to the mid-point of the neck; stopper and cap the bottle. Fill bottles with enough dilution water so that insertion of the stopper will displace all air, creating a water-seal.

7.28 Repeat steps 7.20 to 7.23 until all bottles are read.

7.29 Transfer the bottles to the incubator. Record the times the initial DO is read for each sample in the start time column of the BOD logbook. Start times must be unique for each sample. Allow the samples to incubate for five days +/- 6 hours. The incubator temperature should be 20 ± 1°C. The incubators are equipped with digital thermometers that are programmed to record temperatures every 10 minutes. The QAO maintains temperature documentation.

MEASUREMENT OF FINAL DISSOLVED OXYGEN

7.30 On the fifth day, prepare and calibrate the instrument as described in Steps 7.5 - 7.9.

7.31 Remove the samples from the incubator and record the times that the final DO is read for each sample in the end time column of the BOD logbook. End times must be unique for each bottle.

7.32 Remove the caps from all the bottles. Remove the stopper from each bottle just prior to reading the DO for the bottle. (i.e. Do not remove the stoppers from all bottles at the same time or oxygen exchange between the samples and the atmosphere may occur.) Record the DO reading for each sample, blank, and LCS in the BOD logbook. All measurements must be documented in the BOD logbook as they are taken. The DO electrode must be rinsed in between readings to prevent cross-contamination.

CALCULATIONS AND REPORTING OF RESULTS

7.33 Transfer all raw data to the BOD calculation spreadsheet (Figure 5).

7.34 The “Unseeded Dilution Water” must have a final “DO Consumed” (absolute value) of less than 0.20 mg/L. If either of the unseeded dilution water blanks exceeds this acceptance criterion, the accuracy of the sample results may be biased. Therefore, a Non-Conformance report must be initiated and the reported result will need to be qualified. (In some cases, e.g. permit compliance monitoring, the client may want to resample, therefore it is important to notify the project manager for guidance on how to proceed.) If values are to be reported, refer to the laboratory’s standard list of qualifiers for the appropriate notation. Refer to Figure 8.

7.35 To determine the “Seed Correction Factor” (SCF), the DO consumed for each or all of the “Seeded Dilution Water” samples must be >2.0 mg/L with a final DO >1.0 mg/L. Determine the SCF using the following equation:

$$SCF = \frac{\text{(Average BOD) * 6 mL (or seed volume used for actual samples)}}{300\ mL}$$

Ideally, the seed correction factor should be between 0.6 and 1.0 mg/L, as required by the method. A SCF not meeting this criterion may indicate an unreliable seed source. Please refer to Attachment C for troubleshooting the seed source.

7.36 LCS's and all sample results are calculated using the following equation:

$$BOD\ (mg/L) = \frac{[(\text{Initial DO} - \text{Final DO}) - SCF]\ (mg/L) * 300\ mL}{\text{Initial Sample Volume (mL)}}$$

7.37 Calculate LCS recoveries. LCS results for total BOD and CBOD must be within the method specified criteria of 198 ± 30.5 mg/L (85 - 115% recovery) for the run to be acceptable. This criteria is based on a regression equation determined for method using a theoretical 300 mg/L standard solution. The regression equation for the mean value, X, and the standard deviation, SD, is as follows:

X = 0.658 (300 mg/L true value) + 0.280 mg/L = 198 mg/L
SD = 0.100 (300 mg/L true value) + 0.547 mg/L = 30.5 mg/L

7.38 If the mean of all 3 LCS recoveries is outside the acceptance criteria or if all 3 LCS recoveries are outside the acceptance criteria, a Non- conformance must be initiated and the reported results will need to be flagged. (In some cases, e.g. permit compliance monitoring, the client may want to resample, therefore it is important to notify the project manager for guidance on how to proceed.) If values are to be reported, refer to the laboratory's list of BOD data qualifiers for the appropriate flag. Refer to Figure 7.

7.39 Evaluate the data for the client samples using the following criteria and report results as described below:

7.39.1 The "DO Consumed" must be > 2.0 mg/L and the final DO must be > 1.0 mg/L.

7.39.2 If only one dilution for a given sample meets the criteria, report the calculated BOD for that dilution. If more than one dilution for a given sample meets these criteria, determine the mean calculated BOD for the dilutions and report the mean.

7.39.3 If the DO consumed is not > 2.0 mg/L for all dilutions, report a value of < X mg/L, where X is the BOD value obtained from the equation in 7.38 using a value of 2.0 mg/L of DO consumed for the highest amount of sample used. If X is not <2, then a notation indicating an elevated PQL and insufficient DO consumed must be used (Figure 8).

7.39.4 If the final DO is < 1.0 mg/L for all the dilutions for a given sample, a reported value is calculated from the greatest dilution (i.e., smallest sample volume) assuming the final DO is 1.0 mg/L, and the result is notated with a >

value and with the appropriate notation form the standard notations list (Figure 8).

7.39.5 If the calculated BOD decreases with an increase in sample volume analyzed, matrix interference may be present and inhibition of the seed may have occurred. In this case the reported result is notated as such (Figure 8).

7.39.6 Other dilution scenarios that do not meet the requirements of the method should be reviewed by the Department Manager, Operations Manager or QAO for further reporting instructions. Other reporting alternatives, such as reporting estimated, "J" flagged data (results between the MDL and the PQL) rather than elevated PQLs may be used if approved by the group supervisor or any of the staff listed previously.

7.40 Matrix spike recoveries should be calculated using the native sample result obtained using the same aliquot as used for the matrix spike (i.e. both native sample and matrix spike have the same dilution factor). The matrix spike true values are the same as the LCS for the appropriate TBOD or CBOD.

7.41 Evaluate the spreadsheet results for the criteria specified in Steps 7.30 through 7.36. Corrective action must be initiated when the above criteria are not consistently met. Refer to Attachment C.

7.42 Enter spreadsheet results, including sample preparation information, measured sample concentrations, and quality control data, into the Katahdin Information Management System for calculation and reporting. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information. A batch sheet is generated (Figure 8). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.43 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 section 7 and Table 1 for QC Requirements, Frequency and Acceptance Criteria. 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 the entire QC. In some cases data may be reported, but may not be 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, Operations Manager, and/or

Quality Assurance Officer may be consulted to evaluate data. Due to the 48-hour hold time and 5-day incubation period associated with this method, QC excursions are not discovered until after the expiration of the holding 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.

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 revisions of Method 5210 B for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Methods for Chemical Analysis of Water and Wastes, US EPA-600/4-79-020, 1979 revised 1983, Method 405.1, “Biochemical Oxygen Demand”.

Standard Methods for the Examination of Water and Wastewater", Method 5210B, 5-Day BOD Test, 21st Edition, 2005, approved by Standard Method Committee, 2001.

Standard Methods for the Examination of Water and Wastewater", Method 5210B, 5-Day BOD Test, 22nd Edition, 2012, approved by Standard Method Committee, 2011.

Standard Methods for the Examination of Water and Wastewater", Method 4500O G, Oxygen (Dissolved) Membrane Electrode Method, 22nd Edition, 2012, approved by Standard Method Committee, 2001, editorial revisions 2011.

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 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: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

LIST OF TABLES, FIGURES AND ATTACHMENTS

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
BOD - EPA 405.1 SM 5210B	Unseeded Dilution Waters	Three per prep batch	< |0.40| mg/L	Qualify data. Refer to Figure 8 for appropriate notation.
	Seeded Dilution Waters	Four per prep batch	0.6 to 1.0 mg/L	Investigate seed source or other possible sources of error. See Attachment C.
	Laboratory Control Samples (LCS)	Three per prep batch	198 mg/L ± 30.5 mg/L (85-115%R)	Investigate seed source or other possible sources of error. See Attachment C.
	Matrix Spike	One MS per 10 samples	80-120 %R	Investigate seed source or other possible sources of error. See Attachment C.
	Sample Duplicate	One per batch of 20 samples	RPD $\leq$30 for sample > 3X the PQL and <100% RPD for samples < 3X the PQL.	If the sample duplicate has a reportable result, report with narration
	Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter.	Must pass all applicable QC requirements for method	Repeat analysis until able to perform passing QC: document successful performance in personal training file
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-701-10	Method EPA 405.1 SM 5210B (21st)
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 1

EXAMPLE OF BOD LOGBOOK PAGE

WG244240 R498657

KATAHDIN ANALYTICAL SERVICES - BIOCHEMICAL OXYGEN DEMAND

Check the appropriate Test and Method:	If CBOD is checked below, this indicates that 0.16g of nitrification inhibitor has been added to each bottle below.	pH Meter Calibration	Probe ID:
Total BOD: SM5210B EPA 405.1	**PQL: 2.0 mg/L**	True Value / Lot ID / Actual Value / Accept? ± 0.05	
Carbonaceous BOD: SM5210B	**PQL: 2.0 mg/L**	4.01	
Glutamic Acid Std (GGA) ID: GGA 1/9/19	GGA Preparation:	Seed Used Source ID and Amount:	7.00
Nitirification Inhibitor ID:	0.15g Glucose ID: SWL3604	LAWPCA: 1 mL	10.01
Phosphate Bffr Soln. ID: SWL4404		SSD: ✓ 1 mL	DO Meter ID: B27869 / DO Probe ID: 016407
$H_{14}MgO_{11}S$ ID: 181291	0.15 g Glutamic Acid ID: SWL3653	$Na_2S_2O_3$ ID:	KI Paper ID: 07182018 / pH Paper ID: 170704005
Ferric Chloride Soln. ID: SWL4375		Starch Indicator ID:	Carboy ID: H / 1:50 H_2SO_4 ID:
Calcium Chloride Soln. ID: SWL4369		Potassium Iodide Soln. ID:	Pipet IDs: W3,4,7
Acid 1 ID (A1):	Acid 2 ID (A2):	Base1 ID (B1):	Base 2 ID (B2):

SAMPLE ID	SITE ID	START TIME	pH 6.5 - 7.5?	pH 7.0-7.2?	Acid/Base Used	CL- Y/N	BOTTLE ID#	VOL (ml)	D.O. DAY 1	D.O. DAY 5	END TIME	SMP TEMP DAY 1 17-23 °C Y / N *
Blank		11:00	✓	n/a	NA	N	XYZ	300	8.16	7.96	1115	Y
"		01	"	"	"	"	P90	"	8.12	7.99	1119	"
"		02	"	"	"	"	P40	"	8.15	8.07	1120	"
1		03	"	"	"	"	2129	1	8.16	7.38	1122	"
2		04	"	"	"	"	160	2	8.00	6.61	1123	"
4		06	"	"	"	"	331	4	8.13	5.40	1125	"
6		07	"	"	"	"	101	6	8.05	3.99	1128	"
LCS		08	"	"	"	"	192	"	8.06	3.64	1131	"
"		09	"	"	"	"	T15	"	8.16	3.50	1132	"
"		10	"	"	"	"	1019	"	8.01	3.97	1135	"
SM0215-5	Strem 6M/N	09:54	✓			N	9220	25	8.25	7.04	1224	
"	1/7/19	55	"			"	A17	50	8.20	6.44	1226	
"		56	"			"	608	100	8.22	5.49	1227	
"		57	"			"	T1	300	8.14	2.89	1228	
TSM0192	HP Hood	10:02	✓			N	130	0.25	8.09	5.46	1230	
"		03	"			"	207	0.5	8.24	4.33	1231	
"		04	"			"	240	1	8.08	1.28	1232	

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TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 1 (cont.)

EXAMPLE OF BOD LOGBOOK PAGE

SAMPLE ID	SITE ID	START TIME	pH 6.5 - 7.5?	pH 7.0-7.2?	Acid/Base Used	CL- Y/N	BOTTLE ID#	VOL (ml)	D.O. DAY 1	D.O. DAY 5	END TIME	SMP TEMP DAY 1 17-23 °C Y / N *
SM0192-1	HP Hood	10:05	✓	NA	NA	N	P1	2	8.12	<1	1233	T
		06					Z00	5	8.19	<1	1233	
SM0216-S	Strom 7MW	10:10	✓			N	35 A11	25	7.96	6.40	1234	
	1/7/19	11					50 33P	50	7.82	5.18	1236	
		12					100T18	100	7.28	3.32	1237	
		13					300T3	300	5.23	<1	1238	
SM0193-1	Lone Pine	10:17	✓			N	177K	0.025	8.13	7.18	1239	
		18					F1	0.05	8.17	7.187	1241	
		21					288M	0.1	8.13	6.91	1242	
		22					430	0.25	8.14	6.30	1243	
		23					GZ	0.5	8.16	5.22	1244	
		24					177C	1	8.18	3.01	1245	
SM0194-1	Sebago Brewing	10:29	✓			N	302J	0.1	8.21	<1	1246	
		30					205	0.25	8.01	<1	1246	
		31					129	0.5	8.16	<1	1247	
		32					T19	1	8.19	<1	1248	
		33					1102	2	8.17	<1	1249	
SM0198-1	Clean Harbors	10:40	✓			N	6789	0.05	8.20	6.59	1251	
		41					1778	0.1	8.11	5.76	1252	
		42					903	0.25	8.17	3.49	1252	
		43					816A	0.5	8.14	1.45	1254	
		44					A7	1	8.16	<1	1254	
		45					10K	2	8.12	<1	1254	

ZF 1/9/19

1/14/19

NOTES:

ANALYST 1 ZF	DATE 1/9/19
ANALYST 2 RD/AP	DATE 1/14/19
CHECKED BY: SR	DATE 1-15-19

* If N, please comment why
LAWPCA = Lewiston-Auburn Water Pollution Control Authority, SSD=Scarborough Sanitary District
H14MgO11S = Magnesium Sulfate Heptahydrate

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TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 2

BOD PROBE MAINTENANCE LOG

KATAHDIN ANALYTICAL SERVICES, INC.
BIOCHEMICAL OXYGEN DEMAND (BOD) – METHODS 405.1 & 5210B
BOD PROBE MAINTENANCE LOG

DATE	INIT.	PROBE SERIAL #	PROBLEM OR PREVENTATIVE?	MAINTENANCE	DATE RETURN TO CONTROL
7/19/12	CBT	086030MD	~~REPLACED~~ CBT PROBLEM: DO PROBE STIR PADDLE NOT WORKING	REPLACED DO PROBE	
8/8/12	CBT	"	PROBLEM W/ MEMBRANE CAP LOOSE/RIPPED ON PROBE	REPLACED MEMBRANE CAP & ELECTROLYTE SOLUTION	
10/17/13	DW	"	Problem: OC (LCS) failing low	Replaced Membrane Cap & Electrolyte Solution	
10/29/13	ZS	086030MD	Error 809 – polarization of cathode destabilized	Removed membrane cap and replaced electrolyte solution. Same membrane cap replaced onto probe. Left to polarize for 1.5hrs.	

PLEASE EXPLAIN IN DETAIL WHAT THE PROBLEM IS OR IF IT IS ROUTINE MAINTENANCE. DESCRIBE IN DETAIL THE MAINTENANCE PERFORMED.

QAWL252

0000020

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 3

EXAMPLE OF TYPICAL BOD ANALYTICAL RUN SEQUENCE

1 unseeded dilution water
1 unseeded dilution water
1 unseeded dilution water
1 seeded dilution water
1 seeded dilution water
1 seeded dilution water
1 seeded dilution water
1 glucose/glutamic acid check standard
1 glucose/glutamic acid check standard
1 glucose/glutamic acid check standard
1st dilution – sample a
2nd dilution – sample a
3rd dilution – sample a
4th dilution – sample a
matrix spike – sample a
1st dilution – sample b
2nd dilution – sample b
3rd dilution – sample b
4th dilution – sample b
etc.

Sample dilution schemes may contain not always contain four dilutions. Wastewaters may contain more than four dilutions and groundwater/surface water may contain less than four dilutions. This is an example only.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 4

PREPARATION OF APPROPRIATE SAMPLE DILUTIONS
FOR KNOWN BOD CONCENTRATION RANGES

Table 22-1 BOD measurable with various dilutions of samples

Using percent mixtures		By direct pipeting into 300-ml bottles	
% mixture	Range of BOD	ml	Range of BOD
0.01	20,000–70,000	0.02	30,000–105,000
0.02	10,000–35,000	0.05	12,000– 42,000
0.05	4,000–14,000	0.10	6,000– 21,000
0.1	2,000– 7,000	0.20	3,000– 10,500
0.2	1,000– 3,500	0.50	1,200– 4,200
0.5	400– 1,400	1.0	600– 2,100
1.0	200– 700	2.0	300– 1,050
2.0	100– 350	5.0	120– 420
5.0	40– 140	10.0	60– 210
10.0	20– 70	20.0	30– 105
20.0	10– 35	50.0	12– 42
50.0	4– 14	100	6– 21
100	0– 7	300	0– 7

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 5

BOD CALCULATION SPREADSHEET

KATAHDIN ANALYTICAL SERVICES, INC.
Biochemical Oxygen Demand - Methods EPA 405.1 & SM 5210B

Page 1
Printed 1/15/2019
Filename: TBOD 011419

TBOD5 [X] **CBOD5** [ ]

Data Entry By: ZF/AP

BOD5,mg/L=(DO consumed- Seed Correction Factor)* Total Volume in mL /Sample Volume in mL
DO consumed = Initial DO - Final DO
Total Volume = 300 mL
Volume of seed added to each sample including (LCSs and MS) in mL = 1

Sample ID	Seed Volume mL	Initial DO D1	Final DO D2	Cons. DO D1-D2	Seed Correction Factor	In Criteria?
Blank 1	0	8.16	7.96	0.2	N/A	Yes
Blank 2	0	8.12	7.99	0.13	N/A	Yes
Blank 3	0	8.15	8.07	0.08	N/A	Yes
Seed Control 1	1	8.16	7.38	0.78	0.78	No
Seed Control 2	2	8.00	6.61	1.39	0.695	No
Seed Control 3	4	8.13	5.40	2.73	0.6825	0.6825
Seed Control 4	6	8.05	3.99	4.06	0.676666667	0.67666667
			Mean Seed Correction Factor (SCF):			**0.67958333**

LCS Recovery Calculations

GGA Added (mL):	**6**		**LCS True Value (mg/L):**	**198**
LCS ID	**Calc mg/L**	**% Recovery**	**Recovery Acceptance Limits (%): 84.6 TO 115.4**	
LCS 1	187.020833	94.5	LCS Within Criteria ?	Yes
LCS 2	199.020833	100.5	LCS Within Criteria ?	Yes
LCS 3	168.02	84.9	LCS Within Criteria ?	Yes
Mean	184.6875	93.3	LCS Within Criteria ?	Yes

Matrix Spike Recovery Calculations (75-125%)
Spike Recovery=(Seed Corrected DO MS-Seed Corrected DO Sample)/(Total mg Spike)

Katahdin Sample Number	Sample Volume (mL)	Sample BOD (mg/L)	Spk. Sample BOD (mg/L)	mL GGA Spike Added	MS True Value (mg/L)	Spike Recovery (%)
SM0217-5	25.00	2.06	48.73	6	47.52	98.2
					#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!

All Data From This Batch Must Be Qualified As Follows:
LCS 1
LCS 2
LCS 3
Blank 1
Blank 2

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 6

BOD DATABASE BATCH SHEET

WET CHEMISTRY BATCH REPORT
Jan 15 2019, 02:17 pm
Batch: WG244240

Parameter: Total Biochemical Oxygen Demand

Prep Date: 09-JAN-19

Date Analyzed: 14-JAN-19

Prep Method: SM 5210B

Analyst Initials: RO/AP

Prep Chemist: ZF

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SM0192-1	SAMP	SM 5210B	0.58000mL	300.00mL	520	3.77	2000 mg/L	NA	2	120	1000		
SM0193-1	SAMP	SM 5210B	0.75000mL	300.00mL	400	3.38	1400 mg/L	NA	2	93.	800		
SM0198-1	SAMP	SM 5210B	0.28000mL	300.00mL	1100	3.89	4200 mg/L	NA	2	250	2100		
SM0215-5	SAMP	SM 5210B	200.00mL	300.00mL	1.5	3.06	4.6 mg/L	NA	2	0.35	3.0		
SM0216-5	SAMP	SM 5210B	75.000mL	300.00mL	4	2.62	10. mg/L	NA	2	0.93	8.0		
SM0217-5	SAMP	SM 5210B	300.00mL	300.00mL	1	2.06	2.1 mg/L	NA	2	0.23	2.0		
SM0218-5	SAMP	SM 5210B	200.00mL	300.00mL	1.5	4.48	6.7 mg/L	NA	2	0.35	3.0		
SM0243-5	SAMP	SM 5210B	3.5000mL	300.00mL	86	4.28	370 mg/L	NA	2	20.	170		
SM0245-1	SAMP	SM 5210B	300.00mL	300.00mL	1	2.46	2.5 mg/L	NA	2	0.23	2.0		
SM0247-1	SAMP	SM 5210B	0.38000mL	300.00mL	790	2.82	2200 mg/L	NA	2	180	1600		
WG244240-1	MBLANK	SM 5210B	300.00mL	300.00mL	1	.08	U1.0 mg/L	NA	2	0.23	2.0		
WG244240-2	LCS	SM 5210B	6.0000mL	300.00mL	50	3.694	180 mg/L	NA	2	12.	100		93
WG244240-3	DUP	SM 5210B	100.00mL	300.00mL	3	.73	U6.0 mg/L	NA	2	0.70	6.0	6	
WG244240-4	MS	SM 5210B	25.000mL	300.00mL	12	4.06	49. mg/L	NA	2	2.8	24.		98

Comments:

WG244240-1	SM0217-5
WG244240-2	SM0217-5
WG244240-3	SM0217-5
WG244240-4	SM0217-5

Entered by: ZF/AP Date: 1/15/19 Accepted by: SC Date: 1-15-19

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 7

INORGANIC DATA QUALIFIERS

KATAHDIN ANALYTICAL SERVICES – INORGANIC DATA QUALIFIERS

The sampled date indicated on the attached Report(s) of Analysis (ROA) is the date for which a grab sample was collected or the date for which a composite sample was completed. Beginning and start times for composite samples can be found on the Chain-of-Custody.

U	Indicates the compound was analyzed for but not detected above the specified level. This level may be the Practical Quantitation Level (PQL) (also called Limit of Quantitation (LOQ)), the Limit of Detection (LOD) or Method Detection Limit (MDL) as required by the client. Note: All results reported as "U" MDL have a 50% rate for false negatives compared to those results reported as "U" PQL "U" LOQ or "U" LOD, where the rate of false negatives is <1%.
E	Estimated value. This flag identifies compounds whose concentrations exceed the upper level of the calibration range of the instrument for that specific analysis.
J	Estimated value. The analyte was detected in the sample at a concentration less than the laboratory Practical Quantitation Level (PQL) (also called Limit of Quantitation (LOQ)), but above the Method Detection Limit (MDL).
I-7	The laboratory's Practical Quantitation Level (PQL) or LOQ could not be achieved for this parameter due to sample composition, matrix effects, sample volume, or quantity used for analysis.
A-4	Please refer to cover letter or narrative for further information.
H_	Please note that the regulatory holding time for _____ is "analyze immediately". Ideally, this analysis must be performed in the field at the time of sample collection. _____ for this sample was not performed at the time of sample collection. The analysis was performed as soon as possible after receipt by the laboratory. H1 - pH H2 - DO H3 - sulfite H4 - residual chlorine
T1	The client did not provide the full volume of at least one liter for analysis of TSS. Therefore, the PQL of 2.5 mg/L could not be achieved.
T2	The client provided the required volume of at least one liter for analysis of TSS, but the laboratory could not filter the full one liter volume due to the sample matrix. Therefore, the PQL of 2.5 mg/L could not be achieved.
M1	The matrix spike and/or matrix spike duplicate recovery performed on this sample was outside of the laboratory acceptance criteria. Sample matrix is suspected. The laboratory criteria was met for the Laboratory Control Sample (LCS) analyzed concurrently with this sample.
M2	The matrix spike and/or matrix spike duplicate recovery was outside of the laboratory acceptance criteria. The native sample concentration is greater than four times the spike added concentration so the spike added could not be distinguished from the native sample concentration.
R1	The relative percent difference (RPD) between the duplicate analyses performed on this sample was outside of the laboratory acceptance criteria (when both values are greater than ten times the PQL).

MCL	Maximum Contaminant Level	NL	No limit
NFL	No Free Liquid Present	FLP	Free Liquid Present
NOD	No Odor Detected	TON	Threshold Odor Number

D-1	As required by Method 5210B, APHA Standard Methods for the Examination of Water and Wastewater (21st edition), the BOD value reported for this sample is 'qualified' because the check standard run concurrently with the sample analysis did not meet the criteria specified in the method (198 +/- 30.5 mg/L). These results may not be reportable for compliance purposes.
D-2	The measured final dissolved oxygen concentrations of all dilutions were less than the method-specified limit of 1 mg/L. The reported BOD result was calculated assuming a final oxygen concentration equal to 1 mg/L. The reported value should be considered a minimum value.
D-3	The dilution water used to prepare this sample did not meet the method and/or regulatory criteria of less than 0.2 or 0.4 mg/L dissolved oxygen (DO) uptake over the five day period of incubation. These results may not be reportable for compliance purposes.

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TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

FIGURE 8

DISSOLVED OXYGEN SOLUBILITY TABLES

Solubility of oxygen in fresh water at various temperatures and pressures
[Solubility shown in milligrams per liter. Values based on published equations by Benson and Krause (1980 and 1984).
deg C, degrees Celsius; mm Hg, millimeters of mercury]

Results from DOTABLES program at
https://water.usgs.gov/software/DOTABLES/.

Temp.	Barometric Pressure (mm Hg)					
(deg C)	750	760	770	780	790	800
15.00	9.95	10.08	10.22	10.35	10.49	10.62
15.20	9.91	10.04	10.17	10.31	10.44	10.58
15.40	9.86	10.00	10.13	10.26	10.40	10.53
15.60	9.82	9.95	10.09	10.22	10.35	10.49
15.80	9.78	9.91	10.05	10.18	10.31	10.44
16.00	9.74	9.87	10.00	10.13	10.27	10.40
16.20	9.70	9.83	9.96	10.09	10.22	10.36
16.40	9.66	9.79	9.92	10.05	10.18	10.31
16.60	9.62	9.75	9.88	10.01	10.14	10.27
16.80	9.58	9.71	9.84	9.97	10.10	10.23
17.00	9.54	9.66	9.79	9.92	10.05	10.18
17.20	9.50	9.62	9.75	9.88	10.01	10.14
17.40	9.46	9.58	9.71	9.84	9.97	10.10
17.60	9.42	9.55	9.67	9.80	9.93	10.06
17.80	9.38	9.51	9.63	9.76	9.89	10.02
18.00	9.34	9.47	9.59	9.72	9.85	9.98
18.20	9.30	9.43	9.55	9.68	9.81	9.93
18.40	9.26	9.39	9.52	9.64	9.77	9.89
18.60	9.23	9.35	9.48	9.60	9.73	9.85
18.80	9.19	9.31	9.44	9.56	9.69	9.81
19.00	9.15	9.28	9.40	9.53	9.65	9.77
19.20	9.11	9.24	9.36	9.49	9.61	9.74
19.40	9.08	9.20	9.33	9.45	9.57	9.70
19.60	9.04	9.17	9.29	9.41	9.54	9.66
19.80	9.01	9.13	9.25	9.37	9.50	9.62
20.00	8.97	9.09	9.21	9.34	9.46	9.58
20.20	8.93	9.06	9.18	9.30	9.42	9.54
20.40	8.90	9.02	9.14	9.26	9.39	9.51
20.60	8.86	8.99	9.11	9.23	9.35	9.47
20.80	8.83	8.95	9.07	9.19	9.31	9.43
21.00	8.79	8.92	9.04	9.16	9.28	9.40
21.20	8.76	8.88	9.00	9.12	9.24	9.36
21.40	8.73	8.85	8.97	9.08	9.20	9.32
21.60	8.69	8.81	8.93	9.05	9.17	9.29
21.80	8.66	8.78	8.90	9.01	9.13	9.25
22.00	8.63	8.74	8.86	8.98	9.10	9.22
22.20	8.59	8.71	8.83	8.95	9.06	9.18
22.40	8.56	8.68	8.79	8.91	9.03	9.15
22.60	8.53	8.64	8.76	8.88	8.99	9.11
22.80	8.49	8.61	8.73	8.84	8.96	9.08
23.00	8.46	8.58	8.69	8.81	8.93	9.04
23.20	8.43	8.55	8.66	8.78	8.89	9.01
23.40	8.40	8.51	8.63	8.74	8.86	8.97
23.60	8.37	8.48	8.60	8.71	8.83	8.94
23.80	8.34	8.45	8.56	8.68	8.79	8.91
24.00	8.30	8.42	8.53	8.65	8.76	8.87
24.20	8.27	8.39	8.50	8.61	8.73	8.84
24.40	8.24	8.36	8.47	8.58	8.70	8.81
24.60	8.21	8.32	8.44	8.55	8.66	8.78
24.80	8.18	8.29	8.41	8.52	8.63	8.74

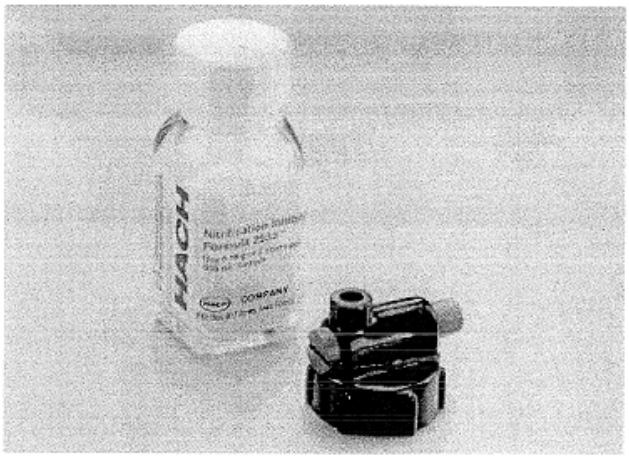
TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT A

HACH NITRIFICATION INHIBITOR SPECIFICATIONS

Formula 2533
Nitrification Inhibitor

BOD determinations may be inadequate for evaluating efficiency of wastewater treatment processes if nitrifying bacteria are present. These bacteria, in the presence of ammonia and dissolved oxygen, are able to convert ammonia to nitrate (nitrification). Because the BOD test measures oxygen demand, nitrification may be included as nitrogenous oxygen demand. When this interference exists, test results may overestimate the BOD of secondary effluents, resulting in an under-estimation of the actual plant removal efficiency. (The bacteria responsible for nitrification usually are not present in influent or primary effluent.)



Nitrification Inhibitor simplifies carbonaceous BOD testing.

Hach's Formula 2533™ Nitrification Inhibitor should be added to eliminate this interference when testing samples such as biologically treated effluents (effluents from secondary treatment processes), samples seeded with biologically treated effluents, and river waters.** Results of BOD tests completed without the use of Nitrification Inhibitor commonly are referred to as total BOD, while those tests with inhibitor are referred to as carbonaceous BOD (CBOD). Nitrification Inhibitor can be used with either the USEPA-accepted BOD dilution method (method 8043) or with Hach's BODTrak Apparatus.

Determine CBOD in a sample by adding Formula 2533 to each BOD bottle prior to incubation. All other steps in the BOD test procedure remain the same. Simultaneous tests for total BOD (no Nitrification Inhibitor added) and CBOD will provide information about oxygen demand due to nitrification. Subtracting the CBOD from total BOD yields the nitrogenous BOD.

** *Standard Methods for the Examination of Water and Wastewater*, 18th edition. Hach offers bulk TCMP (Cat. No. 2579-24), which is USEPA accepted. However, for ease of use, we recommend using Formula 2533 Nitrification Inhibitor (2.2% TCMP), which dissolves quickly and completely in samples. Formula 2533 Nitrification Inhibitor is cited in the 18th edition of *Standard Methods for the Examination of Water and Wastewater* 5210B 3.G.

Easy handling, quick dissolving

Formula 2533 Nitrification Inhibitor, 2-chloro-6-(trichloromethyl) pyridine (TCMP), is plated on an inert salt for easy handling. Formula 2533 also dissolves quickly in samples. (Pure TCMP dissolves slowly and can float on the top of the sample.**) The use of Formula 2533 Nitrification Inhibitor should be noted when reporting test results.

Formula 2533 is supplied in quantities of 35 or 500 grams. Add approximately 0.16 grams of inhibitor to each empty BOD bottle, or add sufficient amounts to the dilution water to make a final concentration of 10 mg/L TCMP. An optional dispenser cap makes measurement of the inhibitor easy.

Install the dispenser cap on the 35-gram reagent bottle. Using the dispenser cap, add two "shots" (0.08 grams/shot) of inhibitor to each BOD bottle. If 60-mL BOD bottles are used (in the dilution method), add approximately 0.03 grams to each bottle.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT B

POLYSEED® SPECIFICATIONS

InterLab® POLYSEED®

Visit us at www.polyseed.com for your complete e-Video Guide

PolySeed® Application Procedure
BOD_5 Seed Inoculum

PolySeed® is a blend of broad spectrum bacteria designed specifically as a seed inoculum for the Biochemical Oxygen Demand (BOD_5) test as conducted in accordance to ***Standard Methods of the Examination of Water and Wastewater***. **PolySeed®** is an EPA approved BOD_5 seed inoculum that has been used to seed both municipal and industrial wastes for almost 20 years.

Overview: The following are the most important parts of the BOD_5 test. **First,** the BOD water must be made properly and stored at 20° C. **Next,** the PolySeed® solution must be properly rehydrated and tested to determine its' effect on the test (i.e., the Seed Control Factor – SCF). **Finally,** the seed inoculum must be tested against a known Glucose-Glutamic Acid (GGA) standard. With these tests in order, a very reliable and accepted BOD_5 test can be performed.

1st Step: Control – Dilution Water ("BOD Water"): Prepare the dilution water (some call it "BOD Water" or "Blank Water") in accordance with *Standard Methods*. Be sure to use fresh deionized water and remineralize with the appropriate nutrients and chemicals. Store the Control-Dilution water at 20° C until ready to use. Run a control ***"Blank"*** on the neat Control-Dilution water at 20° C along with the actual BOD test. To insure an acceptable final test the ***"Blank"*** must have an oxygen depletion of less than 0.2 mg/liter over the 5-day period. If you have any questions, refer to *Standard Methods,* InterLab®'s e-Guide Videos or our Frequently Asked Questions ("FAQ") page available at www.polyseed.com.

2nd Step: Seed Solution (i.e. "PolySeed® Solution"): To make the seed solution, place the entire contents of one PolySeed® capsule (discard the gelatin capsule) into 500mls of "DILUTION WATER" prepared in accordance to *Standard Methods* **(do not use DI water by itself)**. Normal dilutions are one (1) PolySeed® capsule to 500mls of BOD water; however, the concentration of seed can be adjusted to compensate for variations in BOD water and established internal laboratory testing protocol. This seeded dilution water will be referred to as the "PolySeed® solution". ***Note: Bran, which acts as the carrier for the microorganisms, will neither dissolve nor inhibit microbial activity, but must be settled out of the PolySeed® solution prior to use.***

Next, aerate and stir the PolySeed® solution for one (1) hour. Finally, decant the supernatant carefully so as not to allow any bran in the biological solution. Pour the decanted PolySeed® solution in a clean 500 ml beaker with a sterile stir bar, place on magnetic stirrer and gently stir for the remainder of the test. (Note: Our lab uses a Nalgene separatory funnel for this purpose) For best results, the PolySeed® solution should be used within six (6) hours of rehydration of the capsule.

01/2007

InterLab® POLYSEED®

3rd Step: Seed Control Factor ("SCF"): After following Step 2, carefully draw an aliquot from the PolySeed® solution. It is best to prepare the seed control using 15, 20, 25, & 30mls of PolySeed® solution; however, these aliquots may vary depending upon laboratory procedures. The resulting SCF should fall between 0.60 and 1.0 (see calculations below).

At the end of the 5-day test period calculate the **SEED CONTROL FACTOR ("SFC")** of the PolySeed® solution per *Standard Methods* by using [(B1 - B2) x f] where:

B1 = DO of seed control before incubation, mg/L
B2 = DO of seed control after incubation, mg/L and,
f = (Volume of seed in diluted sample)/(volume of seed in seed control)

Note: This can be automatically calculated using InterLab's BOD calculator.

4th Step: Glucose-Glutamic Acid Standard: After the glucose-glutamic acid (GGA) standard solution is prepared (refer to *Standard Methods* or our FAQ page at www.polyseed.com), use 4mls of PolySeed® solution for each BOD_5 bottle. Again, make sure there is no undissolved bran in the pipette. No other seed is required. (Note: PolySeed® solution volume can be adjusted to compensate for variations in DI water, laboratory procedures and established internal laboratory testing protocol.)

5th Step: BOD Sample Analysis: Prepare the live BOD samples in accordance with *Standard Methods*. Insure that the PolySeed® Solution is prepared and stirred in accordance with Step 2 above. Add 4mls of PolySeed® solution (this volume can be adjusted for varying BOD water) to each BOD_5 bottle when preparing the wastewater samples. No other seed is required. Follow *Standard Methods* procedures for incubation, seed correction, GGA, and dilution water preparation. When reporting results using PolySeed® it is best to use the BOD calculator located at www.polyseed.com or hand calculations in accordance with *Standard Methods*.

International Laboratory Supply, Ltd. (InterLab®)
4200 Research Forest Drive, Suite 150
The Woodlands, TX 77381
281-298-9410
www.polyseed.com

01/2007

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT C

BOD TROUBLESHOOTING GUIDE

Packaging

1. Did you receive a desiccant pillow with your product?
Each bottle of PolySeed should contain one desiccant pillow to keep the PolySeed fresh.

2. Did you receive a copy of an MSDS and the Application Procedure?
Each PolySeed purchase should come with one MSDS and one set of Application Procedures.

Storage

1. What is the expiration date written on the PolySeed bottle?
PolySeed has a shelf life of 1 year. If your PolySeed has passed it's expiration date, order a fresh bottle.

2. Under what conditions is your PolySeed stored ?
Temperature range: PolySeed should be stored between 65- 80oF (18-25oC). If PolySeed has been stored outside of this range, microbial activity may be diminished. Humidity: PolySeed should be stored in a dry area. If possible, it helps to store your PolySeed in a dessicator. If PolySeed is stored near a moisture source, microbial activity may be diminished. If the product inside the PolySeed capsule is difficult to pour into the dilution water, your PolySeed may have been stored near too much humidity. Location: PolySeed should be stored away from any source of heat. This includes ovens, autoclaves, furnaces and direct sunlight.

3. Has the PolySeed bottle been closed at all times?
When storing PolySeed, make sure that the bottle is always closed tightly. If the bottle has not been kept closed, moisture may affect microbial activity.

Application Procedures

The Application Procedure for PolySeed is based on the BOD method in "Standard Methods for the Examination of Water and Wastewater" an APHA-AWWA-WEF publication.

1. When preparing your PolySeed solution, was the capsule opened in order to add the product?
PolySeed capsules are not soluble. To use PolySeed, you must open the capsule and pour the contents into dilution water (500 ml is recommended). For good activity, ensure that all of the capsule's contents are added to the dilution water.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT C, CONT.

BOD TROUBLESHOOTING GUIDE

2. When preparing your PolySeed solution, did you add the capsule contents to dilution water?
It is a common mistake to add PolySeed to deionized water instead of dilution water. If this mistake is made, start over with a fresh PolySeed capsule using dilution water - the chemicals in dilution water act as a buffer for the microbes in PolySeed, which will create ionic balance.

3. Did you make your PolySeed solution fresh on the day of the test?
PolySeed must be prepared fresh on the day of the test. The solution must be used within six hours of hydration.

4. How long did you stir and aerate your PolySeed solution prior to use?
The PolySeed solution should be stirred and aerated for a minimum of 1 hour prior to use. Aeration and stirring should continue until all the tests are set-up.

5. When stirring and aerating your PolySeed solution, was your aeration stone(s) clean?
Ensure that your aeration stone (or whatever you are using to aerate the solution) is as clean as possible.

Dilution Water

1. Did you prepare your dilution water on the day that the test was performed?
For best results, it is advised that the dilution water be prepared fresh on the day of the test.

2. Was the dilution water aerated prior to use?
Dilution water should be aerated prior to use until it is saturated with dissolved oxygen.

3. When stirring and aerating your dilution water, was your aeration stone (s) clean?
Ensure that your aeration stone (or whatever you are using to aerate the solution) is as clean as possible.

4. When preparing your dilution water, were the following reagents used prior to their expiration dates? Were your stock solutions prepared in accordance with the Standard Methods?
Reagents:
Phosphate Buffer - Verify pH = 7.2.
Magnesium sulfate heptahydrate
Calcium chloride
Ferric chloride hexahydrate
Each of these reagents must be used before their expiration dates and your stock solutions should be prepared in accordance with the Standard Methods.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT C, CONT.

BOD TROUBLESHOOTING GUIDE

5. When preparing your dilution water, was 1 ml of each stock solution added to 1 liter of deionized water (distilled water)?
To prepare your dilution water, add 1 ml of each stock solution to 1 liter of deionized or distilled water.

6. When preparing your dilution water, did you ensure that the stock solutions were not contaminated?
The stock solutions should be stored in a refrigerator (4-7¼ C) in the dark when not in use. There also should be no evidence of biological contamination in these solutions. Evidence of biological contamination is provided by increased turbidity and also greening at the sides and base of the storage container.

7. Was deionized, distilled or tap water used in the preparation of the dilution water and stock solutions?
When preparing dilution water, it is necessary to use good quality deionized or distilled water. Tap water is not suitable for the preparation of dilution water.

8. Did you check the pH of the dilution water prior to use?
The pH of the dilution water should be between pH 7.0 - pH 7.2.

9. If adjustment of the dilution water was necessary, did you use acid or base?
It is recommended to use: 1N NaOH (base) or 1N H2SO4 (acid).

10. Did you conduct a quality check on the dilution water?
The dilution water should have a DO uptake not greater than 0.2 mg/l and preferably not greater than 0.1 mg/l.

If DO uptake exceeds 0.2 mg/l, there needs to be improved purification of the deionized or distilled water supply or use an alternative water source.

11. Was the temperature of the dilution water adjusted to 20oC prior to use?
It is important that the temperature of the dilution water is adjusted to 20oC prior to use. In fact, it is good practice to maintain the dilution water at this temperature from the moment it is made.

12. How long in advance of use was the dilution water prepared?
For optimal results, the dilution water should not be prepared more than 8 hours in advance of use.

13. Did you use a ready-to-use reagent for your dilution water?
Sometimes the reagents for the dilution water are purchased as ready-to-use solutions or powders. If you have used a ready-to-use reagent: - Confirm that the correct reagents were used. - Confirm that the reagents have not exceeded their expiration dates. - Confirm that the reagents have been stored and used in accordance with the manufacturer's directions.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT C, CONT.

BOD TROUBLESHOOTING GUIDE

Glucose-Glutamic Acid

1. What grade of Glucose and Glutamic acid was used?
It is recommended to only use reagent grade Glucose and Glutamic acid.

2. What form of Glutamic acid was used, D-Glutamic acid or L-Glutamic Acid?
It is recommended to use L-Glutamic acid.

3. Have the Glucose and Glutamic acid reagents exceeded their expiration dates?
Ensure that the Glucose and Glutamic acid reagents have not exceeded their expiration dates prior to use.

4. Were the Glucose and Glutamic acid reagents dried prior to use ?
Glucose and Glutamic acid should be dried for 1 hour at 103oC, on the day of test.

5. Following drying, were the reagents placed in a desiccator/vacuum jar?
After drying, the Glucose and Glutamic acid reagents should immediately be placed in a desiccator or vacuum jar to ensure that they do not gain moisture. Once cool, the reagents are ready for use.

6. Is your desiccator or vacuum jar functioning correctly?
Ensure that the desiccant is active.

7. Was the Glucose-Glutamic acid stock solution and 2% working solution prepared in deionized water (or distilled water) or was it prepared in dilution water?
The Glucose-Glutamic acid stock solution and 2% working solutions should only be prepared in dilution water.

8. How much Glucose and Glutamic acid did you use to make the stock solution?
150 mg Glucose and 150 mg Glutamic acid should be used to make up a 1-liter solution.

9. Was a 2% working solution of Glucose-Glutamic acid prepared from the above stock solution?
A 2% working solution of Glucose-Glutamic acid should be prepared from the stock solution.

10. Were the Glucose-Glutamic acid stock solution and 2% working solution prepared fresh on the day of use?
These solutions must be prepared fresh on the day of use.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT C, CONT.

BOD TROUBLESHOOTING GUIDE

BOD bottle containing the Glucose-Glutamic acid 2% working solution.

12. How many Glucose-Glutamic acid tests were set-up?
It is recommended that at least three Glucose-Glutamic acid tests be set up.

13. Were the Glucose-Glutamic acid seeded preparations read immediately or were they left at room temperature for a period of time?
It is important that the Glucose-Glutamic acid seeded preparations are read within 2 hours. In the event that the preparations are not read immediately, they should be placed at 20oC until the time of reading.

Seed Controls

1. What volumes of PolySeed solution were used when setting up the seed controls?
The recommended volumes to prepare for your seed controls are 10, 15, 20 and 25 mls (in triplicate).

2. Did the largest volume of PolySeed solution (25 ml) deplete the DO by at least 50%?
It is important that the largest volume of PolySeed solution (25 ml) deplete the DO by at least 50%. This indicated good microbial activity in your PolySeed solution.

Other Possible Problems

1. Confirm that:
- Your BOD test was conducted over 5 days - Your bottles were stored at 20oC throughout the 5-day incubation period - Your bottles were stored in the dark throughout their 5-day incubation period

2. Were any bubbles present in any of the preparations initially or after 5 days?
No bubbles should be present in any of the preparations at any time.

3. Was a water tight seal still present on each bottle after 5 days of incubation?
It is necessary for a tight seal to be present on each bottle after 5 days of incubation. To achieve a watertight seal, BOD bottle should be filled to the lip during the preparation stages. Stopper should be put in place gently, taking care not to spill. Lip and stopper should then be sealed with Parafilm or equivalent to minimize water evaporation during incubation.

TITLE: **BIOCHEMICAL OXYGEN DEMAND IN AQUEOUS SAMPLE MATRICES**

ATTACHMENT C, CONT.

BOD TROUBLESHOOTING GUIDE

4. Was all glassware, pipette tips, aeration stones, etc. clean prior to use?
All glassware, pipette tips, aeration stones, etc. should be well cleaned prior to use. Glassware should have a final rinse in deionized or distilled water.

5. Are all pieces of equipment such as pipettes, balances, incubators, ovens, thermometers, pH meters and waterbaths accurate?
All equipment used in the 5-day BOD test should be accurate and calibrated regularly.

6. Were the DO meter and electrode calibrated prior to use?
Always calibrate the DO meter and electrode prior to use. It is also a good practice to check calibration again at regular intervals during the test.

7. Was the electrode rested between readings?
Always rest the electrode between readings in accordance with manufacturer's instructions.

8. Was the electrode washed in deionized water between readings?
Always wash the electrode in deionized water between readings.

9. Were all tests performed in triplicate?
All tests e.g. dilution water checks, seed controls, wastewater dilutions and Glucose-Glutamic acid should be performed in triplicate.

10. What type of pipette tips was used for the testing?
It is recommended to use wide mouthed pipette tips so that the cereal component in PolySeed does not block the tip.

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Ryan Oliver

Review Date: 7/24/19

SOP Number: CA-701-~~09~~ 10 UAD 072519

SOP Title: Biochemical Oxygen Demand in Aqueous Sample Matrices

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:** 07/25/19

QAO Signature: Susan Dimond **Date:** 07.25.19

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-709
Revision History
Cover Page
Page 1

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tanguay Date: 02/13/01

Operations Manager: John C. Benton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2/13/01

General Manager: Dennis P. Hurley Date: 2/12/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 9045C	Format changes, added pollution prevention, database and operation of Accumet pH meter and calibration.	DH	2/13/01	2/13/01
04 9045C	Addition to Scope and Application to include reference for 9040B use when aqueous phase is 720%	DH	8/27/02	8/27/02
05 9045C	added KIMS Minor changes throughout added wording to sect. 6 New fig. 1 and 2	LAD	120104	120104
06 9045C	Added SW-846 reference. Minor formatting changes throughout.	LAD	03/07	03/07
07 9045D	Section 7.18 - Renamed "Equipment Maintenance" and revised for current practices. Add Wet Chem. Data Entry SOP reference. Updated references in Section 10. Updated log book example.	LAD	08/09	08/09

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 9045D	Added references to sections 7 and 10.	LAD	06/10	06/10
09 9045D	Sect. 7.- Updated calibration procedure. Changed buffer pH probe stored in. Updated archived of reports. Added and edited references. Updated Figures 1 & 2 added 3. Changed H.T. from ASAP to 28 days.	LAD	05/12	05/12
10 9045D	Sect. 6 - Updated holding time criteria. Sect. 7 - Updated calibration procedure to reflect current practice. Sect. 10 - Updated and added references. Updated fig. 1 → 3	LAD	07/14	07/14
11 9045D	Sect. 7. Added requirement to reprep the sample if no standing water is present.	LAD	08/16	08/16

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

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-709-11**, titled **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-709-11**, titled **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**.

Recipient: _____ Date: _____

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and techniques followed by Katahdin Analytical Services personnel to determine the pH of soils and waste samples in accordance with EPA method 9045 (current promulgated revision). Method 9045 is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample. If the aqueous phase is greater than 20%, pH determination should be performed in accordance with EPA method 9040 (current promulgated revision). Refer to the current revision of Katahdin SOP CA-708, pH Concentration Measurements in Aqueous Samples.

The procedures in this SOP are applicable to all non-CLP pH measurements performed for all soil matrices analyzed in the laboratory.

1.1 Definitions

pH - A measure of the acidity or alkalinity of a solution, defined as -log $[H^+]$.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pH in solids by EPA Method 9045. 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 pH concentration measurements in solid matrices 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 pH 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 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 and 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 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.

2.0 SUMMARY OF METHOD

A representative aliquot of sample, measured in grams, is mixed with an equivalent volume of laboratory reagent grade water, measured in mL. The solution is allowed to settle, and the pH of the standing water (decanted) is determined electrometrically.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated with an oily material. See section 7.18 for special cleaning instructions.

4.0 APPARATUS AND MATERIALS

4.1 pH meter, Accumet Model 20 or equivalent with Automatic Temperature Compensation (ATC)

4.2 Glass beakers, 25 mL and 400 mL

4.3 25 mL dose cups

4.4 Teflon coated stir-bars

4.5 Stir-bar retriever

4.6 Magnetic stirplate

4.7 Shaker, 12 place

4.8 Analytical balance, capable of weighing to 0.1 g

5.0 REAGENTS AND STANDARDS

5.1 Buffer solutions (pH 4.0, 6.0, 7.0, 8.0, 10.0, 12.0)

5.2 Laboratory reagent grade water (Lab Water)

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in soil jars and stored at 4°C until analysis. Samples are collected in plastic or glass jars and stored at 4°C until analysis.

pH samples require immediate analysis upon receipt by the laboratory.

SW846 Chapter 3 states the holding time for pH is “immediate”.

Katahdin project managers will remind clients that in order to meet the regulatory requirements for holding times, a field pH is required. If requested to perform a laboratory pH, the analysis must be performed as soon as possible and the data must be notated as being performed out of hold time.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

7.0 PROCEDURES

SAMPLE PREPARATION

7.1 Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling. Mix samples thoroughly. Discard any foreign objects such as sticks, leaves and rocks. Decant any standing liquid. Using the balance, weigh out 20.0 g of sample into a 400 mL glass beaker. Record weight in pH logbook (Figure 1).

7.2 Add 20 mL of laboratory reagent grade water to the sample. Cover the top of the beaker with parafilm.

7.3 Place the sample on the shaker and allow it to shake, at medium speed, for five minutes. (CLP methods require the sample to shake for one hour.)

7.4 After five minutes (or one hour), remove the sample from the shaker and allow it to settle for one hour.

7.5 After one hour, decant the standing liquid into a 25 mL beaker. If no standing liquid is present, reprep the sample using 20g of sample and 40 mL of laboratory reagent grade water, cover with parafilm, and repeat steps 7.3 and 7.4.

7.6 Record total volume of laboratory reagent grade water added to sample in pH logbook. If volume of laboratory reagent grade water (in mL) added to sample exceeds the initial gram weight of the sample, flag sample data in pH logbook and record the reason for addition of excess laboratory reagent grade water (eg. minimum volume of water required in order to cover pH probe).

NORMAL RANGE CALIBRATION (pH range 3.5 – 10.5)

7.7 Meter should be calibrated daily. As described in the following steps, conduct a three-point calibration with pH buffers 4, 7 and 10. Perform a calibration check using pH 7 buffer. The source/lot number of each solution at the time of analysis must be recorded in the logbook (Figure 1).

7.8 Rinse pH electrode and temperature probe with laboratory reagent grade water. Gently blot dry with kimwipe.

7.9 Place pH 4 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer. Push **Standardize** key. Then push *2,* to clear previous calibration. Push **Standardize** key, then push 1, to update. Enter value of pH buffer, once stabilized record the value in the pH calibration logbook (Figure 3).

7.10 Remove pH 4 buffer. Rinse pH electrode and temperature probe. Blot dry.

7.11 Repeat step 7.1.3 with the pH 7 buffer. Record the value in the pH calibration logbook (figure 3). Remove pH 7 buffer. Rinse and dry pH electrode and temperature probe.

7.12 Repeat step 7.1.3 with the pH 10 buffer. Record the value in the pH calibration logbook (figure 3). Remove pH 10 buffer. Rinse and dry pH electrode and temperature probe.

NOTE: If any buffer readings are not within 0.05 pH units of expected values prior to calibration, the electrode may need cleaning. Note any maintenance performed and rerun the calibration.

LOW RANGE CALIBRATION

7.13 For samples with a pH less than 3.5, the meter must also be calibrated with pH buffer 2.

7.14 Rinse pH electrode and temperature probe with laboratory reagent grade water. Gently blot dry with kimwipe.

7.15 Place pH 2 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer. Push **Standardize** key. Push **Standardize** key, then push 1, to update. Enter value of pH buffer, once stabilized record the value in the pH calibration logbook (Figure 3).

7.16 The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

HIGH RANGE CALIBRATION

7.17 For samples with a pH greater than 10.5, the instrument must also be calibrated using a ph buffer 12.

7.18 Rinse pH electrode and temperature probe with laboratory reagent grade water. Gently blot dry with kimwipe.

7.19 Place pH 12 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer. Push **Standardize** key. Push **Standardize** key, then push 1, to update. Enter value of pH buffer, once stabilized record the value in the pH calibration logbook (Figure 3).

7.20 The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

CALIBRATION CHECK / LABORATORY CONTROL SPIKE (LCS)

7.21 Place pH 7 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer., but DO NOT press any keys as this reading is a calibration check. Record the reading in pH logbook as the LCS. Results must be within 0.05 pH units of the true value for analysis to proceed.

ANALYSIS OF SAMPLES

7.22 Sample analysis may proceed once the meter has been calibrated for the day with three buffers that bracket the expected pH of the sample.

7.23 Run the pH 7 buffer as the LCS for the analytical batch (Section 7.21). An LCS is required at the beginning of every batch of twenty or fewer samples.

7.24 Record date, time and initials for this analytical session.

7.25 The decanted samples should be equilibrated to room temperature prior to analysis (i.e., at the same temperature as the calibration buffers, □2 □C). A more accurate pH reading will be achieved when the buffers and the samples are at the same temperature. However, the Accumet□ pH meter is equipped with automatic temperature compensation (ATC) for when samples and buffers are not at the same temperature. Refer to the Accumet□ Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C) for information on the ATC probe.

7.26 Pour about 25 ml of the supernatant into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.

7.27 When meter locks, record value displayed.

7.28 Rinse pH electrode and temperature probe. Blot dry

7.29 Place probe in pH 7 buffer solution to store until next analysis.

EQUIPMENT MAINTENANCE

7.30 If an electrode becomes coated with an oily material that will not rinse free, the electrode can either (refer to instrument manual):

- be cleaned with an ultrasonic bath, or

- be washed with detergent, rinsed several times with laboratory reagent grade water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with laboratory reagent grade water.

An electrode that will not calibrate properly must be replaced.

REPORTING OF RESULTS

7.31 All pH measurements less than 10.0 are to be reported using two significant figures.

Examples: 2.46 = 2.5
6.32 = 6.3
9.94 = 9.9

7.32 All pH measurements which are at or greater than or round up to 10.0 are to be reported to three significant figures.

Examples: 9.95 = 10.0
12.25 = 12.3
13.76 = 13.8
11.95 = 12.0

7.33 When a sample duplicate is analyzed, both the original result and duplicate result are recorded in the pH logbook; however, the original sample result is to be reported to the client.

7.34 After completion of each test, the logbook must be signed and dated by the person performing the test. All unused lines are to be “z-ed” out and initialed and dated.

7.35 The sample data results, with any appropriate notations, are entered into KIMS by the analyst. Refer to the current revision of SOP CA-762 for instructions on data entry. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.36 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.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

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 judgements. These decisions are based on holding time considerations, remaining sample volume 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 One sample duplicate is to be analyzed per batch or every 10 sample analyses.

8.1.1 Acceptance criteria for duplicates is a difference of less than or equal to 20% relative percent difference between sample and duplicate results.

8.1.2. If criterion is not met, check calibration and reanalyze sample in duplicate.

8.2 One Laboratory Control Sample (LCS) is to be analyzed per batch or every 20 samples.

9.0 METHOD PERFORMANCE

Refer to method 9045.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, and IV, February 2007, Method 9045D.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

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), current revision.

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

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.

LIST OF TABLES AND FIGURES

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

TABLE 1

QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW9045	PH (soil)	3-5 point calibration with pH buffers with a midrange cal. check	Once per day, prior to use	± 0.05 pH units for each buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration
		LCS	One per batch of twenty or fewer samples	90-110% recovery	Correct problem, recalibrate
		Sample duplicate	One sample duplicate per every ten field samples	RPD ≤20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-709-11	METHOD SW846 9045, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	1) Shake, at medium speed, for one hour. 2) Add more liquid after shaking and settling if there is no standing liquid left. 3) All buffers and samples are analyzed at room temperature. pH meter is equipped with automatic temperature compensation.	1) Continuously stir the suspension for five minutes. 2) No guidance for samples with no standing liquid left. 3) Report both pH and temperature at the time of analysis.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 1

EXAMPLE OF pH LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.

CORROSIVITY pH / pH Soil

Accumet 20 pH Meter - SN - C0024321 pH Probe SN - 2089100P

SW 846 9045D

CALIBRATION STDS:	CALIBRATED TO:	LOT NO:	NOTES:
pH 2.00	2.00	SWL 3617	WG147312
pH 4.00	4.00	3578	R284017
pH 7.00	7.00	3577	
pH 10.00	10.02	3579	
pH 12.00	11.98	3618	

LAB SAMPLE ID	ANALYSIS TIME	SAMPLE VOL (mL)	SAMPLE WEIGHT(g)	SAMPLE TEMP. (°C)	pH	REPORTED pH
LCS	15:11	—	—	22.3°C	7.00	7.0
SH5400-1	15:14	100	21.01	22.0°C	7.45	7.5
-1 Dup	15:18	"	21.91	21.9°C	7.67	7.8
SH5481-1	15:20	50	21.66	22.0°C	9.64	9.6
SH5494-1	15:22	X 100	20.65	22.0°C	12.77	>12
SH5497-1	15:23	"	19.16	22.0°C	6.71	6.7
SH5585-1	15:26	50	20.85	22.0°C	6.73	6.7
-2	15:27	"	20.56	22.2°C	7.13	7.1
-3	15:31	"	20.37	22.0°C	7.24	7.2
-4	15:33	"	20.35	22.1°C	6.50	6.5
-5	15:35	"	19.97	22.2°C	6.69	6.7
CCV	15:37	—	—	22.5°C	7.02	7.0
SH5585-6	15:40	50	21.82	22.4°C	10.21	10.2
-6 Dup	15:46	"	20.98	22.6°C	9.71	9.7
-7	15:48	"	20.20	22.3°C	7.90	7.9
-8	15:51	"	20.05	22.3°C	7.44	7.4
-9	15:52	"	20.44	22.4°C	7.33	7.3
CCV	15:53	—	—	22.5°C	7.03	7.0
Blank	15:55	—	—	20.9°C	5.47	5.5
			ZS 7.29.14			

PREP ANALYST: ZS DATE/TIME: 7·28·14 16:00
ANALYST: ZS DATE: 7.29.14
CHECKED BY: DATE:

WL-015 - Revision 2 - 04/30/2012 QAWL693 0000099

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 2

EXAMPLE OF BATCH SHEET FOR pH

WET CHEMISTRY BATCH REPORT
Jul 15 2014, 01:23 pm
Batch: WG146539

Parameter: pH(Soil)
Prep Date: 15-JUL-14

Date Analyzed: 15-JUL-14
Prep Method: SW846 9045C

Analyst Initials: ZS
Prep Chemist: ZS

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SB4998-1	SAMP	SW846 9045D	10.63g	30mL	1	8.4	8.4 pH	92.	.1	0.10	0.10		
SB4998-2	SAMP	SW846 9045D	11.06g	30mL	1	7.58	7.6 pH	91.	.1	0.10	0.10		
SB5046-1	SAMP	SW846 9045D	10.51g	50mL	1	5.3	5.3 pH	33.	.1	0.10	0.10		
SB5074-3	SAMP	SW846 9045D	12.17g	30mL	1	9	9.0 pH	83.	.1	0.10	0.10		
SB5074-4	SAMP	SW846 9045D	11g	30mL	1	8.94	8.9 pH	81.	.1	0.10	0.10		
SB5150-1	SAMP	SW846 9045D	25.64g	150mL	1	7.03	7.0 pH	14.	.1	0.10	0.10		
WG146539-1	LCS	SW846 9045D	20g	20mL	1	7	7.0 pH	NA	.1	0.10	0.10		100
WG146539-2	DUP	SW846 9045D	10.99g	20mL	1	9.08	9.1 pH	NA	.1	0.10	0.10	2	
WG146539-3	MBLANK	SW846 9045D	20g	20mL	1	5.69	5.7 pH	NA	.1	0.10	0.10		

Comments:

SB5074-3 NYC-NYCDEP-SED-SPLIT-21: MS/DUP on metals and cyanide. DUP on Black Carbon, TS, pH. MS/MSD on Ammonia, TKN, N+N and Total P. Can be moved to another sample if needed.
SB5074-4 NYC-NYCDEP-SED-SPLIT-22: MS/MSD on DOBS and SVOC,MS/DUP on TOC. Can be moved to another sample if needed.
WG146539-1 SB5074-4
WG146539-2 SB5074-4
WG146539-3 SB5074-4

Entered by: ZS Date: 7.15.14 Accepted by: Date: 7/15/14

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 3

EXAMPLE OF PH CALIBRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

pH METER CALIBRATION RECORD

FISHER ACCUMET 20 - SERIAL NUMBER C0024321

DATE	INITIALS	pH TRUE VALUES AND ACCEPTABILITY														
		2.00	± 0.05 (3)	Lot #	4.00	± 0.05 (3)	Lot #	7.00	± 0.05 (3)	Lot #	10.00	± 0.05 (3)	Lot #	12.00	± 0.05 (3)	Lot #
6-16-14	BN	2.00	✓	SWL 3617	3.96	✓	SWL 3578	7.00	✓	SWL 3577	10.00	✓	SWL 3579	11.95	✓	SWL 3618
6-17-14	BN	1.98	✓	"	3.96	✓	"	7.01	✓	"	9.99	✓	"	11.95	✓	"
6-18-14	ZS	2.00	✓	"	4.00	✓	"	7.00	✓	"	10.00	✓	"	12.00	✓	"
6/19/14	UNP				3.99	✓	"	6.98	✓	"	9.99	✓	"	12.00	✓	"
6/20/14	UNP				4.00	✓	"	6.98	✓	"	9.98	✓	"	12.03	✓	"
6/23/14	UNP	1.99	✓	"	3.98	✓	"	6.99	✓	"	9.99	✓	"	12.01	✓	"
06-24-14	[illegible]	2.00	✓	SWL 3617	4.00	✓	"	7.03	✓	"	10.03	✓	"	11.97	✓	"
6-25-14	DW			↓	4.00	✓	"	7.00	✓	"	10.00	✓	"			
06/26/14	[illegible]	2.01	✓	"	4.00	✓	"	7.00	✓	"	9.99	✓	"			
6-27-14	ZS	2.00	✓	"	3.96	✓	"	6.99	✓	"	9.99	✓	"	12.00	✓	SWL 3618
6/30/14	UNP	2.00	✓	"	3.97	✓	"	6.99	✓	"	9.99	✓	"	12.01	✓	"

MAINTENANCE – Include date, initials and task

QA-070 – Revision 1 – 09/30/2010

QAQC615

0000039

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: RO

Review Date: 1/29/18

SOP Number: CA-709-11

SOP Title: pH concentration measurement in soil matrices - SW846 method 9045

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: K. Brewer **Date:** 07/13/18

QAO Signature: Leslie Dimond **Date:** 07.13.18

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

Prepared By: Wet Chemistry Date: 02/01

Approved By:

Group Supervisor: J Burton for K Tanguay Date: 2/01

Operations Manager: Jol C. Burton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2·15·01

General Manager: Dennis T. Murphy Date: 2/15/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed procedure to reflect titrating 200mL- not entire volume. Added KIMS entry, added Sm reference (Title and Sect. 10), Added Sm QC limits	LAD	01/07	01/07
02	Removed necessity of verifying purchased, certified titrant. Added definitions.	LAD	01/09	01/09
03	Changed method reference to Sm 18th edition Updated Logbook and batch sheet examples. Changed title to correct Sm reference.	LAD	08/09	08/09
04	Updated and or added references to Sect.s 1.3 and 10.	LAD	06/10	06/10
05	Sect. 5 - Added information for purchased $Na_2S_2O_3$. Sect. 7 - removed standardization of $Na_2S_2O_3$ if purchased. Also, added raw data archived. Sect. 8 - Updated SM4500S^2 E LCS acceptance limits. Sect. 9 - Added MDL, LOD, LOQ information. Sect. 10 - added, edited references. Update figures 1 → 3.	LAD	05/12	05/12

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Changed SM4500-S^{2-} F to SM4500-S^{2-} F throughout (including the title). Sect. 10 – Added SM approval date to method reference.	LAD	02/13	02/13
07	Changed 250mL Erlenmeyer to 400mL Beaker. Changed volumetric pipet to calibrated adj. vol. pipet. Changed sodium sulfide prep instructions. Changed scrubber solution volume for reactive sulfide. Changed DUP. frequency (20 to 10). Updated Figs 1→3	LAD	10/15	10/15
08	Sect. 7 - Updated to reflect current practice and for clarity. Sect. 8 - Contingency plan added. Sect. 10 - References added, removed, and updated. Figures 1, 2, + 3 - Updated.	LAD	12/17	12/17

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

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-722-08**, titled **Titrimetric Determination of Sulfide Using EPA Method 376.1,SM4500-S^{2-} F, SW846 9034 And SW846 7.3.4.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-722-08**, titled **Titrimetric Determination of Sulfide Using EPA Method 376.1,SM4500-S^{2-} F, SW846 9034 And SW846 7.3.4.**

Recipient: _____ Date: _____

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure and requirements followed by Katahdin Analytical Services for the titrimetric determination of sulfide. This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, and domestic and industrial wastes in accordance with EPA Method 376.1 and SM4500-S^{2-} F. Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class). This method is suitable for the measurement of sulfide in concentrations above a practical quantitation level of 1 mg/L.

This procedure may also be used as a determinative step for acid-soluble and acid-insoluble sulfides following distillation of the sample by SW846 Method 9030 and Reactive Sulfide, Chapter 7.3.4.2 in accordance with the current revision of Katahdin SOP CA-734, "Reactive Sulfide: SW846 7.3.4.2". Method 9034 is suitable for measuring sulfide concentrations in samples that contain 0.2 mg/Kg to 50 mg/Kg.

1.1 Definitions

Laboratory Duplicate – DUP - A duplicate is a second aliquot of a sample that is analyzed the same way as the original sample in order to determine the precision of the method.

Laboratory Control Spike - LCS - A standard or solid reference material of known value that has been brought through the sample preparation an analysis process. The LCS is used to assess the accuracy of the method.

Method Blank – Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

Practical Quantitation Limit - PQL - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

Method Detection Limit - MDL - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the titrimetric analysis of sulfide by EPA Method 376.1, SM4500-S^{2-} F, SW846 9034 And SW846 7.3.4. 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.

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It is the responsibility of all Katahdin technical personnel involved in titrimetric analysis of sulfide by Methods 376.1, SM4500-S^{2-} F, SW846 9034 And SW846 7.3.4 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 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; not taking food or drink into the laboratory; and knowing 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.

Any residual basic waste generated from the preparation of reactive sulfide in this analysis is placed in satellite “G” or pyridine waste. The acidic waste after titration is put in satellite “A” or acid waste.

Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin 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.

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2.0 SUMMARY OF METHOD

Aqueous samples are treated with zinc acetate to precipitate the sulfide present as ZnS that aids in the removal of potential interferences and may also provide a means to concentrate the sample. The sulfide is resolubilized under acidic conditions, an excess addition of iodine is made to the sample. Under acidic conditions, the iodine oxidizes the hydrogen sulfide to sulfur. The excess iodine is back titrated with sodium thiosulfate using starch as an indicator. The difference between the total iodine added and the iodine consumed by the sodium thiosulfate represents the amount required for the reaction with hydrogen sulfide, thereby allowing the quantitative determination of sulfide in the sample.
In the case of waste samples designated for the determination of rate release of hydrogen sulfide and releasable hydrogen sulfide, the scrubber solution acidified but is not treated with zinc acetate prior to titration with iodine.

3.0 INTERFERENCES

3.1 Reduced sulfur compounds such as sulfite, thiosulfite and hydrosulfite, which decompose in acid, may yield erratic results.

3.2 Also, volatile iodine consuming substances including reducing substances and various organic compounds will give high results.

3.3 Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to a form that is not measurable by the application of this procedure.

4.0 APPARATUS AND MATERIALS

4.1. 25 and 10 mL burettes (Class A)

4.2. Stir plate

4.3. Burettes stand and holder

4.4. Stir bar and retriever

4.5. 400 mL beaker

4.6. Calibrated adjustable volume pipette, 5 mL and 10 mL

4.7. Disposable transfer pipette

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4.8. 250 mL graduated cylinder

4.9. Glass fiber filters: Gelman Type A/E, 47 mm or equivalent

4.10. 500-mL side arm filtering flask

4.11. 300-mL Gelman filtering chimney and filter holder or equivalent

4.12. Glass stir rods

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Grade Reagent Water: Sulfide-free water.

5.2 6N Hydrochloric Acid - CAUTION: In a fume hood add 500 mls of concentrated HCl to 500 ml laboratory reagent grade water, slowly mix and allow to cool.

5.3 Standard Iodine Solution 0.0250N: Dissolve 20 - 25 g KI in 1 L of Laboratory reagent grade water and add 3.2 g iodine. After iodine has dissolved, standardize against 0.0375N sodium thiosulfate using the starch solution as an indicator. (Section 7).

5.4 0.0375N Sodium Thiosulfate Titrant, ($Na_2S_2O_3$): Purchased or prepared from sodium thiosulfate pentahydrate salts, $Na_2S_2O_3 \cdot 5\ H_2O$, MW 248.19. Weigh 9.307 g $Na_2S_2O_3 \cdot 5\ H_2O$, quantitatively transfer to 1-L flask. Add 1.5 mL 6 N NaOH or 0.4g solid NaOH. Dissolve and bring to volume with freshly boiled Laboratory reagent grade water. The standard solution should be stored in the dark and titrated against 0.025N $KH(IO_3)_2$ when prepared by laboratory, or as a corrective action as detailed in Section 7. Note: 12 mole $Na_2S_2O_3$ is equivalent to 1 mole $KH(IO_3)_2$. Use certified value if purchased.

5.5 Standard 0.025N (0.0021M) Potassium Bi-iodate Solution: Purchased or prepare by dissolving 0.8124 mg $KH(IO_3)_2$ in Laboratory reagent grade water and dilute to 1000 mLs. This solution can also be purchased from a reputable supplier.

5.6 Potassium iodide, KI, granular certified ACS

5.7 Starch, 0.5%, preserved with salicylic acid: Either purchase prepared starch or prepare from reagent grade soluble starch as follows: Make a paste consisting of 0.5 g lab grade soluble starch, 0.2 g of salicylic acid, as a preservative, in approximately 100 mLs hot DI reagent water. Then bring up to volume in a 100 ml volumetric flask.

5.8 Sodium Sulfide (Na_2S) Standard: Dissolve 7.50 g reagent grade sodium sulfide nonahydrate ($Na_2S \cdot 9H_2O$; FW240.18) into 1000 mL Laboratory reagent grade water.

This is equivalent to an estimated value of 1001 mg/L S or 1064 mg/L H_2S. This must be standardized in accordance with the procedure described in Section 7 below. The sodium sulfide standard is stable for 6 months from the date of preparation. Store in an amber glass container in the refrigerator.

5.9 2 N Zinc Acetate Solution, $Zn(C_2H_3O_2)_2$: Dissolve 55.0 g of reagent grade zinc acetate in 250 mL Laboratory reagent grade water.

5.10 Sodium Hydroxide, 10 N: Purchased

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Samples must be taken with a minimum of aeration.

6.2 To analyze samples for total sulfide 4 drops of 2N zinc acetate / 100 mL sample with NaOH to a pH of >9 should be added to preserve the sample. The bottle should be filled to the top and capped with no headspace.

6.3 The holding time for aqueous samples is seven days from the date of collection.

6.4 When samples are suspected to contain reducing substances or in order to concentrate the sulfide pretreat the sample with zinc acetate with NaOH to a pH of >9 as described in Section 7.4.3.

6.5 Samples are maintained at 4^oC prior to analysis.

7.0 PROCEDURES

7.1 STANDARDIZATION OF SODIUM THIOSULFATE

7.1.1 If using purchased sodium thiosulfate standard solution, use the vendor-supplied certified value and skip standardization of sodium thiosulfate.

7.1.2 Repeat in triplicate. Base normality on the mean of the titrations unless an outlier is established by statistical rationale.

7.1.3 Dissolve ~2g KI in a 400 mL beaker with 100-150 mL reagent water. Add 1 mL 6 N H_2SO_4 or a few drops of conc. H_2SO_4.

7.1.4 Deliver 20.0 mL 0.025 N potassium biodate solution volumetrically. Dilute to 200 mL and titrate liberated iodine with Sodium Thiosulfate titrant.

7.1.5 When the solution is a pale straw (yellow) color. Add approximately 1 mL of starch indicator with a disposable transfer pipette. The color will turn blue.

7.1.6 Titrate with sodium thiosulfate until blue color disappears and the solution is clear and colorless. Take care not to overrun the end-point.

7.1.7 Record mLs titrant used. The normality of the $Na_2S_2O_3$ may be calculated by the following equation:

$$Normality\ Na_2SO_3 = \frac{mLs\ KH(IO_3)_2 \times Normality\ KH(IO_3)_2}{mLs\ Na_2SO_3\ Titrant\ used}$$

7.2 STANDARDIZATION OF IODINE

7.2.1 Repeat the titration in triplicate. Base normality on the mean of the titrations unless an outlier is established by statistical rationale.

7.2.2 Using a calibrated adjustable volume pipette, transfer 10.0 mL of the Standard Iodine Solution (5.3) followed by 2 mL 6N HCL into 200 mL of laboratory reagent grade water in a 400 mL beaker.

7.2.3 Place beaker on a stir plate; stir gently so as not to excessively aerate the sample. Titrate with 0.0375N sodium thiosulfate (5.4) from a 10- mL burette with the tip submerged. Titrate until a straw color (pale yellow) develops.

7.2.4 Add approximately ~1 mL of starch indicator with a disposable transfer pipette. The color will turn blue. Titrate with sodium thiosulfate until blue color disappears and the solution is clear and colorless.

7.2.5 Record to three decimal places (to the nearest 0.005 mL) the total volume of sodium thiosulfate used for each of the three replicates. Using the average of the of the mLs of sodium thiosulfate used in the triplicate determinations calculate the normality of the I_2 solution as follows:

$$Normality\ I_2 = \frac{mLs\ Na_2SO_3 \times Normality\ Na_2SO_3}{mLs\ I_2\ Titrated}$$

7.3 STANDARDIZATION OF SODIUM SULFIDE

7.3.1 To standardize the Sodium Sulfide Standard (5.8) repeat the titration in triplicate. Base normality on the mean of the titrations unless an outlier is established by statistical rationale. The analysis is accomplished by placing

an excess of iodine based upon the sulfide concentration in the flask and back titrating the excess iodine with sodium thiosulfate.

7.3.2 Using a calibrated adjustable volume pipette, transfer 10.0 mL of the Standard Iodine Solution (5.3) into 200 mL of laboratory reagent grade water in a 400 mL beaker.

7.3.3 Place beaker on a stir plate; stir gently so as not to excessively aerate the sample. Quantitatively Transfer 2.0 mL of standard sulfide solution (5.11) dispensing below the surface of the liquid in the beaker.

7.3.4 Add 2 mL of 6 N HCl and immediately begin titration with 0.0375 N sodium thiosulfate (5.4); following steps described in 7.2.3, and 7.2.4.

7.3.5 Add approximately ~1 mL of starch indicator with a disposable transfer pipet. The color will turn blue. Titrate with sodium thiosulfate until blue color disappears and the solution is clear and colorless.

7.3.6 Record to three decimal places (to the nearest 0.005 mL) the total volume of sodium thiosulfate used for each of the three replicates.

7.3.7 Using the average of the of the mLs of sodium thiosulfate used in the triplicate determinations calculate the concentration of sulfide as S^{2-} mg/L as follows:

$$mg\ S^{2-}/L = \frac{(A \times B) - (C \times D) \times 16{,}000}{mLs\ Na_2S}$$

Where: A = mLs iodine solution
B = normality of iodine solution
C = mLs $Na_2S_2O_3$ solution
D = normality of $Na_2S_2O_3$ solution, and
16,000 = mg equivalence S^{2-}, 32,066 mg / 2 equivalence

7.3.8 **For the determinations of Reactive Sulfide and Rate of Release of Sulfide ONLY**; convert the S^{2-} mg/L to H_2S multiply determined concentration of the sulfide solution times 1.06 where 1.06 = 34.08 g (MW H_2S)/32.07 g (MW S). The concentration as H_2S is entered into the spreadsheet for further calculations.

7.4 SAMPLE ANALYSIS

7.4.1 For samples other than **Reactive Sulfide** and **NOT** requiring pretreatment with zinc acetate

7.4.1.1 Transfer 200 mL aliquot of sample to a 400 mL beaker. Add 2 mL 6 N HCl. Using a calibrated adjustable pipette, transfer 10.0 mL of the Standard Iodine Solution (5.3) into the flask.

7.4.1.2 Note that the sample solution should be yellow which is indicative of excess iodine. If yellow color is not persistent (indicated by the addition of $\leq$ 1 mL titrant to reach a straw color) or evident, add more of the iodine solution in 10 mL increments until the yellow color returns.

7.4.1.3 Record all iodine additions appropriately in the logbook.

7.4.1.4 Place beaker on a stir plate; stir gently so as not to excessively aerate the sample. Titrate with 0.0375N sodium thiosulfate (5.4) from a 10- mL burette with the tip submerged. Titrate until a straw color (pale yellow) develops.

7.4.1.5 Add approximately ~1 mL of starch indicator with a disposable transfer pipette. The color will turn blue. Titrate with sodium thiosulfate until blue color disappears and the solution is clear and colorless.

7.4.1.6 Record to two decimal places to the nearest 0.02 mL the total volume of sodium thiosulfate used.

7.4.1.7 Calculation of mg S^{2-} /L is performed as follows:

$$mg\ S^{2-}/L = \frac{(A \times B) - (C \times D) \times 16{,}000}{mLs\ Sample\ Titrated}$$

Where: A = mLs iodine solution
B = normality of iodine solution
C = mLs $Na_2S_2O_3$ solution
D = normality of $Na_2S_2O_3$ solution, and
16,000 = mg equivalence S^{2-}, 32,066 mg / 2 equivalence

7.4.2 For scrubber solution for analysis of **Reactive Sulfide**

7.4.2.1 Transfer 190 mL aliquot of scrubber solution to a 400 mL beaker. Add 15 mL 6 N HCl. Using a calibrated adjustable volume pipet, transfer 10.0 mL of the Standard Iodine Solution (5.3) into the flask.

7.4.2.2 Note that the sample solution should be yellow which is indicative of excess iodine. If yellow color is not persistent (indicated by the addition of < 1 mL titrant to reach a straw color) or evident, add more of the iodine solution in 10 mL increments until the yellow color returns.

7.4.2.3 Record all iodine additions appropriately in the logbook.

7.4.2.4 Place beaker on a stir plate; stir gently so as not to excessively aerate the sample. Titrate sample with 0.0375N sodium thiosulfate (5.4) as described in 7.2.3 and 7.2.4.

7.4.2.5 Record to three decimal places (to the nearest 0.005 mL) the total volume of sodium thiosulfate used.

7.4.2.6 Calculate the concentration of mg S^{2-} /L in the scrubber as presented in 7.4.1.7. mg S^{2-} /L must be converted to mg H_2S/L prior to the final calculation of Rate of Release Sulfide or Reactive Sulfide as performed in accordance with the most current version of Katahdin SOP CA-734, "Reactive Sulfide: SW846 7.3.4.2", Section 7.5.3 and 7.5.4.

7.4.3 For samples requiring pretreatment with zinc acetate as follows:

7.4.3.1 For turbid samples or samples with particulate, shake sample gently and transfer 200 mL to a 400 mL beaker. For samples that do not contain solid matter transfer 200 mL to a 400 mL beaker with little agitation.

7.4.3.2 Adjust the pH of sample to 9 or greater with 10 N sodium hydroxide.

7.4.3.3 Add 4 drops of the 2 N zinc acetate solution/ 100 mL sample to the sample and mix well with glass stir rod. Sparingly rinse rod into flask.

7.4.3.4 In the presence of sulfide a white precipitate will form. Allow the sample to settle out for at least 30 minutes.

7.4.3.5 Filter sample through a pre-washed glass fiber filter. Add the filter precipitate and filter to the 400 mL beaker used in the precipitation step.

7.4.3.6 Add 200 mL laboratory reagent grade water. Let the filter rest undisturbed in the water for 5 minutes. The filter is retained in the

flask during the titration. Stir with a magnetic stirrer to resuspend the precipitate.

7.4.3.7 With the flask still on the stir plate, quantitatively add 10 ml of the iodine standard solution and then 2 ml of 6N HCL. Note that the sample solution should be yellow which is indicative of excess iodine. If yellow color is not persistent (indicated by the addition of $\leq$ 1 mL titrant to reach a straw color) or evident, add more of the iodine solution in 10 mL increments until the yellow color returns.

7.4.3.8 Titrate sample with 0.0375N sodium thiosulfate (5.4) as described in 7.2.3 and 7.2.4.

7.4.3.9 Record to *three* decimal places (to the nearest 0.005 mL) the total volume of sodium thiosulfate used.

7.4.3.10 Calculate the concentration of mg S^{2-} /L in the sample as presented in 7.4.1.7. **NOTE: The volume sample titrated is the volume that was treated with zinc acetate to precipitate the ZnS.**

7.4.4 For samples preserved with zinc acetate at the time of sampling

7.4.4.1 Shake bottle vigorously and filter 200 mL through pre-washed glass fiber filter. Record volume in the logbook. **NOTE: In the event that the sample presents difficulty upon filtration, the sample should be delivered sparingly to the chimney and filters should be replaced as needed. All filters will be required for the titration. As an alternative an unpreserved sample can be used with a smaller aliquot for precipitation and analysis if required or analysis directly.**

7.4.4.2 Transfer each filter to a 400 mL beaker containing 200 mL of laboratory reagent grade water and discard the filtrate in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

7.4.4.3 Place beaker on a stir plate; stir gently so as not to excessively aerate the sample and allow filter and precipitate to resuspend into solution.

7.4.4.4 Using a calibrated adjustable volume pipette, transfer 10.0 mL of Standard Iodine Solution (5.3) into the beaker containing the filters, followed by 2.0 mL of 6N HCl.

7.4.4.5 Note that the sample solution should be yellow which is indicative of excess iodine. If yellow color is not persistent (indicated by the addition of $\leq$ 1 mL titrant to reach a straw color) or evident, add more of the iodine solution in 10 mL increments until the yellow color returns.

7.4.4.6 Record all iodine additions appropriately in the logbook.

7.4.4.7 Place 400 mL beaker on a stir plate; stir gently so as not to excessively aerate the sample. Titrate sample with 0.0375N sodium thiosulfate (5.4) as described in 7.2.3 and 7.2.4.

7.4.4.8 Record to three decimal places (to the nearest 0.005 mL) the total volume of sodium thiosulfate used.

7.4.4.9 Calculate the concentration of mg S^{2-} /L in the sample as presented in 7.4.1.7. **NOTE: The volume sample titrated is the volume of the filtrate measured in 7.4.4.2.**

7.5 Analyze one laboratory control sample (LCS) for every twenty samples or each day's batch, whichever is more frequent. The LCS should consist of 200 mL of Laboratory reagent grade water spiked with 2.0 mL of the sodium sulfide standard (section 5.8) as described in 7.3.3 and 7.3.4.

7.6 Analyze one method blank for every twenty samples or each day's batch, whichever is more frequent. The method blank consists of 200 mL of Laboratory reagent grade water carried through the entire analysis.

7.7 Analyze one sample duplicate for every ten samples and one sample spike for every twenty samples or each day's batch. The sample selected as the matrix spike should be spiked with 2.0 mL of the sodium sulfide standard (Section 5.8) as described in 7.3.3 and 7.3.4.

7.8 Record keeping requirements are illustrated in the example logbook entries shown in Figure 1.

7.9 Enter results, including sample preparation information, measured sample concentrations, and quality control data, into the Katahdin Information Management System for calculation and reporting. Refer to the current revision of Sop CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.10 A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.11 Printouts of sample data are filed in the lab for approximately 3 months for reference by analysts.

7.12 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 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, 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 Acceptance criteria for the titrimetric LCS is 80-120% recovery for EPA 376.1, SM4500-S^{2-} F and SW846 9034. Acceptance criteria for reactive sulfide (SW846 7.3.4) is 50 –150% recovery. If LCS is out-of-criteria, reanalyze prior to beginning the titrations of the samples. If the LCS remains out, check the standardization of sulfide, iodine, and sodium thiosulfate. Reprepare the associated reagents and standards and repeat the LCS.

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8.2 Acceptance criteria for the method blank is a sulfide concentration less than or equal to the PQL (1.0 mg/L). For reactive sulfide the PQL is 27 mg/kg. If the method blank is out-of-criteria associated samples that are at a concentration of $\leq$ 10 x the associated blank value should be reanalyzed if possible. Samples with concentrations $\geq$ 10 x the blank may be reported with a "b" flag. If samples cannot be reanalyzed due to hold time expiration or insufficient sample volume the data will be reported with the appropriate narration, flag, or footnote.

8.3 Acceptance criteria for the titrimetric matrix spike are 75-125% for EPA 376.1; 50-150% for SW846 7.3.4; and 80-120 for SM4500-S^{2-} F. Recovery for the matrix spike and relative percent difference, (Results > three times the PQL), between sample and duplicative results < 20% for EPA 376.1, SW846 7.3.4, and SW846 9034; < 10% for SM 4500-S^{2-} E. For sample results less than three times the PQL, the RPD between sample and duplicate should be < 100%. If matrix spike recovery or duplicate RPD is out of criteria narrate or flag appropriately.

8.4 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.

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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 376.1 and SW846 Method 9034 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

EPA Method 376.1, "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983.

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 9034, Rev. 0, December, 1996.

American Public Health Association (APHA) (2005) Standard method for examination of water and wastewater, 21st edn. APHA, AWWA, WPCF, Washington, Method 4500-S^{2-} F,

American Public Health Association (APHA) (1992) Standard method for examination of water and wastewater, 18th edn. APHA, AWWA, WPCF, Washington, Method 4500-S^{2-} E

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, 10/06/2010.

Katahdin SOP CA-734, Reactive Sulfide: SW-846 7.3.4.2, 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
Titrimetric Sulfide EPA Method 376.1; SW846 9034 SW846 7.3.4 SM4500-S^{2-} F	Method blank	One per prep batch	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 where possible or flag results with "B"
	LCS	One per prep batch	EPA376.1 SW846 9034: 80-120 %R; SW8467.3.4: 50-150%R SM4500-S^{2-} F: 80-120%R	(1) If the LCS fails repeat LCS determination (2) Restandardize sulfide, iodine, and/or thiosulfate and repeat LCS. Reprepare affected reagent or standard and repeat LCS
	Matrix Spike	One for every set of 10 samples	EPA376.1; SW846 9034: 75-125 %R SM4500-S^{2-} F: 80-120%R	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. Low recovery may be due to acid-insoluble sulfides.
	Sample Duplicate	One sample duplicate per ten samples	(1) RPD $\leq$20 and <10% for SM4500-S^{2-} F (2) For sample results less than three times the PQL, the RPD between sample and duplicate should be < 100% for other methods.	(1) If RPD is outside criteria report original result with notation or narration.
	Demonstration of analyst proficiency – 4 replicates	Once per analyst	P&A meet method criteria	Repeat P&A study
	MDL study and/or LOD/LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-722-08	METHOD EPA 376.1; SW846 9034; SW846 7.3.3.4; SM4500-S^{2-} F, current revisions
Apparatus/Materials		
Reagents	0.0375 N sodium thiosulfate	0.025 N sodium thiosulfate
Sample preservation/ handling		
Procedures	(1) Pretreated and dewatered samples are transferred to beaker for titration with acidic iodine rinse of the bottle	(1) Pretreated and dewatered samples are titrated in the original sample container for E376.1 and SM4500-S^{2-} F
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

R449618
WG217790 E376.1

KATAHDIN ANALYTICAL SERVICES
SULFIDE

WG217931 SM4500

Methods (check one)		PQL: 1.0 mg/L
EPA 376.1	SW846 9034	Pipette IDs: WII // WQ
SM4500S^{2-} F (21st ED)	SM4500S^{2-} E	

REAGENTS

Iodine Standard: WI5815	HCL-6N: WI5907
Na2S Solution: WI5401	Na2S2O3 Titrant-0.0375N: SCWL4191
Starch End Indicator: WI5830	Other:

STANDARDIZATION OF I2 and Na2S

Vol. (ml) I2	Vol. (ml) Na2S2O3	CALC OF I2N	Vol. (ml) I2	Vol. (ml) Na2S2O3	Vol. (ml) Na2S	CALC Na2S mg/mL
10	6.65		10	4.85	2	
10	6.75		10	4.70	2	
10	6.70		10	4.75	2	
	X:			X:		

I2 meq Consumed = [(N I2 * Vol I2) - (N Na2S2O3 * Vol Na2S2O3)] * 16

S2- mg/L = I2 meq * 16 mg S2/meq * 1000 ml/L/Sample Volume (ml)

Time of Analysis	Sample ID	Sample Vol. (ml)	Na2S2O3 Vol. (ml)	I2 Vol. (ml)	S2 Vol. (ml)	S mg/L	Comments
15:40	Blank	200	6.70	10			
	LCS	200	4.70	10	2		
	TK0583-1	200	6.90	10			
	-2	200	6.50	10			
	-3	200	6.60	10			
	DUP -3	200	6.60	10			
	-4	200	6.60	10			
	MS -4	200	4.50	10	2		
	-5	200	6.80	10			
	MS -5	200	4.70	10	2		
	-8	200	6.80	10			
	-9	200	6.85	10			
	TK0486-1	200	6.90	10			
	-2	200	6.95	10			
	-5	200	6.95	10			
	-7	200	7.00	10			
	-9	200	6.70	10			
	TK0550-1	200	6.75	10			
	-2	200	6.70	10			
	-3	200	6.65	10			
16:50	TK0582-1	200	6.75	10			

NOTES:

ANALYST: [signature] DATE: 11-14-17

CHECKED BY: RD DATE: 11/20/17

WL-027 - Revision 2 - 08/23/2017 QAWL864 0000021

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

FIGURE 2

EXAMPLE OF CALCULATION SPREADSHEET

WG217930 R449618 WG217931 R449619

SULFIDE-TITRIMETRIC (EPA METHOD 376.1 / SW846 9030 / SM4500S2- E / SM4500S2- F [21ST EDITION])

Normality of $Na_2S_2O_3$ =	0.0375

Analysis Date:	11/14/2017
Analyst:	AZ
PQL, mg/L:	1.0
Matrix:	Aqueous

Standardization of I_2

Vol I_2 (mL)	Vol $Na_2S_2O_3$ (mL)
10.0	6.65
10.0	6.75
10.0	6.70
Mean =	6.7
Calc I_2 N =	0.0251

Standardization of Na_2S (CCV)

Vol I_2 (mL)	Vol $Na_2S_2O_3$ (mL)	Vol Na_2S (mL)
10.0		2.00
10.0		2.00
10.0		2.00
Means =	#DIV/0!	2.00
Calc Na_2S (mg/mL) =		#DIV/0!

Standardization of Na_2S (LCS/MS)

Vol I_2 (mL)	Vol $Na_2S_2O_3$ (mL)	Vol Na_2S (mL)
10.0	4.85	2.00
10.0	4.70	2.00
10.0	4.75	2.00
Means =	4.77	2.00
Calc Na_2S (mg/mL) =		0.580

Sample ID	QC Type	Initial Vol. (mL)	Volume I_2 (mL)	Volume $Na_2S_2O_3$ (mL)	Calculated S_2 (mg)	Calculated S_2 (mg/L)	Dil. Fctr.	Reported S_2 Concentration (mg/L)	Spike Added (mg/L)	CCV/LCS Rcvy. (%)	MS Rcvy. (%)	DUP RPD (%)
BLANK		200	10.0	6.70	0.000	0	1.00	<1.00		#DIV/0!	#DIV/0!	#DIV/0!
LCS	LCS	200	10.0	4.70	1.200	6.000	1.00	6.00	5.80	103.45%		
TK0583-1		200	10.0	6.90	-0.120	-0.600	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0583-2		200	10.0	6.50	0.120	0.600	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0583-3		200	10.0	6.60	0.060	0.300	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0583-3 DUP	DUP	200	10.0	6.60	0.060	0.300	1.00	< 1.00				NC
TK0583-4		200	10.0	6.60	0.060	0.300	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0583-4 MS	MS	200	10.0	4.50	1.320	6.600	1.00	6.60	5.80		113.79%	
TK0583-5		200	10.0	6.80	-0.060	-0.300	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0583-5 MS	MS	200	10.0	4.70	1.200	6.000	1.00	6.00	5.80		103.45%	
TK0583-8		200	10.0	6.80	-0.060	-0.300	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0583-9		200	10.0	6.85	-0.090	-0.450	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0486-1		200	10.0	6.90	-0.120	-0.600	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0486-2		200	10.0	6.95	-0.150	-0.750	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0486-5		200	10.0	6.95	-0.150	-0.750	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0486-7		200	10.0	7.00	-0.180	-0.900	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0486-9		200	10.0	6.70	0.000	0	1.00	<1.00		#DIV/0!	#DIV/0!	-100.00%
TK0550-1		200	10.0	6.75	-0.030	-0.150	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0550-2		200	10.0	6.70	0.000	0	1.00	<1.00		#DIV/0!	#DIV/0!	-100.00%
TK0550-3		200	10.0	6.65	0.030	0.150	1.00	< 1.00		#DIV/0!	#DIV/0!	NC
TK0582-1		200	10.0	6.75	-0.030	-0.150	1.00	< 1.00		#DIV/0!	#DIV/0!	NC

TITLE: TITRIMETRIC DETERMINATION OF SULFIDE USING EPA METHOD 376.1, SM4500-S^{2-} F, SW846 9034 and SW846 7.3.4

FIGURE 3

EXAMPLE OF BATCH SHEET

WET CHEMISTRY BATCH REPORT
Nov 20 2017, 11:49 am
Batch: WG217931

Parameter: Sulfide-Iodometric
Prep Date: N/A

Date Analyzed: 14-NOV-17
Prep Method: N/A

Analyst Initials: AZ
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
TK0486-1	SAMP	SM4500-S2-F	200.00mL	200.00mL	1	-.6	U0.80 mg/L	NA	1	0.69	1.0		
TK0486-2	SAMP	SM4500-S2-F	200.00mL	200.00mL	1	-.75	U0.80 mg/L	NA	1	0.69	1.0		
TK0486-5	SAMP	SM4500-S2-F	200.00mL	200.00mL	1	-.75	U0.80 mg/L	NA	1	0.69	1.0		
TK0486-7	SAMP	SM4500-S2-F	200.00mL	200.00mL	1	-.9	U0.80 mg/L	NA	1	0.69	1.0		
TK0486-9	SAMP	SM4500-S2-F	200.00mL	200.00mL	1	0	U0.80 mg/L	NA	1	0.69	1.0		
TK0550-1	SAMP	SM4500S F	200.00mL	200.00mL	1	-.15	U1.0 mg/L	NA	1	0.69	1.0		
TK0550-2	SAMP	SM4500S F	200.00mL	200.00mL	1	0	U1.0 mg/L	NA	1	0.69	1.0		
TK0550-3	SAMP	SM4500S F	200.00mL	200.00mL	1	.15	U1.0 mg/L	NA	1	0.69	1.0		
TK0582-1	SAMP	SM4500S F	200.00mL	200.00mL	1	-.15	U1.0 mg/L	NA	1	0.69	1.0		
WG217931-1	MBLANK	SM4500S F	200.00mL	200.00mL	1	0	U0.80 mg/L	NA	1	0.69	1.0		
WG217931-2	LCS	SM4500S F	200.00mL	200.00mL	1	6	6.0 mg/L	NA	1	0.69	1.0		103

Comments:

TK0486-2	MS/MSD on VOA/SVOA only
WG217931-1	TK0486-1
WG217931-2	TK0486-1

Entered by: _____ Date: 11-2017 Accepted by: _____ Date: 11/20/17

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Ryan Oliver

Review Date: 1/18/19

SOP Number: CA-722-08

SOP Title: Titrimetric Determination of Sulfide Using EPA method 376.1, SM4500-S2F, SW846 9034 & SW8467.3.4

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: K. Brewer **Date:** 01/24/19

QAO Signature: Jessie Dimond **Date:** 01.24.19

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

Prepared By: Gary Brewer Date: 05/04/07

Approved By:

Department Manager: Gary Brewer Date: 05/04/07

Lab Operations Mgr: Deborah J. Nadeau Date: 5.4.07

QA Officer: Leslie Dimond Date: 5-4-07

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout to reflect current practices. Updated Figures 1-4. Added glassware modifications to Table 2. Corrected calculation. Added PQL of 0.1 umole/g. Added stability time criteria to sect. 7.	LAD	05/09	05/09
02	Updated and/or added references to Sect.s 1.2, 1.3, 9 and 10. Added LOQ/LOD to Table 1.	LAD	06/10	06/10
03	Sect. 5 - Added metals spiking solutions. Sect. 7 - Added instructions for spiking LCSs and MSs. Sect. 9 - Added MDL, LOD & LOQ information. Sect. 10 - Added and edited references. Updated Figures 1→3.	LAD	05/12	05/12
04	Sect. 1 - Added MDR safety concerns, added additional waste disposal directions. Sect. 7 - Added wording for procedural clarification. Sect. 8 - Added contingency plan. Sect 10 - Updated references	LAD	01/19	01/19

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

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-738-04**, titled **Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-738-04**, titled **Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediments.**

Recipient: _____ Date: _____

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel for the determination of: 1) acid volatile sulfide (AVS) and 2) the simultaneously extractable metals (SEM) that are solubilized during the acidification step. The conditions used have been reported to measure amorphous moderately crystalline monosulfides. As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailibility of metals in anoxic sediments. If the molar ratio of toxic metals measured by SEM to AVS exceeds one, the metals are potentially bioavailable. Because the relative amounts of AVS and SEM are important in the prediction of potential metal bioavailability, it is important to use the SEM procedure for sample preparation for metal analysis. This uses the same conditions for release of both sulfide and metal from the sediment and thus provides the most predictive means of assessing the amount of metal associated with sulfide.

1.1 Definitions

Method Blank - A deionized water sample that is carried through the entire analytical procedure in the same manner as a sample.

ICV - Initial Calibration Verification. One ICV per batch is prepared from a separate source from the CCV and calibration curve standards. ICV verifies the calibration curve.

LCS- Laboratory Control Standard. One LCS per batch is carried through the entire analytical procedure in the same manner as a sample. The LCS is prepared from the same source as the ICV.

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 deionized water with no reagents 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 AVS and SEM by EPA Draft Method 821-R-91-100. 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 AVS or SEM by EPA Draft Method 821-R-91-100 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.

Mixed Diamine Reagent (MDR): Corrosive and toxic, a face shield should be worn in addition to standard PPE when preparing solution.

Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of approximately 0.002 ppm. Handling of acid samples should be performed in a hood or in a well ventilated area.

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 Waste 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 Analytical 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.

Place all analyzed samples, standards, rinsates, and unsaved portions of SEM samples (reacted AVS LCS and MS's) in a Satellite Waste "A" Acid for proper disposal in main waste area "A". W1, W2, & filtered portions of S1 & S2 standards, as well as leftover AVS solutions from distillation should be disposed of in Satellite Waste "N-Hi" for proper disposal in main waste area "N-Hi".

2.0 SUMMARY OF METHOD

The method measures the concentrations of AVS and SEM liberated during the acidification of a 10 g sediment sample. Hydrochloric acid is added to the sample to liberate the volatile sufides as hydrogen sulfide (H_2S) at room temperature, and then the H_2S is purged from the sample and collected into an aqueous buffer solution. The amount of AVS that has been trapped is then measured colorimetrically, by reacting sulfide with N-N-dimethyl-p-phenylenediamine to form methylene blue. SEM are determined after filtration of the sediment-acid slurry using ICP spectrometry.

3.0 INTERFERENCES

Oxidation of sulfide in samples may result in a low bias in the results obtained for AVS and SEM. For this reason, contact of the samples with oxygen must be avoided in all stages from sampling through analysis. Collecting samples in containers with minimal headspace, minimizing contact of samples with the air during sample preparation, and using deaerated reagents during sample preparation and analysis all aid in preventing oxidation of samples.

To avoid metals contamination during the SEM digestion part of the method, all reaction vessels and scrubbers should be acid washed prior to use.

4.0 APPARATUS AND MATERIALS

4.1 Polyethylene gas-washing bottles with rubber stoppers and fritted polyethylene spargers, 250 mL capacity, for use as reaction vessels and sulfide traps.

4.2 Flexible tubing for connection from the nitrogen supply to the apparatus and from the flask to the absorber unit.

4.3 High purity nitrogen gas tank with regulator

4.4 Flow meters with needle valves

4.5 Spectrophotometer capable of measuring absorbance at 670 nm

4.6 Spectrophotometer cells, 1 cm path length

4.7 Analytical balance capable of weighing to 0.001 g

4.8 10 mL buret, calibrated in 1/10 mL increments

4.9 Calibrated adjustable pipettors, 0.1 mL, 1.0 mL, and 5.0 mL capacity

4.10 250 mL snap cap graduated containers

4.11 50 mL polyethylene graduated containers

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water, deaerated by sparging with nitrogen for one hour just prior to use. Dissolved oxygen should be close to 1 mg/L.

5.2 Sodium hydroxide (NaOH) solution, 0.5 M: Dilute 50.0 mL of 10 N NaOH solution to 1 liter with laboratory reagent grade water. This solution could also be made by dissolving 20 g of NaOH in DI water and diluting to 1 liter. This solution must be deaerated by sparging with nitrogen for one hour just prior to use.

5.3 Starch solution: Make a paste consisting of 2 g lab grade soluble starch, 0.2 g of salicylic acid in a few mL of DI water. Then bring up to volume in a 100 mL volumetric flask. Or use a purchased solution.

5.4 Standard sodium thiosulfate solution, 0.0375 N: Use a purchased primary standard sodium thiosulfate solution with its concentration certified by the vendor.

5.5 Hydrochloric acid, 6 N: In a 1 L volumetric flask, add 500 mL of concentrated HCl to approximately 400 mL of laboratory reagent grade water, slowly mix and allow to cool. Bring to a final volume of 1 L with laboratory reagent grade water. This solution should be deaerated by sparging with nitrogen for one hour prior to use.

5.6 Standard iodine solution, approximately 0.0250N: Dissolve 20 - 25 g potassium iodide in 200 mL of laboratory reagent grade water and add 3.2 g iodine. After the iodine has dissolved, dilute to 1 L with laboratory reagent grade water and standardize against 0.0375 N sodium thiosulfate using the starch solution as an indicator, as explained in Sections 7.8 – 7.11.

5.7 Saturated sodium sulfide standard solutions: Because sulfide is unstable, a standard of pre-determined concentration cannot be prepared. However, a saturated sulfide standard solution can be prepared by dissolving 20 g of sodium sulfide crystals in 10 mL of deaerated laboratory reagent grade water. The sodium sulfide crystals will not all dissolve (if at any point all the crystals in this solution dissolve add more crystals). Two saturated sodium sulfide solutions are required for the analysis – one standard (labeled "S2") is used to prepare the calibration curve and the continuing calibration verification standard, and the other standard (labeled "S1") is used to prepare laboratory control samples and matrix spikes. The concentrations of these saturated standards are determined titrimetrically just prior to use as described in Sections 7.12 – 7.15.

5.8 Sulfide working standard "W1": Fill a 100 mL volumetric flask with deaerated laboratory reagent grade water to about 1 cm below the 100 mL graduation. Using a calibrated adjustable pipet, pipet 0.10 mL of saturated sodium sulfide stock standard "S1" into the flask, dispensing the standard below the surface of the water in the flask. Bring the volume in the flask to the 100 mL graduation with deaerated laboratory reagent grade water, stopper, and invert to mix. Sulfide working standard "W1" must be prepared just prior to use, and may be used to prepare an initial calibration standard for the colorimetric analysis. The sulfide concentration of this standard is $1/1000^{th}$ that of saturated sodium sulfide stock standard "S1", which is determined titrimetrically (see Sections 7.12 – 7.15).

5.9 Sulfide working standard "W2": Fill a 100 mL volumetric flask with deaerated laboratory reagent grade water to about 1 cm below the 100 mL graduation. Using a calibrated adjustable pipet, pipet 0.10 mL of saturated sodium sulfide stock standard "S2" into the flask, dispensing the standard below the surface of the water in the flask. Bring the volume in the flask to the 100 mL graduation with deaerated laboratory reagent grade water, stopper, and invert to mix. Sulfide working standard "W2" must be prepared just prior to use, and is used to prepare the calibration standards and a continuing calibration standard for the colorimetric analysis. The sulfide concentration of this standard is $1/1000^{th}$ that of saturated sodium sulfide stock standard "S2", which is determined titrimetrically (see Sections 7.12 – 7.15).

5.10 Metals spiking standards: Four different standards, named CLPP-SPK-1, CLPP-SPK-INT1, CLPP-SPK-INT2, and Intermediate Mercury Standard A, are required. Intermediate Mercury Standard A has a mercury concentration of 1000 ug/L, and instructions for its preparation are contained in Section 5.9 of the current revision of SOP CA-615 (Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470). Details of the compositions and preparation of the other metals spiking standards are contained in Figures 2 and 3 of the current revision of SOP CA-604 (Acid Digestion of Aqueous Samples By EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals).

5.11 Mixed Diamine Reagent (MDR): Prepare Components A and B as follows, and then mix them together:

5.11.1 Component A - Add 660 mL concentrated sulfuric acid to 340 mL of laboratory reagent grade water. After the solution cools, dissolve 2.25 g N-N-dimethy-p-phenylenediamine oxalate in it.

5.11.2 Component B - Dissolved 5.4 g ferric chloride hexahydrate ($FeCl3 \cdot 6H2O$) in 100 mL concentrated hydrochloric acid and dilute to 200 mL with reagent water.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Sulfide ion is unstable in the presence of oxygen. Protect sediment samples from exposure to oxygen during sample collection and storage.

6.2 Samples should be collected in wide mouth jars, and tightly capped with a minimum of air space above the sediment. The jar lids must have Teflon or polyethylene liners. Alternatively, samples may be collected in syringe samplers (syringes with the tip ends cut off) by plunging the syringe into the sediment while drawing up the plunger, and then tightly capping the open end of the syringe. The syringe sampling method effectively eliminates contact with atmospheric oxygen.

6.3 Samples should be cooled to 4^{o} C as soon as possible after collection, and stored at 4^{o} C until analysis.

6.4 Holding time for samples should not exceed 14 days.

7.0 PROCEDURES

GENERATION OF HYDROGEN SULFIDE FROM SAMPLES

7.1 Assemble the sulfide generation apparatus as shown in Figure 5. The nitrogen gas supply must be shut off. Fill each sulfide trap with 170 mL of 0.5 M NaOH solution.

7.2 Add 100 mL of deaerated laboratory reagent grade water into each reaction vessel. Weigh out approximately 10 g of sample directly into each reaction vessel, minimizing contact with the air. If the sample has been collected in a wide mouth jar, quickly open the jar, scrape away the top centimeter of sediment, scoop up an aliquot of the sample, and transfer it to the tared, water-filled reaction vessel. If the sample has been collected in a syringe sampler, quickly uncap the syringe and extrude the contents of the syringe directly into the tared, water-filled reaction vessel. Record the

sediment weight. Stopper the gas reaction vessel and attach its outlet to the inlet of the sulfide trap.

7.3 All Acid Volatile Sulfide (AVS) spiking should be done beneath the surface of the water in the reaction vessel by penetrating the water's surface with the pipette tip containing the spike and expelling it while submerged. Prepare a Laboratory Control Sample for AVS by spiking the deaerated laboratory reagent grade water in a reaction vessel with 0.10 mL of saturated sodium sulfide stock standard "S1". Prepare AVS matrix spike samples by spiking 0.10 mL of saturated sodium sulfide stock standard "S1" into the reaction vessel containing deaerated water and a sample aliquot.

7.4 In order to prevent reactions between added AVS spiking solutions and SEM spiking solutions, Laboratory Control Samples (LCS) and matrix spikes for Simultaneously Extractable Metals (SEM) must be prepared in separate reaction vessels from the AVS Laboratory Control Sample and AVS matrix spikes. Prepare each SEM LCS and matrix spike by adding the following amounts of each spiking solution to the appropriate reaction vessels.

SEM Spiking Solution	Amount Added (mL)
CLPP-SPK-1	0.20
CLPP-SPK-INT1	2.00
CLPP-SPK-INT2	2.00
Intermediate Mercury Standard A	1.00

7.5 There are three valves in which nitrogen passes through to reach the reaction vessel. The main valve (located in the Extractions lab), the hood valve (located between the two hoods in the Wet lab, left position), and the sample valve. Turn on the main valve on the nitrogen tank and make sure the high pressure gauge reads 300 PSI or more.

7.6 Connect the inlets of the reaction vessels to the nitrogen gas supply. Make sure all tubing connections and stoppers are tight. Make sure the hood valve is in the off position (turn clockwise). Turn the sample valves to the off position (clockwise). Unstopper the reaction vessel and quickly add 20 mL of 6.0 M HCl to the water and sediment in the vessel. Quickly re-stopper the reaction vessel tightly. Some samples may effervesce with the addition of HCl. If this happens, open the sample valve and the hood valve slightly to encourage the airflow from the reaction vessel to the trap vessel. This prevents back flow. Once all samples have been acidified, slightly open all the sample valves and if it isn't already, open the hood valve. Adjust the valves so all samples are bubbling equally.

7.7 After 60 minutes, disconnect the sulfide trap, transfer the sodium hydroxide solution from the trap into a 250 mL plastic container, and shut off the main valve on the nitrogen tank. Retain the sodium hydroxide solution for colorimetric analysis of sulfide.

7.8 Filter the solution sediment / acid slurry remaining in the reaction vessel through a membrane filter (pore size 0.45um) and save the filtrate in a 250 mL snap cap container for the SEM analysis. Bring the volume of filtrate to 200 mL in each snap cap container with deionized water, cap the vessel, and shake well to mix. The filtrate is now ready for SEM analysis.

TITRATION OF STANDARD IODINE SOLUTION

7.10 Add 15 mL of standard iodine solution and 2.0 mL of 6 N HCl to 200 mL of deaerated laboratory reagent grade water in a 250 mL Erlenmeyer flask. The solution will be an orange-yellow color.

7.11 Using the 10 mL buret, titrate this solution with 0.0375 N sodium thiosulfate standard solutions until a pale straw yellow color is obtained.

7.12 Add a few drops of starch solution to produce a deep blue color in the solution. Continue the titration with sodium thiosulfate until a colorless endpoint is reached. Record the total volume of sodium thiosulfate solution that was added to reach the endpoint.

7.13 Repeat the titration two more times, recording the volume of sodium thiosulfate required to reach the endpoint each time. Enter the appropriate volumes and normalities in the AVS Calculation Spreadsheet to calculate the normality of the standard iodine solution. The spreadsheet calculates the mean normality from the three titrations.

TITRATION OF SATURATED SODIUM SULFIDE STOCK STANDARDS

7.14 Add 200 mL of deaerated laboratory reagent grade water to a 250 mL Erlenmeyer flask. Pipet 0.10 mL of one of the saturated sodium sulfide stock standards to the water in the flask, placing the pipet tip below the surface of the water while adding the sulfide standard. Add 10.0 mL portions of standard iodine solution to the flask until a persistent orange-yellow color is obtained. Record the volume of iodine solution used, and then add 2.0 mL of 6 N hydrochloric acid to the flask.

7.15 Using the 10 mL buret, titrate this solution with 0.0375 N sodium thiosulfate standard solution until a pale straw yellow color is obtained.

7.16 Add a few drops of starch solution to produce a deep blue color in the solution. Continue the titration with sodium thiosulfate until a colorless endpoint is reached. Record the total volume of sodium thiosulfate solution that was added to reach the endpoint.

7.17 Repeat the titration two more times, recording the volume of sodium thiosulfate required to reach the endpoint each time. Enter the appropriate volumes and normalities in the AVS Calculation Spreadsheet to calculate the concentration of the saturated sodium sulfide stock standard. The spreadsheet calculates the mean sulfide concentration of the standard from the three titrations. Triplicate titrations must be performed on both of the saturated sodium sulfide stock solutions.

PREPARATION OF CALIBRATION STANDARDS AND THE CONTINUING CALIBRATION VERIFICATION STANDARD

7.18 Prepare a calibration curve as follows. Add 40 mL of 0.5 M sodium hydroxide solution into each of eight 50 mL graduated polyethylene containers. Using calibrated adjustable pipets, pipet the following volumes of Sulfide Working Standard W2 into the containers, dispensing the standard below the surface of the sodium hydroxide solution: 0.00 mL, 0.04 mL, 0.10 mL, 0.30 mL, 0.50 mL, 0.70 mL, 0.90 mL, 1.00 mL. The sulfide concentrations of the calibration standards will vary depending on the actual concentration the sulfide stock standard, as determined in Sections 7.12 – 7.15. The standard that contains 0.50 mL of Sulfide Working Standard W2 is analyzed throughout the colorimetric analysis as the Continuing Calibration Verification standard.

COLORIMETRIC ANALYSIS OF SAMPLES AND STANDARDS

7.19 Standards: A face shield should be worn. Working in the fume hood, add 5.0 mL MDR to each standard, and bring the volume to the 50 mL graduation with deaerated laboratory reagent grade water. These must be mixed by inversion and vented immediately to prevent the pressure buildup causing snap cap to burst open. Solution is extremely dangerous to come in contact with. Open and close snap caps with care to prevent splashing. After 30 minutes, measure the absorbance of the standards at 670 nm. The absorbance of the reacted samples is stable for no more than two hours.

7.20 Samples: A face shield should be worn. Working in the fume hood, transfer an appropriate volume aliquot of the scrubber solution (depending on the expected sulfide concentration) into a 50 mL graduated polyethylene container. Add sufficient 0.5M sodium hydroxide solution to bring the total volume to 40 mL. Add 5.0 mL MDR to the container and bring the solution to the 50 mL graduation with deaerated laboratory reagent grade water. These must be mixed by inversion and vented immediately to prevent the pressure buildup from causing snap cap to burst open.

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

Solution is extremely dangerous to come in contact with. Open and close snap caps with care to prevent splashing. After 30 minutes, measure the absorbance of the samples at 670 nm. The absorbance of the reacted samples is stable for no more than two hours.

CALCULATIONS

7.21 Calculate the concentration of the saturated sodium sulfide stock standards as follows:

$$S^{2-}\ (mg/L) = \frac{(A \times B) - (C \times D) \times 16000}{E}$$

where:
- A = mL of I_2 solution
- B = Normality of I_2 solution
- C = mL of Thiosulfate Titrant
- D = Normality of Thiosulfate Titrant
- E = Volume of Sulfide Standard added to flask

7.20 Calculate the LCS and Spike level as follows:

$$LCS/Spike\ (mg/kg) = (F \times G) / H$$

where:
- F = mL of the sulfide
- G = concentration of the sulfide standard
- H = weight of sample

7.21 Calculate the concentration of sulfide in samples as follows:

$$\frac{umole}{g}\ S^{2-} = \frac{Final\ Vol \times umole\ S-}{analysis\ volume} \times \frac{1}{sample\ wt\ g} \times \frac{1}{\%\ Solids}$$

7.22 The above calculations can be done using a spreadsheet template (Figure 2) on which the analyst enters the sample number, date prepared, date analyzed, sample weight and final volume, volume of sample, volume of Iodine and the normality of both the iodine and thiosulfate. Manually calculated results should be reported to 2 significant figures.

7.23 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.

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

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.

Refer to Table 1 for summary of QC requirements.

8.1 Distill and analyze a method blank with every batch or analytical session. The concentration of the blank is expected to be less than the Practical Quantitation Limit (.1umole/g).

8.2 Analyze a Continuing Calibration Verification standard at the beginning of every analytical session, after every 10 samples, and at the end of each analytical session. The acceptance limits for the CCV are 80% - 120% recovery.

8.3 Prepare and analyze one duplicate sample in every batch of 20 or fewer samples. The results for duplicate samples must agree within 20% relative percent difference (RPD).

8.4 Prepare and analyze one matrix spike sample with every 10 client samples. The acceptance limits for matrix spike recoveries are 75% - 125% of the added analyte concentration.

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 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 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 the applicable methods for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, Allen et al, 1991, Document 821/R-91-100

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

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 QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
AVS SEM	Initial Calibration – Minimum of 5 pt. plus blank	Each day of analysis	r $\geq$ 0.995	(1) Recalibrate
	Method blank	One per prep batch of 20 or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank
	LCS/ICV	One of each per prep batch	80-120 %R; Statistically derived from lab data	(1) Recalibrate and reanalyze sample batch
	CCV	At beginning of run, after every 10 samples and at the end of the run. Same conc. As LCS/ICV	80-120 %R	(1) Reanalyze all samples back to last acceptable CCV recovery
	Matrix Spike	One for every set of 10 samples	75-125 %R	(1) Notate sample result in raw data with Notation I-1
	Sample Duplicate	One sample duplicate per twenty samples or per batch	RPD $\leq$20	(1) If lab QC 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	Refer to KAS SOP QA-806, “, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision		
	CCB	At beginning of run, after every 10 samples and at the end of the run.	No analyte detected > PQL.	Reanalyze all samples after last acceptable CCB.

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

TABLE 2

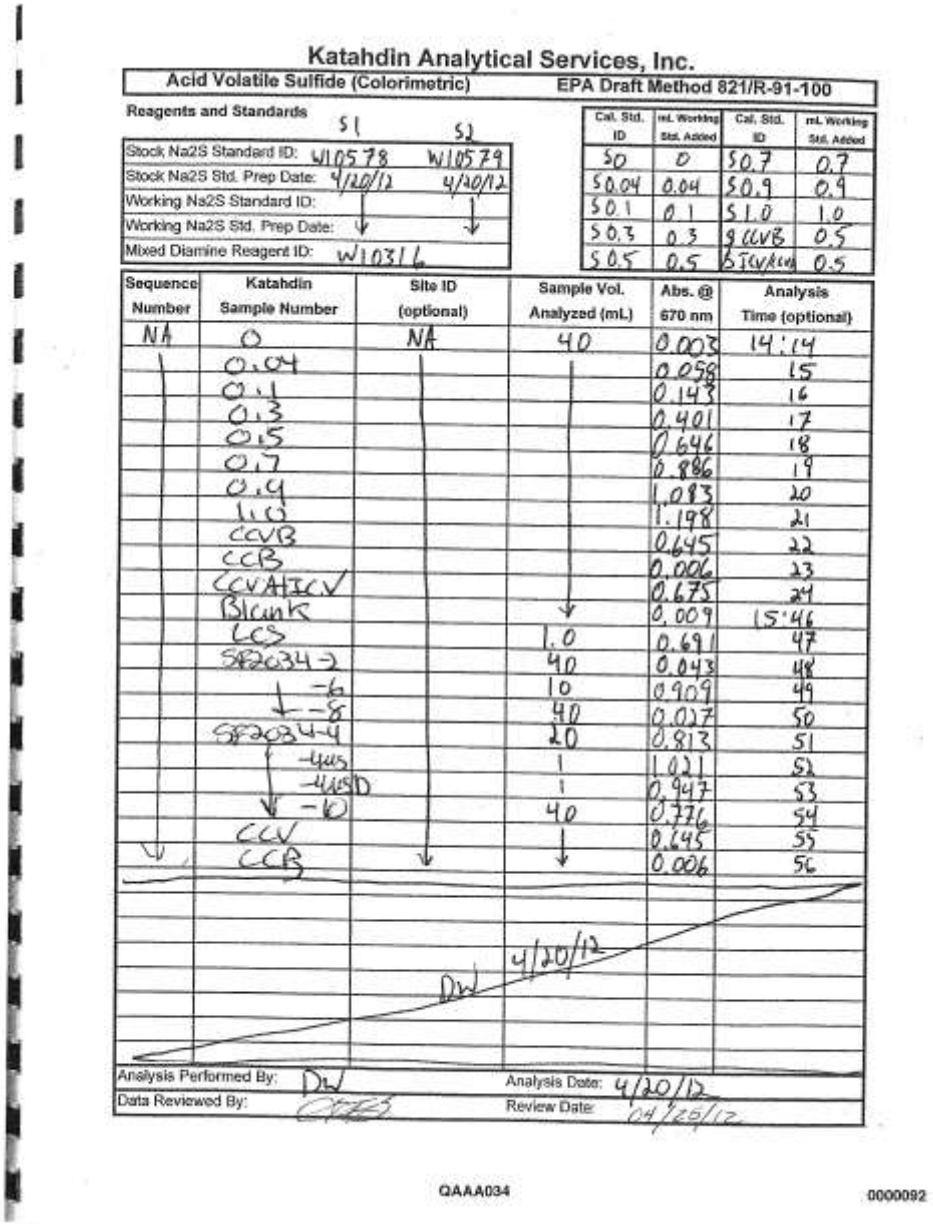
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-738-04	Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment
Apparatus/ Materials	Use gas washing bottles	Use glassware with ground glass connections
Reagents		
Sample preservation/ handling		
Procedures	170 mL 0.5N NaOH used	80 mL 0.5N NaOH used
QC – Spikes	Recovery acceptance limits = 75% - 125%	Recovery acceptance limits = 85% - 105%
QC – LCS	Recovery acceptance limits = 80% - 120%	Recovery acceptance limits = 85% - 105%
QC - Accuracy/Precision		
QC – MDL		

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 1

EXAMPLE OF WATERS AND SOILS RUNLOG PAGE



Katahdin Analytical Services, Inc.

Acid Volatile Sulfide (Colorimetric) — **EPA Draft Method 821/R-91-100**

Reagents and Standards

	S1	S2
Stock Na2S Standard ID:	W10578	W10579
Stock Na2S Std. Prep Date:	4/20/12	4/20/12
Working Na2S Standard ID:	↓	↓
Working Na2S Std. Prep Date:	↓	↓
Mixed Diamine Reagent ID:	W10316	

Cal. Std. ID	mL Working Std. Added	Cal. Std. ID	mL Working Std. Added
S0	0	S0.7	0.7
S0.04	0.04	S0.9	0.9
S0.1	0.1	S1.0	1.0
S0.3	0.3	2 CCVB	0.5
S0.5	0.5	BICVICV	0.5

Sequence Number	Katahdin Sample Number	Site ID (optional)	Sample Vol. Analyzed (mL)	Abs. @ 670 nm	Analysis Time (optional)
NA	0	NA	40	0.003	14:14
	0.04			0.058	15
	0.1			0.143	16
	0.3			0.401	17
	0.5			0.646	18
	0.7			0.886	19
	0.9			1.083	20
	1.0			1.198	21
	CCVB			0.645	22
	CCB			0.006	23
	CCVAICV			0.675	24
	Blank		↓	0.009	15:46
	LCS		1.0	0.691	47
	SB2034-2		40	0.043	48
	-6		10	0.909	49
	↓ -8		40	0.027	50
	SB2034-4		20	0.813	51
	-4MS		1	1.021	52
	-4MSD		1	0.947	53
	↓ -10		40	0.776	54
	CCV			0.645	55
↓	CCB	↓	↓	0.006	56

4/20/12 DW

Analysis Performed By: DW — Analysis Date: 4/20/12
Data Reviewed By: [illegible] — Review Date: 04/20/12

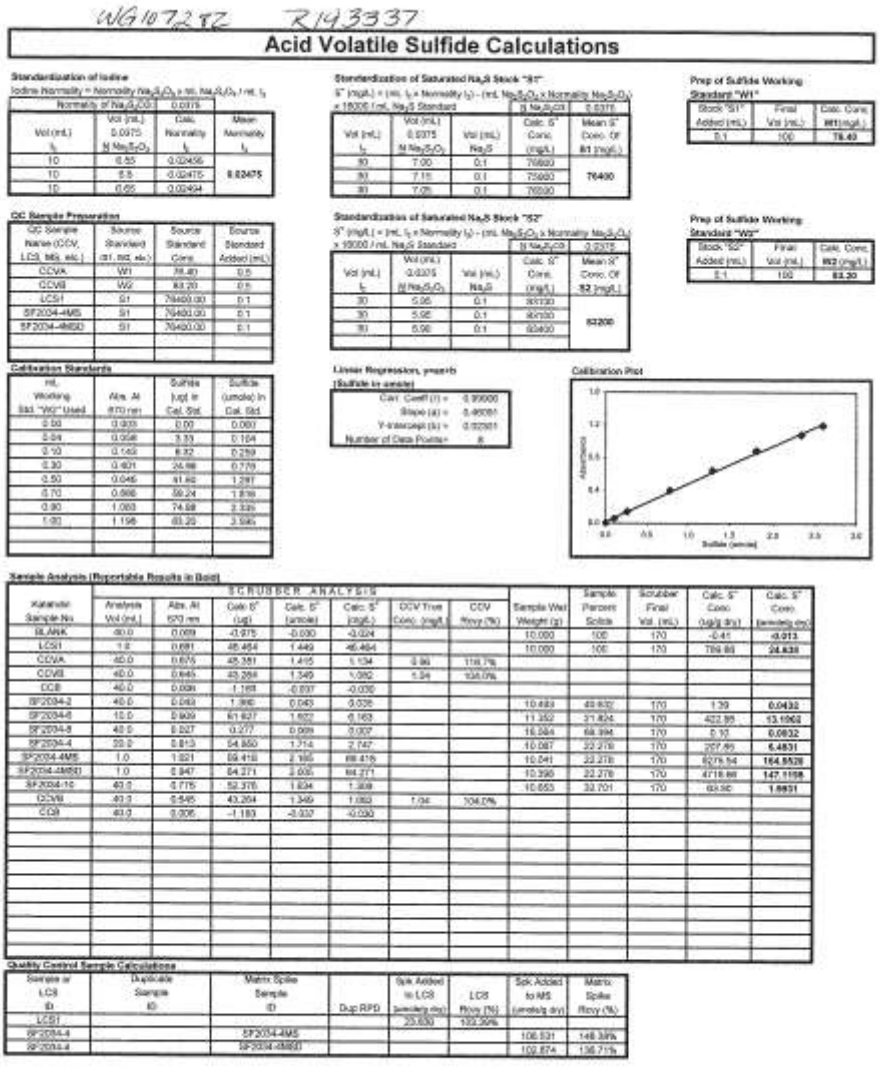
QAAA034

0000092

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 2

EXAMPLE OF AVS CALCULATION SPREADSHEET



WG107282 R143337

Acid Volatile Sulfide Calculations

Standardization of Iodine

Iodine Normality = Normality $Na_2S_2O_3$ x mL $Na_2S_2O_3$ / mL I_2

Normality of $Na_2S_2O_3$		0.0375	
Vol (mL) I_2	Vol (mL) 0.0375 N $Na_2S_2O_3$	Calc. Normality I_2	Mean Normality I_2
10	8.55	0.02466	0.02475
10	8.5	0.02475	
10	8.65	0.02464	

Standardization of Saturated Na_2S Stock "S1"

S$^{=}$ (mg/L) = (mL I_2 x Normality I_2) - (mL $Na_2S_2O_3$ x Normality $Na_2S_2O_3$) x 16000 / mL Na_2S Standard

			N $Na_2S_2O_3$	0.0375
Vol (mL) I_2	Vol (mL) 0.0375 N $Na_2S_2O_3$	Vol (mL) Na_2S	Calc. S$^{=}$ Conc. (mg/L)	Mean S$^{=}$ Conc. Of **S1** (mg/L)
30	7.00	0.1	76800	76400
30	7.15	0.1	75600	
30	7.05	0.1	76800	

Prep of Sulfide Working Standard "W1"

Stock "S1" Added (mL)	Final Vol (mL)	Calc. Conc. **W1** (mg/L)
0.1	100	**76.40**

QC Sample Preparation

QC Sample Name (CCV, LCS, MS, etc.)	Source Standard (S1, W2, etc.)	Source Standard Conc.	Source Standard Added (mL)
CCV4	W1	76.40	0.5
CCV8	W2	83.20	0.5
LCS1	S1	76400.00	0.1
SF2034-4MS	S1	76400.00	0.1
SF2034-4MSD	S1	76400.00	0.1

Standardization of Saturated Na_2S Stock "S2"

S$^{=}$ (mg/L) = (mL I_2 x Normality I_2) - (mL $Na_2S_2O_3$ x Normality $Na_2S_2O_3$) x 16000 / mL Na_2S Standard

			N $Na_2S_2O_3$	0.0375
Vol (mL) I_2	Vol (mL) 0.0375 N $Na_2S_2O_3$	Vol (mL) Na_2S	Calc. S$^{=}$ Conc. (mg/L)	Mean S$^{=}$ Conc. Of **S2** (mg/L)
30	5.95	0.1	83200	83200
30	5.95	0.1	83200	
30	5.95	0.1	83200	

Prep of Sulfide Working Standard "W2"

Stock "S2" Added (mL)	Final Vol (mL)	Calc. Conc. **W2** (mg/L)
0.1	100	**83.20**

Calibration Standards

mL Working Std. "W2" Used	Abs. at 670 nm	Sulfide (ug) in Cal. Std.	Sulfide (umoles) in Cal. Std.
0.00	0.000	0.00	0.000
0.04	0.008	3.33	0.104
0.10	0.140	8.32	0.259
0.30	0.401	24.96	0.779
0.50	0.646	41.60	1.297
0.70	0.866	58.24	1.816
0.90	1.060	74.88	2.335
1.00	1.156	83.20	2.595

Linear Regression, y=a+bx (Sulfide in umole)

Corr. Coeff (r) =	0.99900
Slope (a) =	0.46081
Y-intercept (b) =	0.02301
Number of Data Points=	8

Calibration Plot

Sample Analysis (Reportable Results in Bold)

	SCRUBBER ANALYSIS											
Katahdin Sample No.	Analysis Vol (mL)	Abs. At 670 nm	Calc S$^{=}$ (ug)	Calc. S$^{=}$ (umole)	Calc. S$^{=}$ (mg/L)	CCV True Conc. (mg/L)	CCV Recv (%)	Sample Wet Weight (g)	Sample Percent Solids	Scrubber Final Vol. (mL)	Calc. S$^{=}$ Conc. (ug/g dry)	Calc. S$^{=}$ Conc. (umole/g dry)
BLANK	40.0	0.009	-0.975	-0.030	-0.024			10.000	100	170	-0.41	**-0.013**
LCS1	1.0	0.691	45.464	1.449	45.464			10.000	100	170	769.88	**24.539**
CCV4	40.0	0.675	45.351	1.445	1.134	0.96	118.7%					
CCV8	40.0	0.645	43.264	1.349	1.082	1.04	104.0%					
CCB	40.0	0.009	-1.189	-0.037	-0.030							
SF2034-2	40.0	0.049	1.866	0.049	0.035			10.483	49.830	170	1.39	**0.0432**
SF2034-6	10.0	0.909	61.627	1.922	6.163			11.352	31.824	170	422.85	**13.1062**
SF2034-8	40.0	0.027	0.277	0.009	0.007			10.284	68.394	170	0.10	**0.0032**
SF2034-4	20.0	0.813	54.850	1.714	2.747			10.087	32.278	170	257.85	**8.4831**
SF2034-4MS	1.0	1.021	69.418	2.169	69.418			10.041	32.278	170	6276.54	**194.5528**
SF2034-4MSD	1.0	0.947	64.271	2.008	64.271			10.390	32.278	170	4718.88	**147.1198**
SF2034-10	40.0	0.775	52.376	1.634	1.309			10.653	32.701	170	63.90	**1.9931**
CCV9	40.0	0.645	43.264	1.349	1.082	1.04	104.0%					
CCB	40.0	0.006	-1.189	-0.037	-0.030							

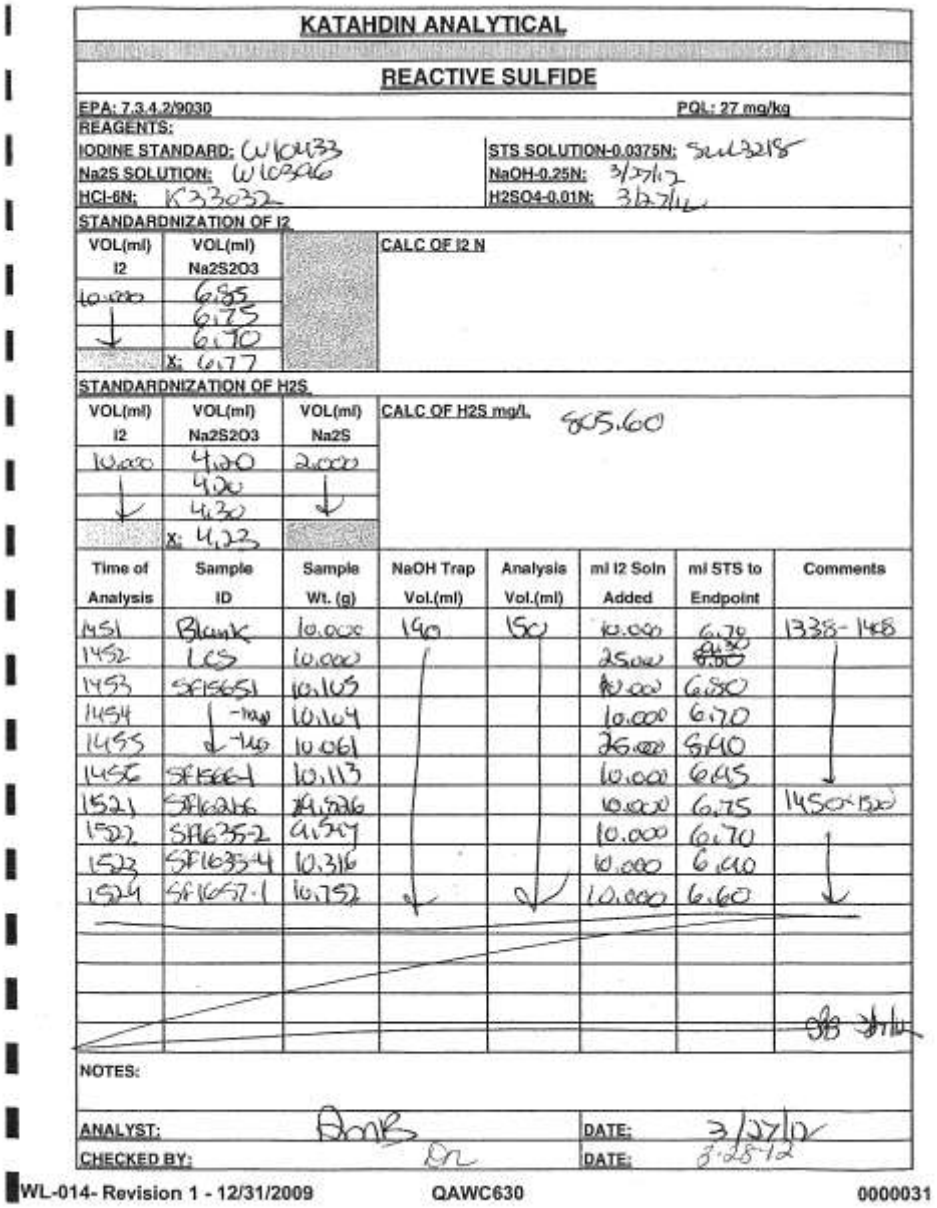
Quality Control Sample Calculations

Sample or LCS ID	Duplicate Sample ID	Matrix Spike Sample ID	Dup RPD	Spk Added to LCS (umoles/g dry)	LCS Recv (%)	Spk Added to MS (umoles/g dry)	Matrix Spike Recv (%)
LCS1				23.630	103.39%		
SF2034-4		SF2034-4MS				106.531	146.39%
SF2034-4		SF2034-4MSD				102.874	136.71%

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 3

REACTIVE SULFIDE LOGBOOK PAGE



KATAHDIN ANALYTICAL

REACTIVE SULFIDE

EPA: 7.3.4.2/9030 PQL: 27 mg/kg

REAGENTS:

IODINE STANDARD: WI04133 STS SOLUTION-0.0375N: SCL3218

Na2S SOLUTION: WI03946 NaOH-0.25N: 3/27/12

HCl-6N: K330321 H2SO4-0.01N: 3/27/12

STANDARDNIZATION OF I2

VOL(ml) I2	VOL(ml) Na2S2O3
10.000	6.85
	6.75
↓	6.70
	X: 6.77

CALC OF I2 N

STANDARDNIZATION OF H2S

VOL(ml) I2	VOL(ml) Na2S2O3	VOL(ml) Na2S
10.000	4.20	2.000
	4.20	↓
↓	4.30	
	X: 4.23	

CALC OF H2S mg/L 805.60

Time of Analysis	Sample ID	Sample Wt. (g)	NaOH Trap Vol.(ml)	Analysis Vol.(ml)	ml I2 Soln Added	ml STS to Endpoint	Comments
1451	Blank	10.000	140	150	10.000	6.70	1338-1458
1452	LCS	10.000	↓	↓	25.000	~~6.80~~ 6.90	↓
1453	SF15651	10.103			10.000	6.80	
1454	-MW	10.164			10.000	6.70	
1455	-MS	10.061			25.000	6.40	
1456	SF1661	10.113			10.000	6.65	↓
1521	SF16216	10.226			10.000	6.75	1450-1520
1522	SF16352	9.309			10.000	6.70	↓
1523	SF16334	10.316			10.000	6.60	
1524	SF16371	10.752	↓	↓	10.000	6.60	↓

NOTES:

ANALYST: DMB DATE: 3/27/12

CHECKED BY: DR DATE: 3-28-12

WL-014- Revision 1 - 12/31/2009 QAWC630 0000031

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 4

ACID VOLATILE SULFIDE BATCH SHEET

WET CHEMISTRY BATCH REPORT
Apr 24 2012, 12:52 pm
Batch: WG107282

Parameter: Acid Volatile Sulfide
Prep Date: 20-APR-12

Data Analyzed: 20-APR-12
Prep Method: EPA 821

Analyst Initials: DW
Prep Chemist: APB

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpc. DF	Result	Rpc Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SF2034-10	SAMP	EPA 821	40.000mL	40.000mL	1	1.49 2.08	2.0 umole/g	33.	.1	0.0	0.30		
SF2034-2	SAMP	EPA 821	40.000mL	40.000mL	1	.0432	00.25 umole/g	41.	.1	0.0	0.25		
SF2034-4	SAMP	EPA 821	40.000mL	40.000mL	1	5.4831	6.5 umole/g	22.	.1	0.0	0.46		
SF2034-6	SAMP	EPA 821	40.000mL	40.000mL	1	13.1902	13. umole/g	20.	.1	0.0	0.46		
SF2034-8	SAMP	EPA 821	40.000mL	40.000mL	1	.0032	00.14 umole/g	69.	.1	0.0	0.14		
WG107282-1	MBLANK	EPA 821	40.000mL	40.000mL	1	-.013	00.10 umole/g	NA	.1	0.0	0.10		
WG107282-2	LCS	EPA 821	40.000mL	40.000mL	1	24.638	25. umole/g	NA	.1	0.0	0.10		103
WG107282-3	MS	EPA 821	40.000mL	40.000mL	1	184.662	160 umole/g	NA	.1	0.0	0.45		148
WG107282-4	MSD	EPA 821	40.000mL	40.000mL	1	147.1198	150 umole/g	NA	.1	0.0	0.45	11	137

Comments:

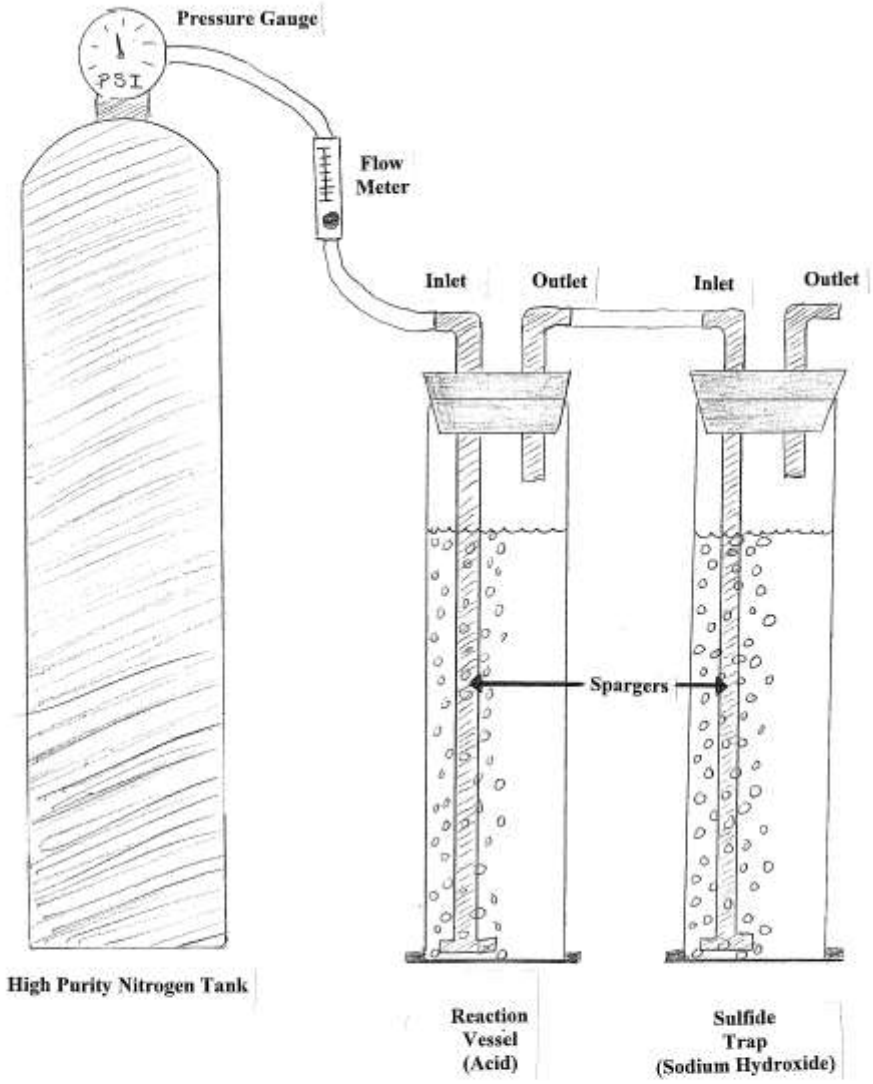
SF2034-10	DUP Use TS from -9
SF2034-2	DUP Use TS from -1
SF2034-4	DUP MS/MSD Use TS from -3
SF2034-6	DUP Use TS from -5
SF2034-8	DUP Use TS from -7
WG107282-1	SF2034-4
WG107282-2	SF2034-4
WG107282-3	SF2034-4
WG107282-4	SF2034-4

Entered by: DW Date: 4/24/12 Accepted by: [signature] Date: 04/26/12

TITLE: DETERMINATION OF ACID VOLATILE SULFIDE AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENTS

FIGURE 5

ACID VOLATILE SULFIDE APPARATUS



TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–CO_2 D

Prepared By: Betsy Carbone Date: 4/98

Approved By:

Group Supervisor: Keith Tangley Date:

Operations Manager: Jal C Burton Date: 2/2/01

QA Officer: Deborah J Nadeau Date: 2·1·01

General Manager: Donald F. Hoffman Date: 2/08/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, SM reference, database + titrant standardization. Other minor changes throughout.	DN	2·1·01	2·1·01
02	Made some changes in section 5.0 to titrants. Added low & high level LCSs to sections 5.0, 7.0 and 8.0. Removed GM references. Added new logbook page.	DN	12·05·02	12·05·02
03	Included SM4500, added KIMS entry, revised text for clarity, retitled and/or updated some figures, removed fig. 3. Minor changes throughout - added wording to Sect. 6 and 8.	LAD	031605	031605
04	Added reference for purchased IN Na_2CO_3 Added reference for CA-107 to Section 1.4	LAD	04/06	04/06
05	Fixed title on title page. Minor formatting changes throughout.	LAD	03/07	03/07

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SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Corrected LCS and MS spike amounts for both Low and High in Sect. 5 and 8. Added S.C. requirement that titrants must be replaced monthly.	LAD	06/08	06/08
07	Added definitions & updated figures with new runlog page & titrant standardization page. Added EHSM, subsampling, QA-806, DoD, NELAC and CA-101 references.	DN	08/09	08/09
08	Section 7 updated to reflect new data processing procedure - (removed printer and added desk-top instructions).	LAD	06/10	06/10
09	Sect. 9 - Added raw data archival information. Sect. 8 and Table 1 - Update LCS acceptance criteria and duplicate frequency. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added and edited references. Updated Figs 1 → 4.	LAD	05/12	05/12
10	Many edits to update SOP for new auto-titrator. Sect. 5 - changed preparing std.s every 2 weeks to monthly. Update 6B example. Changed KAS110C to KAS.	LAD	08/15	08/15
11	Sect. 8 and Table 1 - SM4500CO2 D Continuing calibration criteria of +/- 10% was added	LAD	04/17	04/17
12	Sect. 8 & Table 1 - Updated CCV and LCS acceptance criteria for method SM 2320B. Sect. 1 - LCS source independent of ICAL source.	LAD	06/17	06/17
13	Sect. 1 - Changed LCS to independant from the ICAL. Sect. 9 - Removed MDL information Sect. 10 - Updated references. Updated figures 1 → 3.	LAD	07/18	07/18

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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-739-13**, titled **Titrimetric Determination of Total Alkalinity by EPA Method 310.1 and SM 2320 B using the Mettler Dl25 Autotitrator, and Calculation of the Component Forms of Alkalinity by SM 4500–$C0_2$ D.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-739-13**, titled **Titrimetric Determination of Total Alkalinity by EPA Method 310.1 and SM 2320 B using the Mettler Dl25 Autotitrator, and Calculation of the Component Forms of Alkalinity by SM 4500–$C0_2$ D.**

Recipient: _____ Date: _____

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–$C0_2$ D

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services to determine the total alkalinity (the acid-neutralizing capacity) of an aqueous sample using a Mettler DL25 Autotitrator in accordance with EPA Method 310.1and Standard Methods 2320 B. Other forms of alkalinity (carbonate alkalinity, bicarbonate alkalinity, and caustic alkalinity) are calculated from the measured total alkalinity and pH of a sample in accordance with Standard Methods 4500-CO_2 D.

1.1 Definitions

Batch - Twenty (20) samples or fewer prepared on the same day.

ICV - Initial Calibration Verification. One ICV is read at the beginning of every run. This verifies that the calibration curve is still usable. An ICV is prepared from a separate source from the CCV and calibration curve standards.

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 laboratory reagent grade water with no reagents added. One CCB is run every ten samples.

Method Blank - A laboratory reagent grade water sample that is carried through the entire analytical procedure in the same manner as a sample.

LCS - Laboratory Control Sample. One LCS per batch is made from **a source as independent of the initial calibration verification**. The LCS determines the validity of the batch. For the purposes of this analysis, the LCS also serves as the ICV.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of alkalinity by EPA Method 310.1and Standard Methods 2320B. 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 alkalinity by EPA Method 310.1and Standard Methods 2320B 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 & 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.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical 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 titrants and spent samples have a low pH, and should be disposed of in the satellite waste container for corrosive waste (Waste Stream “A”) that is located in the Wet Chemistry laboratory. Unused standards (composed of sodium carbonate and water) are not hazardous, and may be poured down the laboratory sink.

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2.0 SUMMARY OF METHOD

A measured volume of unaltered sample (i.e., an aliquot that has not been filtered, diluted, concentrated, or otherwise altered) is titrated to two electrometrically-determined end points of pH 8.2 and 4.5. The total alkalinity of the sample as mg/L $CaCO_3$ is determined by calculation using the known normality and volume of titrant that are used to titrate the sample volume to pH 8.2 and 4.5. Component forms of alkalinity (carbonate, bicarbonate, and caustic alkalinities) are calculated from the measured total alkalinity and pH of the sample.

3.0 INTERFERENCES

Substances such as salts of weak organic and inorganic acids present in large amounts in the sample may interfere with the electrometric measurement. Soaps, oily matter, suspended solids, or precipitates may coat the glass electrode and cause a sluggish response. In this case, allow additional time between titrant additions to let the electrode come to equilibrium or clean the electrode occasionally.

Although the alkalinity of many surface waters is primarily a function of carbonate, bicarbonate, and hydroxyl ion content, the presence of borates, phosphates, silicates, or other bases may also contribute to the measured alkalinity of a sample.

4.0 APPARATUS AND MATERIALS

4.1 Mettler Toledo T50 Autotitrator, and peripherals, including a 20 position autosampler and a pH electrode

4.2 Disposable polyethylene or polypropylene autosampler cups

4.3 Analytical balance capable of weighing to 0.0001g

5.0 REAGENTS AND STANDARDS

5.1 1 N sodium carbonate (Na_2CO_3) solution – purchased standard supplied with certificate of analysis. This solution is used to prepare the Continuing Calibration Verification Standards (Section 5.8).

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5.2 Primary standard sodium carbonate (Na_2CO_3) – dried at 250°C for 4 hours, cooled, and then stored in desiccator. This standard is used prepare the Laboratory Control Sample and Matrix Spike Stock Solutions (Section 5.7).

5.3 0.05 N (approximate) Na_2CO_3 solution – Dilute 2.500 ± 0.200 g of dried Na_2CO_3 (Section 5.2) to one liter with laboratory reagent grade water. Record the exact weight of Na_2CO_3 used to 0.0001 g. Use the measured weight of Na_2CO_3 to calculate the exact normality of the solution. The calculated normality is used in subsequent calculations. This solution is used to standardize the H_2SO_4 titrants. Prepare on the day of the titration. Alternatively, 50 mL of purchased 1N aqueous Na_2CO_3 can be diluted to 1 L with laboratory reagent grade water to get a concentration of 0.05 N.

5.4 0.1 N (approximate) sulfuric acid (H_2SO_4) titrant – Dilute 2.8 mL of concentrated H_2SO_4 to one liter with laboratory reagent grade water. Standardize by potentiometric titration of 40 mL of 0.05 N Na_2CO_3 solution (Section 5.3) diluted to 50 mL with laboratory reagent grade water. Refer to Section 7.1, Standardization of Titrants. Prepared titrants must be standardized initially prior to use and restandardized at least quarterly. More frequent standardization may be required for certain states, federal programs, or clients. South Carolina requires titrants to be standardized on a monthly basis.

Note: This titrant will be referred to throughout the following discussion as "0.1 N titrant", with the understanding that the exact measured normality of the titrant is used in all calculations.

5.5 0.02 N (approximate) sulfuric acid (H_2SO_4) titrant – Dilute 200.0 mL of the 0.1 N H_2SO_4 titrant to 1 L with laboratory reagent grade water. Standardize by potentiometric titration of 15.0 mL 0.05 N Na_2CO_3 solution (Section 5.3) diluted to 50 mL with laboratory reagent grade water. Refer to Section 7.1, Standardization of Titrants. Prepared titrants must be standardized initially prior to use and restandardized at least quarterly. More frequent standardization may be required for certain states, federal programs, or clients. South Carolina requires titrants to be standardized on a monthly basis.

Note: This titrant will be referred to throughout the following discussion as "0.02 N titrant", with the understanding that the exact measured normality of the titrant is used in all calculations.

5.6 pH buffers - 4.0, 7.0, 10.0

5.7 Laboratory Control Sample (LCS) and Matrix Spike (MS) Stock Solutions –

5.7.1 For use with 0.1 N H_2SO_4 titrant (high-level MS stock): Prepare by dissolving 1.060 g dried, anhydrous Na_2CO_3 in laboratory reagent grade water and diluting to 100 mL. 1.0 mL = 10.00 mg as $CaCO_3$. The standard must be prepared every 30 days.

5.7.2 For use with 0.02 N H_2SO_4 titrant (LCS/MS stock): Prepare by dissolving 1.060 g dried, anhydrous Na_2CO_3 in laboratory-grade laboratory reagent grade water and diluting to 1 liter. 1.0 mL = 1.00 mg as $CaCO_3$. The standard must be prepared every 30 days.

5.8 Continuing Calibration Verification (CCV) Standards –

5.8.1 For use with 0.1 N H_2SO_4 titrant (high-level CCV): Prepare by diluting 12.5 mL of 1.0 N Na_2CO_3 (Section 5.1) to 500 mL with laboratory reagent grade water. The final concentration is equivalent to 1250 mg/L as $CaCO_3$. The standard must be prepared every 30 days.

5.8.2 For use with 0.02 N H_2SO_4 titrant (low-level CCV): Prepare by diluting 2.5 mL of 1.0 N Na_2CO_3 (Section 5.1) to 1 liter with laboratory reagent grade water. The final concentration is equivalent to 125 mg/L as $CaCO_3$. The standard must be prepared every 30 days.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in plastic or glass bottles, allowed to settle and stored at <6 C°, without freezing, until time of analysis.

Sample analysis must be performed within 14 days from sample collection. Samples are brought to room temperature before beginning analysis.

7.0 PROCEDURES

STANDARDIZATION OF TITRANTS (Initially & quarterly)

7.1 The titrants listed in Sections 5.4 and 5.5 must be standardized in triplicate as described. Using the amount of 0.05 N Na_2CO_3 listed in Section 5.4 or 5.5, titrate to a pH of about 5. Rinse the pH electrode into the beaker. Boil the solution gently for 3 to 5 minutes under a watch glass. Cool to room temperature. Record the pH after boiling and cooling. Rinse the watch glass into the beaker. Titrate to a pH of 4.5. Calculate the normality of H_2SO_4 as follows:

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$$N \text{ of } H_2SO_4 = \frac{A \times B}{53.00 \times C}$$

where: A = weight (g) of Na_2CO_3 used to prepare 0.05 N Na_2CO_3 solution (Section 5.3)
B = volume (mL) of 0.05 N Na_2CO_3 solution used in titration
C = volume (mL) of titrant (Section 5.4 or 5.5)

Record the standardization data for the titrants in the Alkalinity Runlog (Figure 2), and use the exact measured normality of each titrant in calculating sample alkalinities.

Note: If using purchased aqueous Na_2CO_3 to make the 0.05 N solution (section 5.3), calculate the normality of H_2SO_4 as follows:

$$N \text{ of } H_2SO_4 = \frac{0.05\ N \times B}{C}$$

where: B = volume (mL) of 0.05 N Na_2CO_3 solution used in titration
C = volume (mL) of titrant (Section 5.4 or 5.5)

INTRODUCTION

7.2 Pre-program the autotitrator to run alkalinity as “Method #1”.

Use either 0.02 N H_2SO_4 or 0.1 N H2SO4 standardized with 0.05 N Na_2CO_3 solution as the titrant. Each H_2SO_4 titrant has its own auto-burette and reservoir. The choice of the 0.1N or 0.02N titrant is based on sample history. Samples analyzed using the 0.02 N titrant may “error out” due to not reaching the pH end point of 4.5. These samples must be reanalyzed using the 0.1N titrant. Samples analyzed using the 0.1N titrant and having a total alkalinity <1000 mg/L must be reanalyzed using the lower concentration titrant.

7.3 The samples will be titrated in 0.10 mL increments to two pre-selected endpoints: 8.2 (“p value” or “phenolphthalein alkalinity”) and 4.5 (“m value” or “total alkalinity”). A pH electrode is used to detect the endpoints. The pH electrode must be calibrated before use as described below.

7.4 The autosampler has a capacity for twenty samples. Sample runs are pre-programmed in a LabX titration software which assigns a unique sample number and sample identification to each sample. A rinse cup and buffer cup will be placed in pre-selected spots on the autosampler. Consequently, a maximum of 18 samples will be able to run with those cups in place.

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7.5 Data is automatically displayed on the monitor for each sample as it is run. Following each analytical run, a sample summary sheet (Figure 3) should be opened and printed in excel.

SYSTEM START-UP

7.6 Turn on the autosampler and the computer. Open the LabX Titration software. Make sure they connect.

7.7 Remove the glass pH electrode with attached cable from its protective tubing (which contains KCl storage solution) and connect the cable to the back of the titrator console. Remove the fill cap from the electrode. Check the fluid level in the electrode and top off if required. Insert the electrode through the titrator autosampler arm. Check that the bottle containing laboratory reagent grade water for rinsing between samples is full.

CALIBRATION OF pH ELECTRODE

7.8 Place approximately 50 mL of pH 4.0, pH 7.0, and pH 10.0 buffer solution into three sample cups in positions 1- 3 of the turntable. Place a sample cup with 50 mL of DI water in positions 20 and 50 mL of pH 7 in position 19.

7.9 In the LabX titration software, select the analysis tab on the bottom right to bring up preprogrammed methods.

7.10 Select “pHcal”, right click and hit run. Alternatively you can select the “pH Calibration” icon on the bottom right in the shortcuts tab.

7.11 The instrument will read and record the pH of all the buffers. These points will be used to calibrate any samples run after.

7.12 After the run is complete, the probe will return to the home position located in cup 19.

SAMPLE ANALYSIS

7.13 Choose correct normality titrant for the test. Make sure the correct normality burret is inserted into the titrator. Low level alkalities use the 0.02 N H2SO4 burette, high level uses the 0.1 H2SO4 burette. If the wrong burette is inserted the test will not start and will display a warning.

7.14 Ensure that the titrant and rinse water reservoirs are full.

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7.15 Check burette chamber for air bubbles. To remove bubbles or flush burette, press "Rinse, 100%" icon in the shortcuts tab. This will cause the burette to empty and then refill with titrant. Be sure that there is a sample cup in the "up" position at the titration head to collect the titrant dispensed.

7.16 Place autosampler cups in turntable in the desired order. If a sample is oily or contains precipitates, or appears to contain substances which may coat the electrode as outlined in section 3.0, a "rinse" sample of water may be placed after that sample to avoid inaccurate results for the next sample.
Note: Please refer to Katahdin SOP CA-108, Basic Laboratory Technique, current revision, for more information on subsampling.

7.17 To enter sample data, select the correct method in the analysis tab, right click and select run. High levels will use the "2320HI2" method; low levels will use the "2320B2" method.

7.18 A series template screen will appear. Enter the correct number of samples being run. Select the correct start position for the run (usually cup 1). Enter in the sample ID data into the appropriate labeled number. Do not alter the other fields.

7.19 Once the entry fields are filled hit the start button.

7.20 The program will automatically run up to 18 measurements, (IE samples and QC) per run. If more measurements are needed repeat steps 7.16-7.19

7.21 Run data is automatically saved and sorted by date. Select the "Reports" tab on the bottom left of the LabX software. Click the "+" on the appropriate method, then select the corresponding date and time of the sample you wish to report. Right click and select "Export report". Save the file to the TITRATOR DATA folder by hitting the "Make New Folder" button with parent folder selected. Label the new folder with the date the samples were run. Hit ok with the new folder selected to save the file.

7.22 The autotitrator automatically calculates the total alkalinity of the sample using the following equation:

$$\text{Total Alkalinity, mg/L as CaCO3} = \frac{A \times N \times 50{,}000}{V}$$

where: A = titrant volume dispensed (mL)
N = normality of titrant
V = sample volume titrated (mL)

7.23 The components of total alkalinity (e.g. carbonate alkalinity, bicarbonate alkalinity, caustic/hydroxide alkalinity) may be calculated from the initial sample pH and the total alkalinity using the formulas listed in Figure 4. The LCS that is analyzed for total alkalinity has no reference values for these component forms of alkalinity.

7.24 For total alkalinity, carbonate alkalinity, and bicarbonate alkalinity, the measured total alkalinity and sample pH are entered manually into the Katahdin Information Management System (KIMS) for calculation and reporting. Bicarbonate and carbonate alkalinity concentrations are automatically calculated by KIMS from the entered data. For caustic (hydroxide) alkalinity, analyze the pH of the sample using the pH meter. The sample pH must first be entered into a spreadsheet for calculation which is then entered manually in KIMS. After the data is entered, a batch sheet is automatically printed out by KIMS. An example batch sheet appears as Figure 3. Refer to the current revision of Katahdin SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information on data entry and review.

7.25 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 criterion 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 judgements. 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. 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 One method blank consisting of 50 mL laboratory reagent grade water must be analyzed daily or per twenty samples, whichever is more frequent.

Acceptance criteria: < 5 mg/L as $CaCO_3$

Corrective Actions: Report results that are <PQL.
Report results that are >10X the blank level with a "B" flag

Restandardization of titrant and reanalysis of all other samples may be required.

8.2 One laboratory control sample (LCS) must be analyzed for every twenty samples or each day that the analysis is performed, whichever is more frequent. The low-level LCS is prepared by pipetting 6 mL of the LCS/MS Stock Standard into a sample cup and adding 44 mL of laboratory reagent grade water. The high-level LCS is prepared by pouring 50 mL of the LCS/MS Stock Standard into a sample cup. The true value for the low-level LCS is 120 mg/L as $CaCO_3$. The true value for the high-level LCS is 1000 mg/L as $CaCO_3$.

Acceptance Criteria: 80% - 120% of true value for USEPA Method 310.1.
85% - 115% of true value for Standard Methods 2320 B.

Corrective Actions: If LCS fails high, report results, which are <PQL.
Restandardization of titrant and reanalysis of all other samples may be required.

8.3 Continuing Calibration Verification (CCV)/Continuing Calibration Blank (CCB)- One CCV/CCB pair must be analyzed after every 10 measurements. Add 50 mL of the calibration verification solution to an autosampler cup and analyze. The true value for the high-level CCV is 1250 mg/L as $CaCO_3$. The true for the low-level is CCV is 125 mg /L as $CaCO_3$.

Acceptance Criteria: 80 - 120% of true value for 310.1.
90 - 110% of true value for 2320B
90 - 110% of true value for 4500CO2 D

Corrective Actions: If CCV fails high, report results, which are <PQL.

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–$C0_2$ D

Restandardization of titrant and reanalysis of all other samples may be required.

8.4 One sample duplicate is analyzed for every twenty samples or each day's batch, whichever is more frequent.

Acceptance Criteria: 0% - 20% RPD for concentrations > 60 mg/L for high-level and >15 mg/L for low level
0-100% RPD for concentrations < 60 mg/L for high-level and <15 mg/L for low-level

Corrective Actions: Reanalyze sample in duplicate for confirmation. If still out of criteria and no lab problem is suspected, flag sample result with appropriate notation (e.g., sample non-homogeneity).

8.5 One Matrix Spike (MS) is analyzed for every ten samples or each day's batch of samples, whichever is more frequent. The matrix spike is prepared by pipetting 6 mL of the LCS/MS Stock Standard (either high or low) into a 50-mL graduated cylinder and bringing to a final volume of 50 mL with sample. The added spike concentration for the low-level MS is 120 mg/L as $CaCO_3$. The added spike concentration for the high-level MS is 1000 mg/L as $CaCO_3$.

Acceptance Criteria: 75% - 125% recovery (USEPA Method 310.1) or 80% -120% recovery (Standard Methods 2320 B) of the added matrix spike for samples with a concentration < 4X the spike concentration. The spike recovery calculation must take into account the fact that a smaller volume of sample is used to prepare the matrix spike than was used in the original sample titration. The MS recovery is calculated using the following equation:

$$\text{Recovery (\%)} = \frac{[(\text{Sample} + \text{Spike}) - (0.88 \times \text{Sample})]}{(\text{Spike added})} \times 100$$

Corrective Action: (1) Evaluate the samples and associated QC: e.g. If the LCS results are acceptable, narrate.
(2) If both the LCS and MS are unacceptable reanalyze the samples and QC.

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.

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–$C0_2$ D

9.0 METHOD PERFORMANCE

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.

Refer to the current revisions of USEPA Method 310.1 and Standard Methods 2320 B for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method 310.1 – Alkalinity, Titrimetric (pH 4), "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, Revised March 1983.

Method 2320 B . Titration Method , "Standard Methods for the Examination of Water and Wastewater," Method 2320B, 21st Edition, 2005, American Public Health Association.

Method 4500-CO2 D. Carbon Dioxide and Forms of Alkalinity by Calculation, "Standard Methods for the Examination of Water and Wastewater," Method 2320B, 18th Edition, 1992, American Public Health Association.

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, 10/06/2010.

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–$C0_2$ D

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.

LIST OF TABLES AND FIGURES

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–CO_2 D

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of 20 or fewer samples	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	One per prep batch of 20 or fewer samples	**EPA 310.1** 80% - 120% recovery **SM 2320B** 85% - 115% recovery	(1) If the LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
CCV	One after every 10 samples	**EPA 310.1** 80% - 120% recovery **SM2320B & SM4500CO2D** 90-110% recovery	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
Matrix Spike	One MS per 10 field samples	**EPA 310.1** 75%-125% recovery **SM 2320B** 80%-120% recovery	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep the samples and QC.
Sample Duplicate	One sample duplicate per 20 field samples	RPD $\leq$20 for results > 3X PQL; RPD $\leq$ 100 for results < 3X PQL	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.
Demonstration of analyst proficiency; accuracy and precision	analysis of 4 LCSs per analyst, initially, yearly, thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
Method Detection Limit Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–$C0_2$ D

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-739-13	METHOD 310.1/SM 2320B, current revisions
Apparatus/Materials		
Reagents	Sodium carbonate solution purchased.	Sodium carbonate solution prepared from salt.
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–CO_2 D

TABLE 3

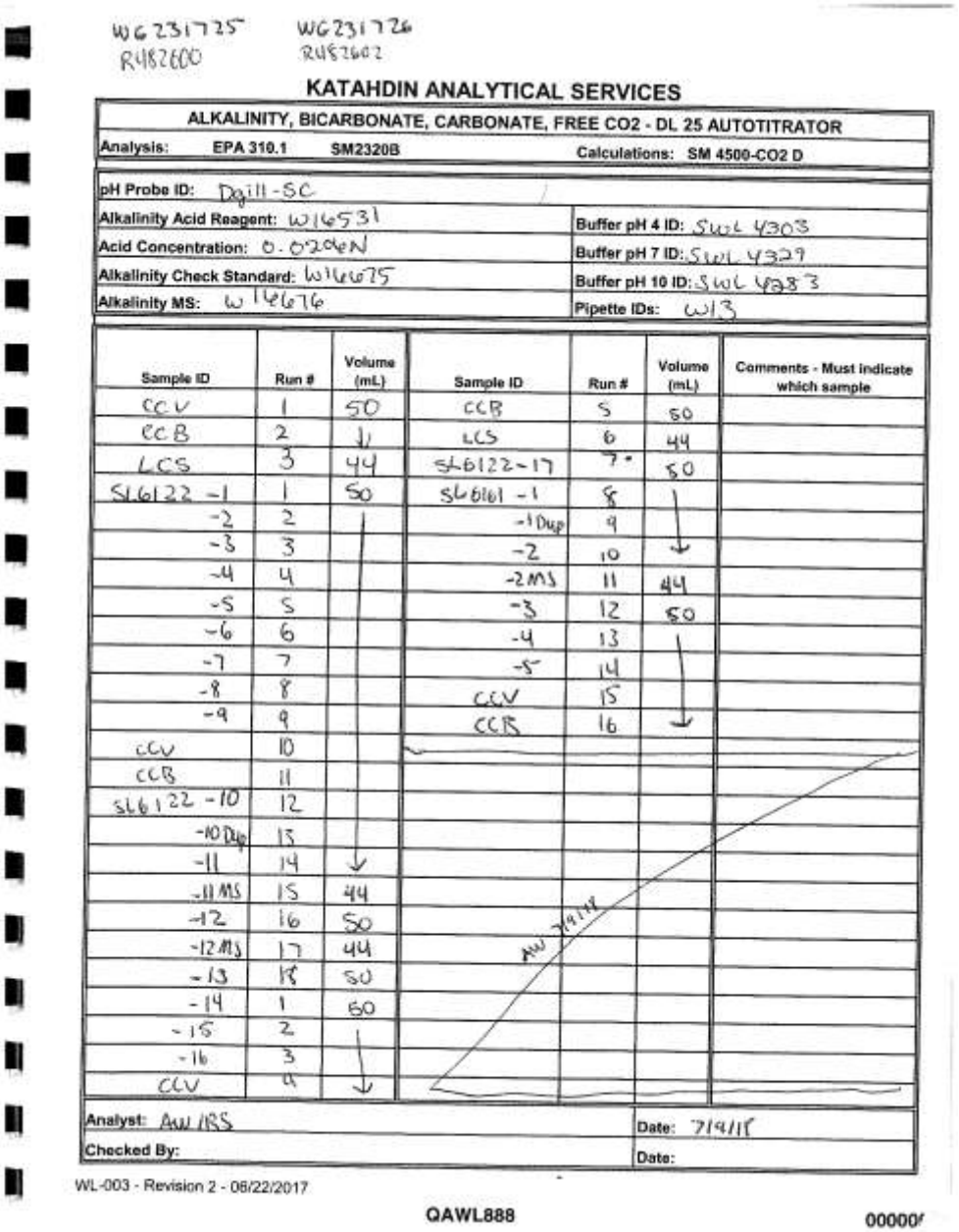
SUMMARY OF OFTEN USED INSTRUMENT COMMANDS

COMMAND	ACTION
1 - MODE	Enter changes to Method Parameters
2 - MODE	Print Method Parameters
METHOD	Display Active Method
n - METHOD	Call up Method # n
pH CALIB	pH Probe Calibration Sequence
4 pH CALIB	pH Probe Calibration Sequence
7 pH CALIB	pH Probe Calibration Sequence
3 - pH CALIB	Display pH of Active Sample
Burette/mL	Dispense Burette Contents and Refill
SERIES INPUT	Enter Series Sample
2 -SERIES INPUT	Print Series Data
9 - SERIES INPUT	Clear Series Data
7 MODE	Turntable Commands Active
7 MODE - 1	Rinse Mode - Active as long as “1” is pressed
7 MODE - 7	Move cup up or down
7 MODE - 8	Rotate Forwards 1 Position
7 MODE - 9	Rotate Backwards 1 Position

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–CO_2 D

FIGURE 1

EXAMPLE PAGE FROM ALKALINITY RUNLOG



WG231725 R482600 WG231726 R482602

KATAHDIN ANALYTICAL SERVICES

ALKALINITY, BICARBONATE, CARBONATE, FREE CO2 - DL 25 AUTOTITRATOR

Analysis: EPA 310.1 SM2320B Calculations: SM 4500-CO2 D

pH Probe ID: Dgill-SC

Alkalinity Acid Reagent: W16531 — Buffer pH 4 ID: SWL 4303

Acid Concentration: 0.0204N — Buffer pH 7 ID: SWL 4329

Alkalinity Check Standard: W166075 — Buffer pH 10 ID: SWL 4283

Alkalinity MS: W166076 — Pipette IDs: W13

Sample ID	Run #	Volume (mL)	Sample ID	Run #	Volume (mL)	Comments - Must indicate which sample
CCV	1	50	CCB	5	50	
CCB	2	↓	LCS	6	44	
LCS	3	44	SL6122-17	7	50	
SL6122-1	1	50	SL6161-1	8	↓	
-2	2	↓	-1 Dup	9		
-3	3		-2	10		
-4	4		-2 MS	11	44	
-5	5		-3	12	50	
-6	6		-4	13	↓	
-7	7		-5	14		
-8	8		CCV	15		
-9	9		CCB	16		
CCV	10					
CCB	11					
SL6122-10	12					
-10 Dup	13					
-11	14					
-11 MS	15	44				
-12	16	50				
-12 MS	17	44				
-13	18	50				
-14	1	60				
-15	2	↓				
-16	3					
CCV	4	↓				

AW 7/9/18

Analyst: AW IRS — Date: 7/9/18

Checked By: — Date:

WL-003 - Revision 2 - 06/22/2017

QAWL888

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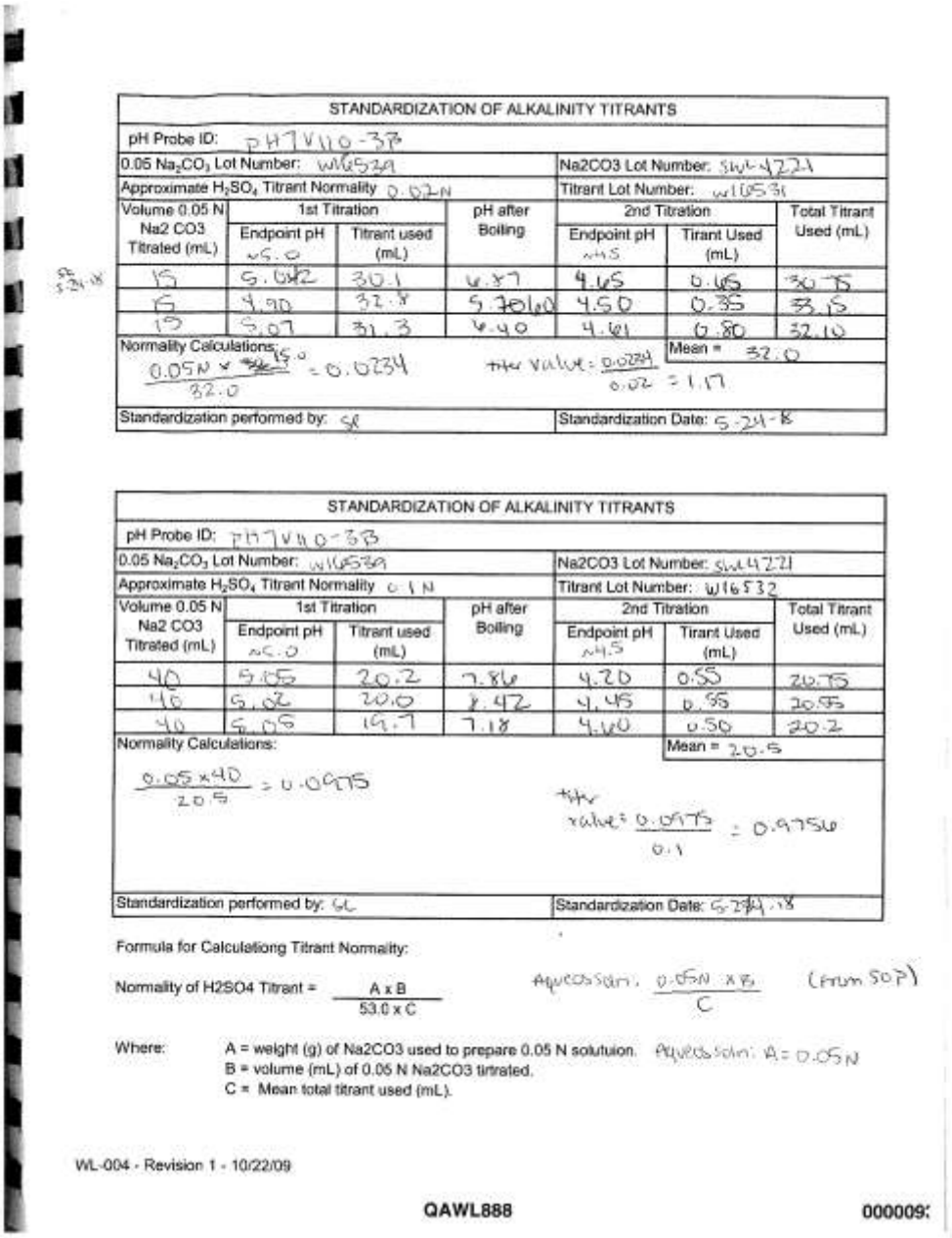
TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–CO_2 D

FIGURE 2

EXAMPLE OF STANDARDIZATION OF TITRANT



STANDARDIZATION OF ALKALINITY TITRANTS

pH Probe ID: pH7V10-38

0.05 Na_2CO_3 Lot Number: WL6529 — Na2CO3 Lot Number: SLV4721

Approximate H_2SO_4 Titrant Normality 0.02N — Titrant Lot Number: W16531

Volume 0.05 N Na2 CO3 Titrated (mL)	1st Titration		pH after Boiling	2nd Titration		Total Titrant Used (mL)
	Endpoint pH ~5.0	Titrant used (mL)		Endpoint pH ~4.5	Tirant Used (mL)	
15	5.042	30.1	6.87	4.65	0.65	30.75
15	4.90	32.8	5.70	4.50	0.35	33.15
15	5.07	31.3	6.40	4.61	0.80	32.10

Mean = 32.0

Normality Calculations: $\frac{0.05N \times 15.0}{32.0} = 0.0234$ — titer value: $\frac{0.0234}{0.02} = 1.17$

Standardization performed by: SL — Standardization Date: 5-24-18

STANDARDIZATION OF ALKALINITY TITRANTS

pH Probe ID: pH7V10-38

0.05 Na_2CO_3 Lot Number: WL6529 — Na2CO3 Lot Number: SLV4721

Approximate H_2SO_4 Titrant Normality 0.1N — Titrant Lot Number: W16532

Volume 0.05 N Na2 CO3 Titrated (mL)	1st Titration		pH after Boiling	2nd Titration		Total Titrant Used (mL)
	Endpoint pH ~5.0	Titrant used (mL)		Endpoint pH ~4.5	Tirant Used (mL)	
40	5.05	20.2	7.86	4.20	0.55	20.75
40	5.02	20.0	8.42	4.45	0.55	20.55
40	5.05	19.7	7.18	4.60	0.50	20.2

Mean = 20.5

Normality Calculations: $\frac{0.05 \times 40}{20.5} = 0.0975$ — titer value: $\frac{0.0975}{0.1} = 0.9756$

Standardization performed by: SL — Standardization Date: 5-24-18

Formula for Calculationg Titrant Normality:

Normality of H2SO4 Titrant = $\frac{A \times B}{53.0 \times C}$ — Aqueous: $\frac{0.05N \times B}{C}$ (From SOP)

Where: A = weight (g) of Na2CO3 used to prepare 0.05 N solutuion. Aqueous: A = 0.05N
B = volume (mL) of 0.05 N Na2CO3 titrated.
C = Mean total titrant used (mL).

WL-004 - Revision 1 - 10/22/09

QAWL888 000009

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–CO_2 D

FIGURE 3

EXAMPLE KIMS BATCH SHEET FOR TOTAL ALKALINITY

WET CHEMISTRY BATCH REPORT
Jul 02 2018, 10:08 am
Batch: WG231263

Parameter: Alkalinity
Prep Date: N/A
Date Analyzed: 26-JUN-18
Prep Method: N/A
Analyst Initials: AW
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	%R(%)	PQL	MDL	Adj PQL	RPD	%Rec
SL5573-2	SAMP	STGM 23208	50.0mL	50.0mL	1	15.251	15. mg/L	NA	5	0.23	5.0		
SL5573-6	SAMP	STGM 23208	50.0mL	50.0mL	1	31.922	32. mg/L	NA	5	0.23	5.0		
SL5573-8	SAMP	STGM 23208	50.0mL	50.0mL	1	53.981	54. mg/L	NA	5	0.23	5.0		
SL5613-2	SAMP	STGM 23208	50.0mL	50.0mL	1	188.031	190 mg/L	NA	5	0.23	5.0		
SL5613-4	SAMP	STGM 23208	50.0mL	50.0mL	1	249.584	250 mg/L	NA	5	0.23	5.0		
SL5611-6	SAMP	STGM 23208	50.0mL	50.0mL	1	22.193	22. mg/L	NA	5	0.23	5.0		
SL5649-1	SAMP	STGM 23208	50.0mL	50.0mL	1	145.037	140 mg/L	NA	5	0.23	5.0		
SL5649-3	SAMP	STGM 23208	50.0mL	50.0mL	1	45.291	45. mg/L	NA	5	0.23	5.0		
SL5649-4	SAMP	STGM 23208	50.0mL	50.0mL	1	54.651	55. mg/L	NA	5	0.23	5.0		
SL5649-5	SAMP	STGM 23208	50.0mL	50.0mL	1	58.57	58. mg/L	NA	5	0.23	5.0		
SL5649-6	SAMP	STGM 23208	50.0mL	50.0mL	1	251.429	250 mg/L	NA	5	0.23	5.0		
WG231263-1	MBLANK	STGM 23208	50.0mL	50.0mL	1	1.021	J1.0 mg/L	NA	5	0.23	5.0		
WG231263-2	LCS	STGM 23208	50.0mL	50.0mL	1	122.932	120 mg/L	NA	5	0.23	5.0		102
WG231263-3	DUP	STGM 23208	50.0mL	50.0mL	1	54.3	54. mg/L	NA	5	0.23	5.0	0	
WG231263-4	MS	STGM 23208	50.0mL	50.0mL	1	286.615	290 mg/L	NA	5	0.23	5.0		101
WG231263-5	MS	STGM 23208	50.0mL	50.0mL	1	337.785	340 mg/L	NA	5	0.23	5.0		98

Comments:

WG231263-1 SL5573-8
WG231263-2 SL5573-8
WG231263-3 SL5573-8
WG231263-4 SL5613-2
WG231263-5 SL5611-6

Entered by: AW Date: 7/2/18 Accepted by: [signature] Date: 07/02/18

TITLE: TITRIMETRIC DETERMINATION OF TOTAL ALKALINITY BY EPA METHOD 310.1 AND SM 2320 B USING THE METTLER DL25 AUTOTITRATOR, AND CALCULATION OF THE COMPONENT FORMS OF ALKALINITY BY SM 4500–$C0_2$ D

FIGURE 4

FORMULAS FOR CALCULATION OF FORMS OF ALKALINITY

A. Bicarbonate Alkalinity

$$HCO_3^- \text{ (mg/L as } CaCO_3) = \frac{T - 5 \times 10^{(pH - 10)}}{1 + 0.94 \times 10^{(pH - 10)}}$$

Where T = Total Alkalinity, mg/L as $CaCO_3$

B. Carbonate Alkalinity

$$CO_3^{2-} \text{ (mg/L as } CaCO_3) = 0.94 \times B \times 10^{(pH - 10)}$$

Where B = Bicarbonate Alkalinity, from A

C. Hydroxide (Caustic) Alkalinity

$$OH^- \text{ (mg/L as } CaCO_3) = 5 \times 10^{(pH - 10)}$$

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: Jol C. Butler 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 - rewrote 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. 9 - 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^{o} 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^{o}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^{o}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^{o}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

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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.\

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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

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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

LIST OF TABLES AND FIGURES

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.		

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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		

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FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

R447211
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: WI16776	TV: 16000ug	Calibration Date:	8/18/18
CCV Spike Source ID / Compound: WI16817	TV: 1	Calibration Analyst:	SC
MS Spike Source ID / Compound: WI16776	TV: 4000 ug	Calibration Source ID:	WI16817
Acid Lot Number: WI16673		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	272.0	Wet (Dry)	
-8	273.5	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

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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:255.1 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 03:35:06 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	SG0488-2	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

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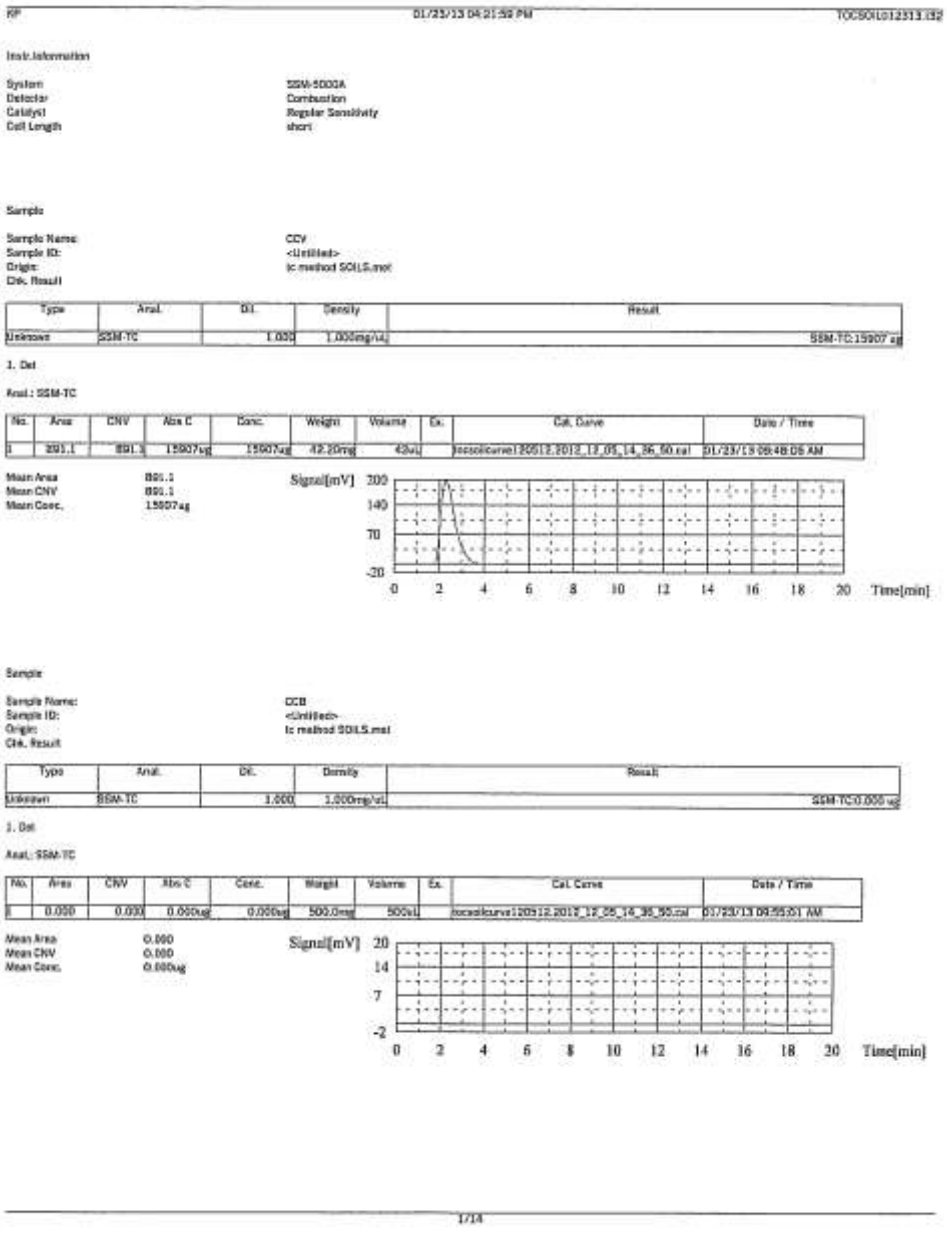
FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT



RP 01/23/13 04:21:58 PM TOC80\LO12313.C02

Instr.Information

System SSM-5000A
Detector Combustion
Catalyst Regular Sensitivity
Cell Length short

Sample

Sample Name: CCV
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:15907 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	891.1	891.1	15907ug	15907ug	42.20mg	42uL		tocsolicurve120312.2012_12_05_14_36_50.cal	01/23/13 09:48:05 AM

Mean Area 891.1
Mean CNV 891.1
Mean Conc. 15907ug

Sample

Sample Name: CCB
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:0.000 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	0.000	0.000	0.000ug	0.000ug	500.0mg	500uL		tocsolicurve120312.2012_12_05_14_36_50.cal	01/23/13 09:55:01 AM

Mean Area 0.000
Mean CNV 0.000
Mean Conc. 0.000ug

1/14

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, 05:05 am
Batch: WG115373

Parameter: TOC In Soil
Prep Date: N/A

Date Analyzed: 23-JAN-13
Prep Method: N/A

Analyst Initials: XP
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SG0443-1	SAMP	SW846 MD06D	141.9mg	200.00ug	1	289215.35693	290000 ug/gdrywt	33.	600	260	1200		
SG0473-1	SAMP	SW846 MD06D	315.4mg	200.00ug	1	414.9499	0500 ug/gdrywt	89.	400	100	500		
WG115373-1	MBLANK	SW846 MD06D	500mg	200.00ug	1	0	0300 ug/gdrywt	NA	400	85.	400		
WG115373-2	LCS	SW846 MD06D	42mg	200.00ug	1	371788.71438	370000 ug/gdrywt	NA	600	85.	400		93
WG115373-3	LCSD	SW846 MD06D	40.6mg	200.00ug	1	375714.28571	380000 ug/gdrywt	NA	600	85.	400	1	96
WG115373-4	MS	SW846 MD06D	264.9mg	200.00ug	1	21537.48061	22000 ug/gdrywt	NA	400	100	500		132
WG115373-5	MSD	SW846 MD06D	282.7mg	200.00ug	1	26092.56574	26000 ug/gdrywt	NA	400	100	500		116

Comments:

SG0473-1	MS/MSD: RE15303-SOIL-011413-726-727
WG115373-1	SG0472-1
WG115373-2	SG0473-1
WG115373-3	SG0473-1
WG115373-4	SG0473-1
WG115373-5	SG0473-1

Entered by: [signature] 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.

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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.

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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).

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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.

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-742
Revision History
Cover Page
Page 1

TITLE: **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056**

Prepared By: Deborah McGrath Date: 05/00

Approved By:

Group Supervisor: Keith Tanguay Date: 05/24/01

Operations Manager: [signature] Date: 5/23/01

QA Officer: Deborah J. Nadeau Date: 5.22.01

General Manager: Deborah J. Nadeau Date: 5/24/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02	Format changes, added pollution prevention, added SW846 method 9056 reference and requirements. Updated logbook attachments.	DN	5.22.01	
03	Added reference to KIMS data entry (Sect. 7.14). Replaced fig.s 2 & 5. added wording to sect. 8. minor changes throughout to reflect current practices	LAD	031805	031805
04	Added information on Retention time Windows. Added reference for SOP CA 107 to Section 1.4	LAD	04/06	04/06
05	Sect. 5.0 - edited to reflect current Std. prep procedures. Sect. 7.0 - changed base range, added syringe pursing procedure, added info for setting up calibration in active sequence, added need for new sequence at beginning of every month	LAD	06/08	06/08
06	Minor revisions to sections 1.1 and 4 for clarity. Revisions to Section 8, 10, Table 2 and 3 to update from SW846 method 9056 to 9056A. Added LLQC criteria to Section 8.	LAD	02/09	02/09

TITLE: **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Sect. 1.1- Added definitions. Sect. 4.1- Added computer and software references. Sect. 8, Table 1 and Table 2 - added DoDQSM requirements. Sect. 10. Added references.	LAD	08/09	08/09
08	Added creating and viewing audit log for any result or method file which has been edited. Added Table 2- DoDQSM QC Criteria.	LAD	10/10	10/10
09	Sect. 4, 5 and 7 - updated throughout for new instrument and the addition of Fluoride. Sect. 9- Added MDL, LOD and LOQ information. Sect. 10- Added & edited references. Updated Fig. 1→3 & 5 (renamed 4). Removed Fig. 4 & 6 (Turbochrom examples).	LAD	06/12	06/12
10	Sect. 5 - updated Std. concs. Sect. 7 - Removed information relating to old instrument. Table 3 - Added DODQSM 5.0 QC criteria. Renumbered subsequent Tables. Changed KASINC to KAS throughout.	LAD	08/15	08/15
11	Sect. 4 - minor changes for ICS-2100 instrumentation. Sect. 7 - Updates throughout for the ICS2100 instrumentation. Sect. 8 - Added contingency plan. Sect. 10 - updated references. Removed Table 2 (QSM 4.2)	LAD	12/17	12/17

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

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-742-11,** titled **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services technical personnel to determine the concentration of the following inorganic anions using ion chromatography (IC) by EPA Method 300.0 and SW-846 9056, current version: Sulfate, Bromide, Phosphate-P, Chloride, Fluoride, Nitrate-N, and Nitrite-N. This method may be used for the analysis of the following matrices: Drinking water, Surface water, Mixed Domestic and Industrial Waste waters, Ground water, Reagent waters, solids (after aqueous extraction), and Leachates (when no acetic acid is used).

1.1 Definitions

Analytical Batch – A group of 20 or fewer samples that are analyzed together on the same day.

Calibration Blank (CB) - A volume of laboratory reagent grade water fortified with the same matrix as the calibration standards but without the analytes. In most cases the CB will consist of laboratory reagent grade water.

Continuing Calibration Blank (CCB) – An aliquot of reagent water that is analyzed after each CCV to ensure continuing calibration accuracy.

Continuing Calibration Verification (CCV) – A midrange standard containing all method analytes that is run at the beginning of each run, after every 10 samples, and at the end of each run to ensure continuing calibration accuracy. The CCV is prepared from the same source as the calibration standards. The CCV is sometimes called the Instrument Performance Check Solution (IPC).

Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV) – An aliquot of reagent water to which known amounts of the method analytes are added, and that is processed through the entire analytical procedure in the same manner as a sample. One LCS is processed and analyzed with each batch of 20 or fewer samples. The LCS/ICV is prepared from a different standard source than the calibration standards. LCSs provide a sample of known concentration to assess the accuracy of the analytical system, and when analyzed in duplicate may be used to a measure of precision for the analytical system. The LCS/ICV is sometimes called the Laboratory Fortified Blank (LFB).

Laboratory Duplicate – A duplicate is a second aliquot of a sample that is analyzed to to assess the precision of the analysis.

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%.

Matrix Spike (MS) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for the background concentrations. The MS is sometimes called the Laboratory Fortified Matrix (LFM).

Method Blank (MB) – An aliquot of reagent water that is carried through the entire analytical procedure in the same manner as a sample. One method blank is processed and analyzed with each batch of 20 or fewer samples.

Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

Practical Quantitation Limit (PQL) – 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 anions by IC using EPA Method 300.0 and SW-846 9056. 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 anions by IC using EPA Method 300.0 or SW-846 9056 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab 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 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.

The outflow from the chromatograph and the autosampler is collected in a single container and disposed of in a Satellite Waste “N-HI” Acid for proper disposal in main waste area “A”. Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and 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 lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, analytical column, suppressor device and conductivity detector. An aqueous extraction procedure must be performed in order to utilize this method for solid matrices.

3.0 INTERFERENCES

3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems. The most common source of interference is high chloride concentrations.

3.2 The water dip or negative peak that elutes near and can interfere with the chloride peak can usually be eliminated by the adding the equivalent of 1mL of eluent concentrate to 100 mL of each sample.

3.3 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

3.4 Method interferences may be caused by contaminants in the water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in the ion chromatograph.

3.5 Samples that contain particles larger than 0.45 μm and reagent solutions that contain particles larger than 0.2 μm require filtration to prevent damage to instrument columns and flow systems.

3.6 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations above 1.5 mg/L fluoride this interference may not be significant.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph capable of delivering 1 to 5 mL of eluent per minute at a pressure of 1000 to 4000 psi. The chromatograph is controlled with a Windows-based PC running Chromeleon software. The chromatograph must be equipped with an injection valve, a 10- to 100-uL sample loop, and set up with the following components.

4.1.1 Autosampler – Dionex Model AS-DV

4.1.2 Ion chromatography system – Dionex ICS-2100 with conductivity detection

4.1.3 Precolumn – A guard column placed before the separator column to protect the separator column from fouling by particulates or organic constituents. Dionex Guard Cartridge AG18 or equivalent.

4.1.4 Separator column – A column packed with an anion exchange resin, suitable for resolving the anions of interest. Dionex Ionpac AS18 or equivalent.

4.1.5 Conductivity suppressor – An ion exchange-based device that is capable of converting the eluent and separated anions into their respective acid forms. Dionex Micromembrane Suppressor Model ASRS 300 or equivalent.

4.2 Analytical balance capable of accurately weighing to the nearest 0.0001g.

4.3 2.0 mL sample vials

4.4 Fisherbrand pipets, assorted volumes

4.5 Class "A" volumetric flasks, assorted volumes

4.6 Chromeleon software

5.0 REAGENTS AND STANDARDS

5.1 Eluent Generator Cartridge, Potassium Hydroxide

5.2 Laboratory reagent water

5.3 Stock Standard Solutions: Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade material. All purchased standards prepared from high purity salts and supplied by the vendors with certificates of purity and analysis. All purchased stock standards are given an expiration date as indicated by the manufacturer.

5.3.1 Bromide (Br^-), 1000 mg/L, purchased.

5.3.2 Chloride (Cl^-), 1000 mg/L, purchased

5.3.3 Nitrate (NO_3^--N), 225.9 mg/L as N (1000 mg/L as NO_3), purchased

5.3.4 Nitrite (NO_2^--N), 304.4 mg/L as N (1000 mg/L as NO_2), purchased

5.3.5 Phosphate (PO_4^{-3}-P), 1000 mg/L as P, purchased

5.3.6 Sulfate (SO_4^{-2}), 1000 mg/L, purchased

5.3.7 Fluoride (F^-), 1000 mg/L, purchased

5.4 Initial Calibration Stock Standard Mix- Combine the following and dilute to 200 mL with laboratory reagent grade water:

Analyte	Amount of Stock Std. Added (mL)	Concen-tration in Stock Std. (mg/L)	Final Volume of Primary Mixed Cal. Std. (mL)	Final Conc. In Primary Mixed Cal. Std. (mg/L)
Cl^-	2.0	1000	200	10.0
NO_2^--N	2.63	304.4		4.0
NO_3^--N	3.54	225.9		4.0
Br^-	4.0	1000		20.0
SO_4^{-2}	4.0	1000		20.0
PO_4^{-3}-P	1.0	1000		5.0
F^-	1.0	1000		5.0

NOTE: At any time a stock may be prepared with an abbreviated list of analytes and should be clearly labeled with the list of analytes contained.

5.5 Initial Calibration Working Standards – Dilute Initial Calibration Stock Standard as follows to prepare the five-point level working calibration standards:

Work-ing Std. ID	Amount of Primary Mixed Cal. Std. Added (mL)	Final Volume of Working Cal. Std. (mL)	Analyte Conc. In Working Cal. Std. (mg/L)						
			Cl^-	F^-	NO_2^- as N	NO_3^- as N	Br^-	SO_4^{-2}	PO_4^{-3} as P
#6	1	1	10	5	4	4	20	20	5
#5	0.5	1	5	2.5	2	2	10	10	2.5
#4	0.25	1	2.5	1.25	1	1	5	5	1.25
#3	0.1	1	1	0.5	0.4	0.4	2	2	0.5
#2	0.01	1	.1	0.05	0.04	0.04	0.2	0.2	0.05

Note: Standard #1 is the calibration blank

5.6 Continuing Calibration Verification Standard (CCV) – The CCV will be prepared using the same stock as the calibration standards and will be prepared at the same concentration as calibration standard #5.

5.7 LCS/MS Stock Standard – LCS/MS Stock must be comprised of independent sources for all analytes relative to the calibration standard stock. Combine the following using purchased standards from second source and dilute to 200 mL with laboratory reagent grade water: 7.5 mL of standard solution “A” and 7.5 mL of standard solution “B”.

5.7.1 Standard Solution "A"- Multi-element standard which contains analytes in the following concentrations:

- Nitrate (as N) – 225.9 mg/L
- Bromide- 1000 mg/L
- Orthophosphate (as P)- 326.1 mg/L
- Chloride – 1000 mg/L
- Fluoride – 1000 mg/L
- Sulfate- 1000 mg/L

5.7.2 Standard Solution "B" – Contains Nitrite (as N) only in the concentration of 304.4 mg

5.8 LCS Working Standard – The Working LCS is made by adding 0.05 mL of LCS Stock Standard to 0.950 mL of DI water for a final volume of 1.00 mL. This will yield analyte concentrations as follows:

F	Cl	NO2 (as N)	NO3 (as N)	Br	SO4	PO4 (as P)
3.75	3.75	1.14	.845	3.75	3.75	1.22

5.9 Matrix Spiked Sample – A spiked sample aliquot is prepared by adding 0.05 mL of Stock Standard (5.9) to 0.950 mL of sample for a final volume of 1.00 mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean glass or polyethylene bottles. Sample preservation and holding times for the anions in water and soil are:

ANALYTE	PRESERVATION	HOLDING TIME
Bromide	None required	28 days
Chloride	None required	28 days
Nitrate-N	Cool to 4^{o}C	48 hrs
Nitrite-N	Cool to 4^{o}C	48 hrs
Ortho-Phosphate-P	Cool to 4^{o}C	48 hrs
Sulfate	Cool to 4^{o}C	28 days
Fluoride	Cool to 4^{o}C	28 days

NOTE: Due to the potential of and persistence of chloride contamination and the associated short hold times for nitrite, nitrate and ortho-phosphate, it is recommended that these ions are run concurrently by alternate methods.

7.0 PROCEDURES

7.1 SAMPLE PREPARATION

Refer to Katahdin Analytical Services SOP CA-106, "Basic Laboratory Technique", current revision for information on sub-sampling.

7.1.1 Solid Samples Extraction – Add an amount of laboratory reagent grade water equal to ten times the weight of the dry sample. This slurry is mixed for ten minutes using a rotary extractor. The resulting slurry is allowed to stand or may be centrifuged prior to filtration through 0.45 μm filter.

7.2 TURNING ON INSTRUMENT

7.2.1 The instrument is configured as illustrated in Figure 1.

7.2.2 Open the chromeleon software

7.2.3 Click on the instrument tab in the lower left corner.

7.2.4 Click "ON" buttons for pump, Eluent Generator, CR-TC, then suppressor, in that order. The eluent should be set at 30mM, the suppressor should be set at 75 mA, and the flow rate should be set at 1.0 mL/min. The eluent fill level should be adjusted whenever water is added to the bottle. The column heater should be set at 30^{o} C. The run time is set in the instrument method for 13 min.

Note: If the water has been changed or the instrument has been off for a few days, the pump should be primed before turning the instrument on for the day. This is done by clicking the prime button on the instrument panel, opening the waste valve, and then clicking OK at the top of the screen. Allow to prime for five minutes or so before clicking the off button and closing the valve.

7.2.5 Monitor and record the backpressure on the HPLC pump. The pressure should
not exceed 3000 psi. If this occurs it would indicate the need to replace or clean the column, see column care and maintenance in the column manual.

7.3 CALIBRATION

7.3.1 A new calibration curve must be prepared at least one time every 3 months or if one of the following occurs:

7.3.1.1 The daily calibration verification is outside of the method criteria (Refer to section 8.0).

7.3.1.2 Major maintenance has been performed on the instrument (Refer to maintenance log, Figure 2).

NOTE: If one of the above is true, a new calibration curve must be prepared even if it has been less than 3 months since the last calibration.

7.3.2 If a calibration curve is required, prepare the standards as described in section 5.0. Pipet 1 mL of each standard into the autosampler cups.

7.3.3 Record the standards in the runlog (Figure 3) and load onto the autosampler. Load samples into the Dionex autosampler.

7.3.4 To start a new sequence, open an old anion sequence in chromeleon by clicking on the data tab and double clicking the desired sequence. Old samples can be deleted by row. Click “save as” and use that day’s date as the sequence name. When entering the calibration points into the sample sequence, change the sample type to “calibration standard” and type the calibrator names in the level column. Two method blanks that will not be reported should be analyzed prior to calibration or sample run to ensure that there is no contamination present in the system. The instrument method and the processing method should not need to be changed. The volume column should read 200uL for all samples. The “fill down” button can be clicked to renumber the position column. Resave the sequence and click the start button at the top of the screen.

7.3.5 When the calibration is finished, the calibration will need to be updated in the processing method. This is done by double clicking on the processing method on the bottom of the data screen in the associated items table. The calibration tab should already be selected. Click the browse button in the global calibration settings box. Double click the desired sequence, then click update. Save the processing method. The chromatograms for the entire sequence can be printed by double clicking on any chromatogram in the ECD_1 column and clicking on report designer in the lower left corner. Click on the integration tab at the bottom of the screen, then click the chromeleon icon in the upper left to print. Click print and check apply to current sequence. Click OK.

7.3.6 To print out all of the calibration curve graphs highlight all of the points in the sequence and right click to select print. Click the button next to report template and select the method folder. Double click on “test” and deselect all but the calibration box. Click OK to print.

7.3.7 The acceptance criteria for the calibration is a 0.995 correlation coefficient for the full curve. Also, the standard at or below the PQL must be within 50% of the true value (for SW 9056A only). It is important to compare curves and responses vs. standard concentration with those previously generated to insure quality of data. Significant changes should be investigated. Switching to

fresh standards or fresh reagents may change response slightly. Check historical data. Operator discretion in approving or rejecting calibrations is encouraged. The decision and rationale should be recorded in the logbook.

7.3.8 Retention time windows should be established when a new column is installed; if there is a change in instrument conditions or at least annually if there are no changes. Retention time windows are established using +/- three times the standard deviation of the retention times of standards run over the course of one day. The experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.4 LOADING SAMPLES

7.4.1 Write sequence in IC Run Log; follow page format and proper sample coding.

7.4.2 If high sample concentrations are suspected, steps should be taken to minimize reruns and protect the system from contamination and/or carryover. In general samples from potable sources, drinking water samples, may be analyzed at an as received concentration. Monitoring wells, leachates, and estuarine samples may have extremely high concentrations of chloride and/or sulfate. To avoid contaminating the system, analyze these samples at an initial dilution. In the event that the analyzed aliquot does have higher concentrations, inject water samples after the sample to clean out the system. For highly concentrated samples it may take as many as 5 or more water injections to clean out the injector and remove the carryover.

7.4.3 It is recommended that the tray is initially set up to run the opening QC before loading samples. Opening QC consists of a CCV, CCB and LCS. Evaluate the opening QC to ensure adequate separation, good chromatography, acceptable recovery as it relates to the calibration, and clean blanks. If the opening QC is within criteria and the chromatographic system is in control proceed to load samples. If QC criteria are not met or chromatography is not acceptable initiate appropriate corrective action.

7.4.4 To automatically run tray:

- Enter samples into the sequence, filling in all dilutions and positions.
- Make sure the injection volume is 25 uL
- Click the “save” icon and press start. Note: if some samples have already been run, you will need to click “remove” and then “resume” at the top of the screen to continue run.

7.5 SHUT DOWN PROCEDURE – **It is CRITICAL to explicitly follow long term shutdown storage for columns to prevent damage.**

7.5.1 Turn off the instrument in the reverse order from how it was turned on.

7.5.2 **If the system will not be used for more than a week it is critical to fill the columns with the appropriate storage solution and cap them to prevent evaporation. If a column dries out it is most likely useless.**

7.5.3 **It is critical to fill the suppressor with fresh regenerant and cap it. If the suppressor dries out it is likely to be ruined.**

7.5.4 It is best to run the instrument for a little while every couple of days to keep everything hydrated.

DATA ANALYSIS, CALCULATIONS & REPORTING

7.6 If a sample is run and the analyte of interest concentration is above the calibration range the sample must be diluted and reanalyzed. Multiple dilutions may be required to obtain results for all analytes of interest in the calibration range. For samples run at multiple dilutions, the analysis of greatest concentration, qualified retention time, good peak shape, and satisfactory resolution should be quantitated and reported. However, all dilutions should be assessed comparing the consistency of the determinations and possible matrix effects. In certain instances, the more diluted analysis may be the more appropriate reported result.

7.7 If a sample is run at dilution and the concentration of the analyte of interest is below the PQL the sample should be reanalyzed at a greater concentration. Where there is coelution of higher relative concentration analytes to adjacent analytes, e.g. chloride to nitrite, a sufficient dilution of the sample should be analyzed to maximize the resolution of the two analytes and report the affected analyte concentration at an elevated PQL narrated to that effect.

7.8 If the chromatogram fails to produce adequate resolution, or if the identification of specific anions is questionable, the sample may be spiked with an appropriate amount of standard and reanalyzed. In some instances dilution of the sample may provide sufficient resolution for identification and quantitation.

NOTE: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

7.9 Calculations are performed by Chromeleon using responses measured during analysis of the calibration standards for the operating curve that has been calculated based upon a linear regression formula. Individual calibration curves are calculated for each detector. The analyst must assure that the method file is calculating against the appropriate curve. Aqueous and soil sample analyte concentrations are calculated using the following equation:

$$A = (mR + B) * DF$$

where: *A* = analyte concentration mg/L
m = slope
R = response in peak area or height
B = y intercept
DF = dilution factor prior to analysis

NOTE: In order for the dilution factor to be applied during calculation by Chromeleon it must be entered at the time the sequence is entered and/or edited.

7.10 In the event that the software interpretation of integration is not appropriate manual integration may be performed. Refer to the Chromeleon software manual for integration techniques. It is expected that the same sound technical judgments and assessments will be equally applied to both samples and standards in the review of integrations and the decisions to perform or not perform manual integrations. In accordance with Section 7.7 of the Katahdin Analytical Services Quality Assurance Manual, any manual integration must be initialed and dated by both the analyst performing the integration and by the reviewer. **Under no circumstances is the original software generated result file to be overwritten with a manually edited file. Both the original software generated integration and the manual integration must be preserved with the raw data.** The analyst should rarely be required to manually integrate any QC elements. This is usually indicative of poor system performance and corrective action should be taken through proper maintenance.

7.11 When analyzing samples using the Ion Chromatograph in the Wet Chemistry Laboratory, the audit log function must be used in order to electronically document the integrity of the data on this instrument. When a change is made to a method or sample file, the user must save the file with the same file number appended with the next letter (i.e., a, b, c, etc.). The audit log for each sample is always available. Click on the sample and then click on the audit trail tab at the bottom of the screen. This will show every step that the sample has gone through.

7.12 The final raw data report will include information on initial and continuing calibrations and results of all quality control data. The instrument printout includes Result File Name, Calibration File Name, Sample #, Analyst, Date Analyzed, Ion Initial Volume, Dilution Factor, Observed Concentration, Reported Concentration, % Recovery.

7.13 Sample preparation information is entered manually into the Katahdin Information Management System (KIMS). Instrument data files are then imported electronically into KIMS for calculation and reporting of sample results and quality control data. Refer to the current revision of SOP CA-762 (“W et Chemistry Data Entry and Review Using Katahdin Information Management System”) for further information.

7.14 A batch sheet is generated (Figure 4). Raw data and batch sheets are reviewed for completeness and accuracy by the Department Manager or other qualified designee.

7.15 Printouts of instrument calibrations and sample data are filed in the lab for approximately 3 months for reference by analysts. Prior calibrations are archived and all are available in the Chromeleon database.

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, 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.

Table 2 is a summary of the QC criteria for work following DoD QSM version 5.0/5.1.

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 Initial Instrument Calibration – Instrument calibration, which is generated using a blank the five standards listed in Section 5.6, is performed at least once every three months or whenever there is a significant change in instrument operating conditions or hardware. One of the calibration standards must be at or below the Practical Quantitation Limit for each analyte. The curve fit is accomplished using least squares linear regression, and the correlation coefficient for the curve must be at least 0.995. Sample results that exceed the calibration range of the instrument may not be reported; the sample must be diluted and reanalyzed until the measured concentration of the analyte is within the calibration range. Because calibration linearity is established each time the instrument is calibrated (at least every three

months) and because sample results that fall outside the calibration range may not be reported, a separate linear calibration range study is not performed.

8.2 Lower Limit of Quantitation - The laboratory should establish the LLOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard prepared at the LLOQ concentration levels or use of the LLOQs as the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LLOQ recoveries must be within 50% of the true values to verify the data reporting limit.

8.3 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) – Ongoing calibration accuracy is verified by analyzing a CCV standard (a mid-range check standard) and a CCB at the beginning of each run, after every ten samples, and at the end of each run. The recovery of each CCV must be within 90% - 110% of the true value for each analyte. The measured concentration of each analyte in the CCB must be below the Practical Quantitation Limit for the analyte. If a CCV or CCB fails, the analysis must be stopped, the problem corrected, and the previous ten samples must be reanalyzed, with the following exception. If one or both CCVs bracketing a sample result are biased high and the sample concentration is <PQL, the sample result may be reported. CCVs or CCBs that are biased high may be indicative of carryover or contamination in the system by high concentration samples.

NOTE: High bias for chloride may be indicative of chloride contamination in the injector. If the run is attended and chloride is an analyte of interest, halt the run and take corrective action if bias is observed.

8.4 Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV) – One LCS/ICV, prepared from a separate standard source from the Initial Instrument Calibration, must be analyzed with each batch of 20 or fewer samples. LCS/ICV recovery acceptance limits are 90% - 110% for EPA Method 300.0 and 80% - 120% for SW846 Method 9056.

For DoD QSM 5.0, use QC acceptance criteria specified by DoD, if available). Otherwise use in-house control limits.

8.5 Method Blank (MB) – A method blank consisting of reagent water that filtered in the same fashion as the associated samples must be analyted with each batch of 20 or fewer samples. The measured concentration of each analyte in the MB must be less than the PQL (for DoD QSM, no analyte may be detected in the MB at a concentration greater than ½ PQL or greater than 1/10 the amount measured in any sample). In the instance where there is a value in excess of the CCB, the filter source should be suspected as contributing contamination. Repeat the analysis with additional cartridges.

8.6 Matrix Spike (MS) Sample – Matrix spike samples must be prepared and analyzed at a frequency of 10% (one sample in 10) for EPA Method 300.0 or 5% (one sample in 20) for SW846 Method 9056A. Matrix spikes is prepared by adding 0.05 mL of Primary Matrix Spike Mixed Standard (Section 5.9) to 1.0 mL of the filtered sample prior to loading. If the concentration of the spike is less than 25% of the native sample concentration the matrix spike recovery should not be calculated. The matrix spike recovery acceptance limits are 90% - 110% for EPA Method 300.0 and 80% - 120% for SW846 Method 9056A. If the recovery of any analyte falls outside the criteria range and the LCS and CCVs are within criteria, the poor recovery should be attributed to sample matrix.

For DoD QSM 5.0/5.1, use QC acceptance criteria specified by DoD, if available). Otherwise use in-house control limits.

8.7 Matrix Spike Duplicate (MSD) – Prepared at a frequency of one per 20 samples. Acceptance limits are 80% - 120% recovery and ≤15% RPD.

8.8 Laboratory Duplicate (Dup) - One duplicate sample must be analyzed with each batch of 20 or fewer samples (one per 10 samples for DoD QSM). Analytes with measured values $\geq$ 5 times the PQL should achieve duplicate/MSD sample precision of $\leq$ 20% RPD (≤10% for DoD QSM).

8.9 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 revisions of the applicable methods for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Methods for the Determination of Inorganic Substances in Environmental Samples", EPA - 600/R - 93 - 100, Method 300.0, Revision 2.1, August 1993

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 9056A.

Katahdin SOP CA-101, Equipment Maintenance and Toubleshooting, 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.

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, 10/06/2010.

Waters 510 HPLC Pump Manual

Dionex 200i Manual

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

LIST OF TABLES AND FIGURES

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Anions by Ion Chromato-graphy / EPA Method 300.0 and SW846 Method 9056A	Retention time (RT) window width calculated for each analyte	After method setup and after major maintenance (e.g. column change)	RT width is ± times standard deviation for each analyte over a 24-hour period.	N.A.
	Initial Instrument Calibration (ICAL): Blank + 5 standards, lowest standard at or below PQL	Every 6 months or with each change in instrument operating conditions or equipment	1) Correlation coefficient ≥ 0.995 2) Recovery of lowest standard within 50%-150%	Correct problem and recalibrate
	Retention time window established for each analyte	Once per ICAL or at the beginning of each day of use	Position shall be set using the midpoint standard of the ICAL when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N.A.
	Method blank	One per prep/analysis batch of 20 or fewer samples	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 prep/analysis batch of 20 or fewer samples, prepared from a separate source than calibration standard	90%-110% Recovery	(1) If the ICV/LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
	CCV	At beginning of run, after every 10 samples, and at end of run	(1) 90%-110% recovery. (2) All analytes within estabilished RT windows.	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.

TABLE 1

QC REQUIREMENTS (Continued)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Anions by Ion Chromato-graphy / EPA Method 300.0 and SW846 Method 9056A	CCB	Immediately following each CCV	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 CCB.
	Matrix Spike	One for every set of 10 samples (EPA 300.0) or one for every set of 20 samples (SW846 9056A)	90%-110% recovery (EPA 300.0) 80%-120% recovery (SW846 9056A)	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, flag result and narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.
	Matrix Spike Duplicate	One per 20 samples	(1) 90%-110% recovery (EPA 300.0) 80%-120% recovery (SW846 9056A) (2) RPD ≤15%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with flagging and narration.
	Sample Duplicate	One per 20 samples	RPD ≤20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with flagging and narration.
	Demonstration of analyst proficiency; accuracy and precision	One time initially by each analyst performing the method and annually thereafter.	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personnel training file
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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	ICAL prior to sample analysis.	r2 ≥ 0.99.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Minimum 3 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per multipoint calibration.	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.	Established for each analyte.
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 over a 24-hour period.	NA.	NA.	Calculated for each analyte.
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 ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Freshly prepared ICV. No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	Before sample analysis; after every 10 field samples; and at the end of the analysis sequence.	All reported analytes within established retention time windows. 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 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. Retention time windows are updated per the method.
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.

TABLE 2

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 re-prep and reanalyze the LCS and all samples in the associated preparatory batch for all reported 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.	Follow 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 reported analytes. 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. MSD or MD: RPD of all analytes ≤ 15% (between MS and MSD or sample and MD).	Follow 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 reported analytes. The data shall be evaluated to determine the source of difference.

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-742-11	Method 300.0/9056, current revisions
Calibration	1) Calibration consists of blank + 5 standards 2) Linearity verified by performing calibration at least every 6 months, and diluting all samples exceeding calibration range	1) Calibration consists of blank + at least 3 standards 2) Linear range verification required every 6 months (Method 300.0)
QC – Method Blank	Acceptance limit < PQL	Acceptance limit <MDL (Method 300.0) Acceptance limit <10% of LLOQ or regulatory limit or lowest sample (Method 9056A)
QC – LCS/ICV	Combined LCS and ICV with tighter acceptance limits (90%-110% recovery)	LCS acceptance limits 80%-120%, ICV acceptance limits 90%-110% (Method 9056A)
QC – Duplicate / Matrix Spike Duplicate	RPD acceptance limit ≤20%.	RPD acceptance limit ≤15% for samples at or above midrange, ≤50% for samples near LLOQ (Method 9056A)

TABLE 4

EXAMPLE ANALYTICAL SEQUENCE WITH ACCEPTANCE CRITERIA

Sample Number	Instrument Runlog	Acceptance Limit
1	CCV	90%-110%
2	CCB	< PQL
3	ICV / LCS	90 -110% (300.0) 80%-120% (9056A)
4	Sample 1	
5	Sample 2	
6	Sample 3	
7	Sample 4	
8	Sample 5	
9	Sample 6	
10	Sample 7	
11	Sample 8	
12	Sample 9 -Duplicate	20% RPD (10% for DoD)
13	Sample 10 - Matrix Spike	90 -110% (300.0) 80%-120% (9056A)
14	CCV	90%-110%
15	CCB	< PQL
16	Sample 11	
17	Sample 12	
18	Sample 13	
19	Sample 14	
20	Sample 15	
21	Sample 16	
22	Sample 17	
23	Sample 18	
24	Sample 19	
25	Sample 20 - Matrix Spike	90 -110% (300.0) 80%-120% (9056A)
26	CCV	90%-110%
27	CCB	< PQL

FIGURE 1

IC SYSTEM CONFIGURATION

ICS-2000 Ion Chromatography System

A typical IC analysis consists of six stages (see Figure 1-1).

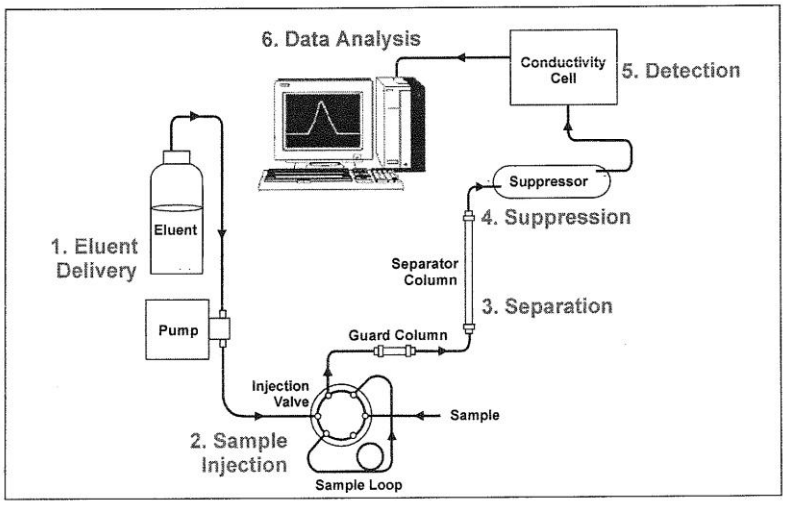

Figure 1-1. Ion Analysis Process

1. **Eluent Delivery**
   - Eluent, a liquid that helps to separate the sample ions, carries the sample through the ion chromatography system. The ICS-2000 includes an eluent generator, which generates eluent online from deionized water.
   - When the ICS-2000 is controlled from the front panel, only isocratic eluent delivery is possible. This means that the eluent composition and concentration remain constant throughout the run. Gradient delivery (a change in concentration over time) is possible when the ICS-2000 is controlled by Chromeleon® Chromatography Management System (the data collection system for the ICS-2000).
2. **Sample Injection**
   - The liquid sample is loaded into a sample loop either manually or automatically (if an automated sampler is installed). When triggered, the ICS-2000 injects the sample into the eluent stream.

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FIGURE 2

EXAMPLE OF IC MAINTENANCE LOG PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
WATERS IC (ION CHROMATOGRAPHY) - SERIAL NO. 001457
MAINTENANCE LOGBOOK

DAILY INSTRUMENT PERFORMANCE

	Date / Initials	Date / Initials	Date / Initials	Date / Initials	Date / Initials	Date / Initials
Record Line Pressure						
	√	√	√	√	√	√
Detector B Base Range - 50						
Detector B Sensitivity - 0.05						
WISP Injector Volume - 20 uL						
WISP Runtime - 10 Min.						
Instrument run w/ no samples						

ROUTINE MAINTENANCE (As Needed)

Task	Date	Initials	Comments
Clean surfaces of system unit as needed.			
Check regenerate pump tubing & replace as needed.			
Clean or regenerate column as needed.			
Change analytical column as needed.			
Change guard column as needed.			
Change suppressor as needed.			

NON ROUTINE MAINTENANCE (Date and Initial each entry)

- 5/5/12 cleaned anion guard and analytical columns w/ oxalic acid to sharpen PO_4 peak.
- 5/09/12 PO_4 RT - 8.59

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0000014

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FIGURE 3

EXAMPLE OF ANIONS BY IC RUNLOG PAGE

Katahdin Analytical Services, Inc **ION CHROMATOGRAPHY RUNLOG** Dionex ICS-2000 (Instrument IC-2)

Analysis Date: 06/26/12	**Analytical Column S/N:** 003709	**Calibration Date:** 06/26/12
Analysis Sequence: 06262012A	**Guard Column S/N:** 003612	**Calibration Sequence:** 06261ZA
Analyst: GFB	**Suppressor S/N:** 12032910 08	☐ If box at left is checked, continued from previous page. Refer to previous page for header information.

Reporting / Reanalysis Codes: ✓ Report without manipulation; S Report, peak automatically reintegrated with SmartPeak; M Report, peak manually integrated; A Report, peak manually assigned; R Do not report, reanalyze sample

Method Codes: E EPA 300.0; SW SW846 9056A

Katahdin Sample Number	Dilution Factor	Method Code	Report or Reanalyze (enter appropriate code): F	Cl	NO_2	SO_4	Br	NO_3	PO_4	Comments
Blank	1.0	E								
Blank										
Cal Std 1				✓	✓	✓	✓	✓	✓	
2										
3										
4										
5										
6										
CCV										
CCB										
Method Blank										
LCS				↓	↓	↓	↓	↓	↓	
3F38606-1				R	✓	✓	✓	✓	✓	Dilute + rerun Cl
-1 Dup					✓	✓	✓	✓	✓	
-1 MS	↓				✓	✓	✓	✓	✓	
-1	10.			✓						
-1	100.									
LOQ Soil NO2	1.0				✓					NO_2 spiked at 0.1 mg/L (10 mg/kg)
CCV	↓			✓	✓	✓	✓	✓	✓	
CCB	↓	↓		✓	✓	✓	✓	✓	✓	
										GFB 06/27/12

Reviewed by: _____ Review Date: _____

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 4

EXAMPLE OF IC BATCH SHEET

WET CHEMISTRY BATCH REPORT
May 29 2012, 10:02 am
Batch: WG108595

Parameter: Sulfate
Prep Date: N/A

Date Analyzed: 24-MAY-12
Prep Method: N/A

Analyst Initials: CT
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SF2996-1	SAMP	SW846 9056	1.0000mL	1.0000mL	1	17.7831	18. mg/L	NA	1	0.064	1.0		
SF2996-2	SAMP	SW846 9056	1.0000mL	1.0000mL	1	2.1434	2.1 mg/L	NA	1	0.064	1.0		
SF2996-3	SAMP	SW846 9056	1.0000mL	1.0000mL	1	2.0938	2.1 mg/L	NA	1	0.064	1.0		
SF2996-4	SAMP	SW846 9056	1.0000mL	1.0000mL	1	2.133	2.1 mg/L	NA	1	0.064	1.0		
SF2996-5	SAMP	SW846 9056	1.0000mL	1.0000mL	1	3.3662	3.4 mg/L	NA	1	0.064	1.0		
WG108595-1	MBLANK	SW846 9056	1.0000mL	1.0000mL	1	.0314	U0.50 mg/L	NA	1	0.064	1.0		
WG108595-2	LCS	SW846 9056	1.0000mL	1.0000mL	1	3.703	3.7 mg/L	NA	1	0.064	1.0		99
WG108595-3	MS	SW846 9056	1.0000mL	1.0000mL	2	21.0206	21. mg/L	NA	1	0.13	2.0		86
WG108595-4	MSD	SW846 9056	1.0000mL	1.0000mL	2	21.0358	21. mg/L	NA	1	0.13	2.0	0	87

Comments:

SF2996-1	MS/MSD
WG108595-1	SF2863-6
WG108595-2	SF2863-6
WG108595-3	SF2996-1
WG108595-4	SF2996-1

Entered by: CT Date: 5/29/12 Accepted by: [signature] Date: 05/30/12

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Alex Pimentel

Review Date: 2/15/19

SOP Number: CA- 742- 11

SOP Title: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

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: Leslie Dimond for George Brewer **Date:** 02.18.19

QAO Signature: Leslie Dimond **Date:** 02.18.19

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

Prepared By: Chad Delaney Date: 07.14.04

Approved By:

Department Manager: George Brewer Date: 07/14/04

Operations Manager: Deborah J. Nadeau Date: 7.14.04

QA Officer: Maria Crouch Date: 07.14.04

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Revised Title and text to incorporate analysis for Total Inorganic Carbon (TIC) minor changes throughout Updated Figures	LAD	04/06	04/06
02	Changed SM5310B to SM5310C throughout. Added reference to SW846 9060 throughout. Updated Figure 6. Updated Table 2 to include that we do not subtract the blank from the sample.	LAD	03/07	03/07
03	Added LCS/ICV, CCV, and CCB definitions. Added calibration criteria. Added quadruplicate criteria for SW9060. Added repeat injection for SM 5310B. Added wording regarding SC criteria to Sect. 8. Changed CCV criteria to 90>~~100~~110% LAD	LAD	06/08	06/08
04	Updated reference from 9060 to 9060A. Sect. 7.9 - Changed C:\npoc\AQ.met for TOC/DOC to C:\doubleinject.met for TOC/DOC.	LAD	03/09	03/09
05	Added EHS4, QA-806, ~~SUBSAMPLING~~, DW 8/101 DOD, NELAC & CA-161 references.	DN	08/09	08/09

TITLE: ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Removed figures 1 and 2. Updated figures 2-4 (formally 4-4). Updated references.	LAD	06/10	06/10
07	Sect. 5 - Updated calibration levels. LCS and MS prep. Sect. 7 - Added regeneration of TC and IC catalysts. Added stand-by mode and shutting instrument down instructions. Added data archival. Sect. 9. Added MDL LOD and LOQ info. Sect. 10 - Added updated references.	LAD	05/12	05/12
08	Sect. 10 - Updated and added References. Updated Fig. 1 → 4	LAD	07/14	07/14
09	Sect. 5 - Updated TOC calibration Standard preparation. Updated Analysis Run Information Sheet (Fig. 3) KAS INC → KAS	LAD	09/16	09/16
10	Sect. 6 - Added H_3PO_4 preservation. Sect. 7 - Corrected MS preparation wording. Sect 8 and Table 1 - Corrected the method Blank and LCS frequency for method SM5310B to a batch of 10 or less.	LAD	06/17	06/17

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

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-763-10** titled **Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-763-10** titled **Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B**.

Recipient: _____ Date: _____

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel for analyzing total organic carbon (TOC), dissolved organic carbon (DOC), and total inorganic carbon (TIC) in aqueous samples using the Shimadzu Carbon Analyzer in accordance with EPA method 415.1, SW846 9060A and SM 5310B.

This procedure applies to drinking and surface waters, domestic and industrial wastewater. The Practical Quantitation Limit (PQL) is 1 mg/L.

1.1 Definitions

Total Organic Carbon (TOC) – Carbon that is bound with hydrogen or oxygen in organic compounds, analyzed from an unfiltered sample.

Dissolved Organic Carbon (DOC) - Carbon that is bound with hydrogen or oxygen in organic compounds, analyzed from a filtered sample.

Total Inorganic Carbon (TIC) – Carbon contained in carbonates, hydrogen carbonates, or dissolved carbon dioxide.

Method Blank - A laboratory reagent grade water sample that 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 laboratory reagent grade water with no reagents 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 preparation and analysis of samples for TOC, DOC, and TIC using EPA Method 415.1, SW846 9060 and SM5310 B. Each analyst must demonstrate his/her 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 TOC and DOC 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 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.

1.4 Pollution Prevention and 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.

Waste generated during the preparation and analysis of samples must be discarded appropriately. Place all analyzed samples, standards, and rinsings in a Satellite Waste “A” Acid for proper disposal in main waste area “A”.

Other wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health & 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: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

2.0 SUMMARY OF METHOD

Organic carbon analysis – Sample is drawn into a syringe in the instrument, and hydrochloric acid is then drawn into the syringe to acidify the sample to pH 2 to 3. Oxygen is then bubbled through the acidified sample in the syringe to drive off the inorganic carbon component. The sparged sample is then introduced into the total carbon combustion tube, where it is heated to 680^{o} C in the presence of an oxidation catalyst. The organic carbon remaining in the sample after sparging is converted to carbon dioxide during combustion. Carrier gas sweeps the sample combustion gases through an electronic dehumidifier, where they are cooled and dehydrated, and then through a halogen scrubber to remove chlorine and other halogens. Finally, the carrier gas delivers the combustion gases to the cell of a non-dispersive infrared (NDIR) gas analyzer, where carbon dioxide is detected and quantitated. The amount of carbon dioxide in a sample is directly proportional to the concentration of organic carbon (TOC or DOC) in the sample. Strictly speaking, this method measures non-purgeable organic carbon (NPOC), because purgeable (volatile) organic compounds are lost during sparging of the sample. However, the amount of purgeable organic compounds in natural waters is small, and NPOC is nearly equivalent to total organic carbon. When necessary, total organic carbon can be indirectly determined by measuring total carbon on an unsparged sample, measuring inorganic carbon on a separate aliquot of the sample, and calculating TOC by difference.

Inorganic carbon analysis – Inorganic carbon (IC) consists of the carbon contained in carbonates and in carbon dioxide dissolved in water. IC is measured by injecting an aliquot of unpreserved sample into the instrument's IC reaction vessel, where it is acidified with hydrochloric acid and inorganic carbon is converted to carbon dioxide. The carbon dioxide is then sparged from solution with oxygen and carried to the NDIR detector, where it is detected and quantitated.

3.0 INTERFERENCES

High salt samples can affect the oxidation rate, leading to low recoveries. Interference begins at ~0.1% total dissolved solids and becomes severe at > 0.35%.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu Carbon Analyzer Model TOC-V

4.2 Shimadzu carousel autosampler Model ASI-V

4.3 Glass 40 mL VOA Vials

4.4 Class A volumetric flasks (100 mL and 200 mL)

4.5 Adjustable pipets (0.1, 1.0 and 5.0 mL)

4.6 Oxygen gas

5.0 REAGENTS AND STANDARDS

NOTE: All standards and reagents should be prepared <u>daily</u> except where noted. Record all standard and reagent preparation in the appropriate logbook.

5.1 Phosphoric acid (H_3PO_4), concentrated, reagent grade.

5.2 Phosphoric acid (H_3PO_4), 1:1 – Slowly add 100 mL concentrated H_3PO_4 to 100 mL and mix. Prepare fresh every six months.

5.3 Hydrochloric acid (HCl), concentrated, reagent grade.

5.4 Hydrochloric acid (HCl), 2M – Slowly add 42.5 mL concentrated HCl to 200 mL of reagent water and bring to a final volume of 250 mL. Prepare fresh every six months.

5.5 Potassium hydrogen phthalate ($HOCOC_6H_4COOK$), primary standard grade. Two different lots of potassium hydrogen phthalate are required – one is used to prepare organic carbon calibration standards, and the other is used to prepare a laboratory control sample (second source).

5.6 Sodium carbonate (Na_2CO_3), 1 N – Purchased certified standard solution, inorganic carbon true value = 6000 mg/L. This solution is used to prepare the inorganic carbon stock standard and the alkalinity check standard.

5.7 Sodium carbonate (Na_2CO_3), anhydrous – ACS reagent grade. Store in a desiccator. This reagent is used to prepare the inorganic carbon laboratory control sample.

<u>Organic Carbon (OC) Standards</u>

5.8 OC Stock Standard (used in preparing OC calibration standards and CCV): Prepare a 2000 mg/L standard by dissolving 2.0324 g of potassium hydrogen phthalate in 500 mL of laboratory reagent grade water. Add 0.50 mL of concentrated phosphoric acid. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.9 OC Calibration Standards – Prepare a series of five calibration standards by diluting the OC Stock Standard with reagent water in accordance with the following table:

Calibration Std. Conc. (mg/L OC)	mL of OC Stock Std. Added	Final Volume (mL)
0	0	40
1.0	0.02	40
5.0	0.10	40
20.0	0.40	40
50.0	1.0	40
100.0	2.0	40
150.0	3.0	40
200.0	4.0	40

Note: The high point (200mg/L) may be dropped if the r >/= 0.995 criteria is not met. If the 200 mg/L level is dropped, any samples with TOC concentration > 150 mg/L need to be reanalyzed at dilution.

5.10 OC CCV Standard - Prepare a 100 mg/L standard for the CCV by diluting 25 mL of the OC Stock Standard to 500 mL using laboratory reagent grade water. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.11 OC Laboratory Control Sample (LCS) – Prepare a 50 mg/L standard from a different source from the calibration standards, as follows. Fill a 1 L volumetric flask half full with laboratory reagent grade water and add one mL of concentrated phosphoric acid as a preservative. Add 0.1062 g of potassium hydrogen phthalate and bring to a final volume of 1 L with laboratory reagent grade water. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.12 Alkalinity Check Sample - Prepare by diluting 2.5 mL of 1.0 N Na_2CO_3 to one liter with laboratory reagent grade water. The inorganic carbon content of this solution is 15 mg/L. This standard is used to monitor for false positives during OC analysis from incomplete removal of inorganic carbon.

Inorganic Carbon (IC) Standards

5.13 IC Stock Standard (used in preparing IC calibration standards and CCV): Prepare a 2000 mg/L standard by diluting 100 mL of 1 N Na_2CO_3 to a final volume of 300 mL in a graduated cylinder. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.14 IC Calibration Standards – Prepare a series of five calibration standards by diluting the OC Stock Standard with reagent water in accordance with the following table:

Calibration Std. Conc. (mg/L IC)	mL of IC Stock Std. Added	Final Volume (mL)
1.0	0.10	200
20.0	2.0	200
100.0	10.0	200
200.0	20.0	200

5.15 IC CCV Standard - Prepare a 100 mg/L standard for the CCV by diluting 25 mL of the OC Stock Standard to 500 mL using laboratory reagent grade water. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.16 IC Laboratory Control Sample (LCS) – Prepare a 100 mg/L standard from a different source from the calibration standards, as follows. Fill a 250 mL volumetric flask half full with laboratory reagent grade water and add 0.2208 g of anhydrous sodium carbonate. Mix the solution until the sodium carbonate has dissolved, and bring to a final volume of 250 mL with laboratory reagent grade water. Stored in a glass bottle in the refrigerator, this solution may be used for three months. Prepare for analysis by adding 20 mL of the 100 mg/L standard to 20 mL of DI Water in a VOA vial. The final concentration is 50 mg/L.

5.17 IC Matrix Spike (MS) sample – IC matrix spike aliquots are prepared by adding 1.0 mL of IC Stock Standard to 39 mL of sample in a VOA vial. The final matrix spike concentration is 50 mg/L.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect samples in glass jars or VOA vials. For organic carbon analysis, preserve with H_2SO_4 or H_3PO_4 at time of collection and store at 4 (±2) °C prior to analysis. For inorganic carbon analysis, do not add acid to the sample, but store at <6 °C, without freezing, prior to analysis. The holding time for TOC, DOC, and IC analysis is 28 days from the time of collection.

If dissolved organic carbon (DOC) is to be determined, the sample must be filtered through a 0.45 micron glass fiber filter prior to preservation with H_2SO_4. If the sample has not been filtered in the field, sample log-in must inform the Inorganic Department Manager that samples need to be filtered upon receipt by the laboratory. Please refer to the current revision of Katahdin SOP SD-902, Sample Receipt and Internal Control, section 7.7.5, for further filtration procedures.

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

7.0 PROCEDURES

ANALYZER START-UP PROCEDURE

7.1 Turn on the TOC-V module, computer, and oxygen tank.

7.2 Fill rinse water reservoir with laboratory reagent grade water. Verify that there is sufficient room in the satellite waste container to contain the day's waste. Verify that the instrument's humidifier water level is between the "High" and "Low" lines**,** and add laboratory reagent grade water if needed. Also verify that the diution water, hydrochloric acid, and phosphoric acid reservoirs are full.

7.3 Carrier gas flow rate should be set at 150 mL/min. The oxygen pressure should read 200 kPa on the gauge inside the instrument.

7.4 From the main screen, double-click on the **TOC-Control V** icon.

7.5 Double-click the **Sample Table Editor** icon. Enter the User ID and Password.

7.6 At the menu bar, click on File , then click New. Double-click on the **Sample Run** icon.

7.7 Make certain that the System setting is set for "TOC-Vcph/ASI-V", then click OK.

7.8 Click on the **Connect (yellow lightning bolt)** icon to connect the computer to the instrument. Click "Use Settings on PC". This turns on the furnace inside the instrument, and turns on the gas flows. The furnace must be allowed to heat up for one half hour before beginning analysis.

7.9 When the instrument is ready, the TC o or the IC catalyst needs to be regenerated. At the menu bar select Instrument, scroll down to maintenance and select either TC Regenerate or IC regenerate. Once selected a new window will drop down. Click on Start and allow the procedure to finish. When the regeneration is complete, close the window. The instrument is now ready for sample analysis.

SAMPLE ANALYSIS

7.10 At the menu bar, click on Insert, then click Auto Generate. Double-click on the appropriate method ("doubleinjection.met for TOC/DOC, "INORG CARB AQ.met" for inorganic carbon). Then click Next.

7.11 Enter the number of samples as "60" and the start vial as "5", then click Next.

7.12 At the Calibration Curve screen (Screen 3), click Next. You must verify the calibration curve is current (within three months of analysis) by going into the NPOC

Method Menu. If the curve is more than three months old, a new curve must be run.

7.13 Click to put a checkmark in all three boxes (beside "At The Beginning", "After Every Ten Samples", and "At The End of Sample Group") under Quality Control.

7.14 Select (1) by clicking on the number (it will highlight the (1) in blue). Click Add. Then click "CCV". This inserts a CCV in autosampler position (1).

7.15 Repeat 7.13 for autosampler position (2), clicking "Blank", and then for autosampler position (3), clicking "LCS".

7.16 Click Finish.

7.17 You may bypass the vial selection screen at this time by clicking OK.

7.18 An autosampler table now appears on the screen. Fill in sample numbers in the "Sample Name" column. If a manual dilution has been performed on the sample in the VOA vial, enter the dilution factor in the "Dilution" column for each diluted sample.

7.19 Unneeded Quality Control and Sample rows may be deleted by selecting each row and then clicking the **Cut (scissors)** icon.

7.20 Place each sample vial in its appropriate position on the autosampler carousel.

7.21 Matrix spikes are prepared by adding 2 mL of the OC or IC Stock Standard to a 50 ml graduated cylinder, adding sample for a final volume of 40 then poured into a 40 mL VOA vial.

7.22 Place the autosampler carousel on the ASI auto sampler and replace the ASI cover.

7.23 Click the **Start (traffic light)** icon, the enter a data file name and click "Save". Data files are named according to the analyte (TOC or TIC), the matrix (AQ), and the date (MMDDYY format), e.g. "TOC_AQ_031505" for an aqueous TOC run performed on 03/15/05.

7.24 Click "Standby".

7.25 An image of the carousel will appear on the screen, along with a table listing the sample numbers and corresponding autosampler positions. Carefully enter the corrected autosampler positions in accordance with the previously printed list that was used to set up the autosampler carousel.

7.26 Click "OK".

7.27 The instrument will then run samples until it has finished with the programmed autosampler sequence.

7.28 When the sequence has finished, the instrument must be set to standby mode before shutting down completely. On the menu bar select standby. Click on Okay. After 30 minutes, the instrument will shut itself down. After the instrument has shut down, turn off the oxygen and click the "Off" button. Though the instrument is already off, the button stills needs to be pressed.

ANALYTICAL QUALITY CONTROL

7.28 A calibration curve is analyzed as necessary but at least every 3 months. Standards are prepared as described in section 5. Acceptance criteria for the calibration is a 0.9950 correlation coefficient for the full curve including the blank. Operator discretion in approving or rejecting calibrations is encouraged. The decision and rational must be recorded in the instrument log.

A linear calibration applying a first order equation is used to prepare the curve. The equation is:

$$y = mx + b$$

where: y = Instrument response
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

Acceptance criterion is a 0.995 correlation coefficient or better.

7.29 An analytical batch consists of 20 or fewer field samples (not counting method blanks, laboratory control samples, duplicates, and matrix spikes).

7.30 The operating range of OC and IC analysis for a 150 uL sample injection is 0-200 mg/L. Samples with measured concentrations greater than 200 mg/L must be diluted and reanalyzed.

7.31 Each analytical sequence must start with analysis of a CCV, followed by analysis of a method blank consisting of laboratory reagent grade water. Analyze an second-source laboratory control sample (LCS) with each analytical batch.

7.32 Each TOC or DOC sample is sparged with oxygen to remove residual inorganic carbon. To check for sparging efficiency, analyze an alkalinity check standard near the beginning of each run.

7.33 Samples analyzed by Standard Methods 5310 B must be injected at least twice and have results within ± 10% of each other. If results are greater than ± 10%, the sample should be reinjected until consecutive measurements are within ± 10% of each other.

7.34 Samples analyzed by method SW846 9060 must be analyzed in quadruplicate

7.35 One matrix spike (MS) should be analyzed for every ten (or fewer) samples.

7.36 One sample in every batch of 20 or fewer samples must be analyzed in duplicate. Method SW9060 requires a sample in every batch of 20 or fewer samples be analyzed in quadruplicate.

DATA REVIEW AND REPORTING

7.37 After completion of analysis, instrument data files are imported electronically into the Katahdin Information Management System (KIMS) for calculation and reporting of sample results and quality control samples. After data processing by KIMS, a batch sheet (Figure 2) listing Katahdin Sample Numbers with associated reported results and associated quality control data is printed out of KIMS for each analytical batch. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.38 Analytical data are stored in the instrument's computer during analysis, and data reports are printed after each analytical run has been completed. The instrument data printout shows the signal versus run time plot that was obtained for each sample, as well as the peak area and measured concentration, the dilution factor, and the analysis date and time. A run data summary table (Figure 1) is also printed out from the instrument, containing all of this information except peak plots in analysis order. Copies of the run data summary tables are bound in the Aqueous Carbon Analysis Run Logbook.

7.39 A Carbon Analysis Run Information Sheet (Figure 3) is completed by the analyst for each analytical run. This form lists the analysis methods, dates, and times, calibration information, and the standard IDs for each standard used in the run.

7.40 The raw data package for each analytical run is assembled by the analyst, and consists of the following information (in the order listed):

- KIMS batch sheets (Figure 2) for each batch contained in the run
- The Carbon Analysis Run Information Sheet (Figure 3)
- The run data summary table (Figure 1)
- The raw data printouts which include peak plots
- Associated calibration data

7.41 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

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 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), in a program specific Quality Systems Manual (QSM) or in state specific criteria. 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 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. 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) standard is analyzed for each analyte after every ten samples and at the end of the analytical run. The CCV solution is as described in Section 5. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, sample results that are associated with (bracketed by) the failing CCV may not be reported from the run. The samples must be reanalyzed after the problem is corrected and a passing CCV has been analyzed.

8.3 A Continuing Calibration Blank (CCB) consisting of reagent water is analyzed after each CCV. The absolute values of results of CCBs must be less than the Practical Quantitation Level (PQL) for each analyte. If a CCB fails, sample results that are associated with (bracketed by) the failing CCB may not be reported from the run, with the following exception. If the absolute value of a result for a CCB 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. All other samples must be reanalyzed after the problem is corrected and a passing CCB has been analyzed.

8.4 An Alkalinity Check Sample, prepared as described in Section 5.13, is analyzed in each TOC and DOC run. This sample is used to monitor for false positives during OC analysis from incomplete removal of inorganic carbon. The absolute value of results of the Alkalinity Check Sample must be less than the Practical Quantitation Level (PQL) for each analyte. If the Alkalinity Check Sample fails, associated sample results may not be reported from the run. The samples must be reanalyzed after the problem is corrected and a passing Alkalinity Check has been analyzed.

BATCH QC SAMPLES

8.5 A Method Blank, consisting of reagent water, is analyzed with each batch of ten or fewer samples for method SM5310B and each batch of twenty or fewer samples for methods SW846 9060A and EPA 415.1. The results of method blanks must be less than the Practical Quantitation Level (PQL) for each analyte. If a method blank fails, results for associated samples may not be reported from the batch, with the following exception. If the result for a method 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. Associated sample results that are less than the PQL may be reported with no additional notation.

8.6 A laboratory control sample (LCS), prepared as described in Section 5, is analyzed with each batch of ten or fewer samples for method SM5310B and each batch of twenty or fewer samples for methods SW846 9060A and EPA 415.1. The LCS recovery must be 80-120% for USEPA Method 415.1 and SW846 9060. The LCS recovery must be 90-110% for SM5310B. If a laboratory control sample fails,

results for the associated samples may not be reported from the batch, and the samples must be reanalyzed, with the following exception: if the LCS recovery is greater than the upper acceptance limit, samples with measured concentrations below the PQL may be reported with appropriate narration.

MATRIX QC SAMPLES

8.7 Duplicate samples are analyzed at a minimum frequency of one per batch of 20 or fewer samples. The relative percent difference (RPD) between 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 result
D_2 = Second (duplicate) sample result

A control limit of 20% RPD is applied to duplicate analysis. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.8 Matrix spiked samples are prepared at a minimum frequency of one per 10 samples. The recovery for each element in a spiked sample must fall within 75%-125% for USEPA Method 415.1 and SW846 9060 or 80%-120% for SM 5310B 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:

$$Recovery\ (\%) = \frac{S-U}{SA} * 100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

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 Quantitaion (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 415.1, SW846 9060 and SM5310B for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

EPA Method 415.1 "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, Revised March, 1983.

Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update IIIB, November, 2004, Method 9060A.

Standard Methods for the Examination of Water and Wastewater, Method 5310 B, High Temperature Combustion Method, 21st Edition, 2005, approved by Standard Method Committee, 2000.

Standard Methods for the Examination of Water and Wastewater, Method 5310 B, High Temperature Combustion Method, 22st Edition, 2012, approved by Standard Method Committee, 2000, editorial revisions 2011.

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

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

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.

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.

LIST OF TABLES AND FIGURES

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

TABLE 1

QC REQUIREMENTS

Analytical Method/ Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 415.1 SW846 9060A & SM 5310B TOC/TIC/ DOC	Initial Calibration (including five standards plus blank)	At a minimum every 3 months or as necessary	Linear Regression Correlation Coefficient ≥0.995	(1) Investigate source of problem (2) Recalibrate
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	90%-110% of true value	Reanalyze all associated samples.
	Continuing Calibration Blank (CCB)	After every CCV	Absolute value <PQL	1) If sample result < PQL, or >10x measured CCB value, report result 2) Else, reanalyze
	Method blank	**EPA 415.1 & SW846 9060:** One per analytical batch of 20 or fewer samples **SM5310B:** One per analytical batch of 10 or fewer samples	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.
	Laboratory control sample (LCS)	**EPA 415.1 & SW846** 9060: One per analytical batch of 20 or fewer samples **SM5310B:** One per analytical batch of 10 or fewer samples	**EPA 415.1 & SW846 9060:** 80%-120% **SM5310B:** 90%-110%	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep the LCS and reanalyze the remaining samples.
	Matrix spike	One MS per ten samples	**EPA 415.1 & SW846 9060:** 75%-125% **SM5310B:** 80%-120%	If LCS in criteria and matrix interference suspected, flag data
	Sample Duplicate	One sample duplicate per twenty samples	RPD ≤ 20	If lab QC in criteria and matrix interference suspected, flag data

	Initial demonstration of performance (4 replicate analyses of LCS)	Once per analyst per year	Precision and accuracy within method acceptance limits	Correct problem and repeat initial demonstration of performance
	MDL study and/or LOD and LOQ verifications.	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-763-0910	METHODS 415.1, SW846 9060 and SM5310B, current revisions
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	None	
QC – Blanks	Blank Subtraction not performed	Method SM5310B requires subtraction of method blank concentration from results for associated samples.
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	None	

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 1

EXAMPLE OF RUN DATA SUMMARY TABLE

	Sample Name	Dilutio	Result	Comment	Date / Time	Vial
1	CCV	1.000	NPOC:94.82 mg/L		06/30/14 06:23:17	1
2	BLANK	1.000	NPOC:0.2570 mg/		06/30/14 06:35:06	2
3	LCS	1.000	NPOC:48.06 mg/L		06/30/14 06:49:06	4
4	ALK CHECK	1.000	NPOC:0.5910 mg/		06/30/14 07:00:53	5
5	SH4749-1	1.000	NPOC:0.2230 mg/		06/30/14 07:12:33	6
6	SH4749-1 MS	1.000	NPOC:95.35 mg/L		06/30/14 07:26:45	7
7	SH4793-4	1.000	NPOC:0.3079 mg/		06/30/14 07:38:29	8
8	SH4793-4 DUP	1.000	NPOC:0.3111 mg/		06/30/14 07:48:18	8
9	SH4016-31	1.000	NPOC:0.3909 mg/		06/30/14 08:00:08	9
10	SH4016-33	1.000	NPOC:0.2564 mg/		06/30/14 08:11:51	10
11	SH4016-45	1.000	NPOC:0.1410 mg/		06/30/14 08:23:18	11
12	SH4016-49	1.000	NPOC:0.3093 mg/		06/30/14 08:34:58	12
13	SH4662-1	1.000	NPOC:1.038 mg/L		06/30/14 08:47:07	13
14	CCV	1.000	NPOC:91.58 mg/L		06/30/14 09:01:34	1
15	BLANK	1.000	NPOC:0.1580 mg/		06/30/14 09:13:07	2
16	SH4662-2	1.000	NPOC:0.5111 mg/		06/30/14 09:25:07	14
17	SH4662-2 DUP	1.000	NPOC:0.5064 mg/		06/30/14 09:34:58	14
18	SH4662-2 MS	1.000	NPOC:92.09 mg/L		06/30/14 09:49:04	15
19	SH4662-2 MSD	1.000	NPOC:91.04 mg/L		06/30/14 10:01:14	15
20	SH4662-3	1.000	NPOC:1.062 mg/L		06/30/14 10:13:19	16
21	SH4734-8	1.000	NPOC:0.8469 mg/		06/30/14 10:25:25	17
22	CCV	1.000	NPOC:93.17 mg/L		06/30/14 10:39:53	1
23	BLANK	1.000	NPOC:0.1535 mg/		06/30/14 10:51:28	2
24						
25						
26						
27						
28						
29						
30						

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 2

EXAMPLE OF TOC BATCH SHEET (FROM KIMS)

WET CHEMISTRY BATCH REPORT
Jul 08 2014, 10:41 am
Batch: WG146121

Parameter: Total Organic Carbon
Date Analyzed: 30-JUN-14
Analyst Initials: ZS

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	Prep	Misc
884749-1	SAMP	SM5310B	20.000mL	20.000mL	1	.223	J0.22 mg/L	NA	1	0.10	1.0		
884793-4	SAMP	SM5310B	20.000mL	20.000mL	1	.3079	J0.31 mg/L	NA	1	0.10	1.0		
WG146121-1	MBLANK	SM5310B	20.000mL	20.000mL	1	.257	J0.26 mg/L	NA	1	0.10	1.0		
WG146121-2	LCS	SM5310B	20.000mL	20.000mL	1	48.06	48. mg/L	NA	1	0.10	1.0		MG
WG146121-3	MS	SM5310B	20.000mL	20.000mL	1	95.35	95. mg/L	NA	1	.1023	1.0		MS
WG146121-4	DUP	SM5310B	20.000mL	20.000mL	1	.3111	31.0 mg/L	NA	1	.1023	1.0	MC	

Comments:

WG146121-1	884749-1
WG146121-2	884749-1
WG146121-3	884749-1
WG146121-4	884793-4

Entered by: ZS Date: 7.8.14 Accepted by: D Radcan Date: 7.8.14

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 3

EXAMPLE OF CARBON ANALYSIS RUN INFORMATION SHEET

KATAHDIN ANALYTICAL SERVICES, LLC
CARBON ANALYSIS RUN INFORMATION SHEET

INSTR. ID: WC2 (Shimadzu TOC-V_{CPH}) **ANALYST:** ZF **DATE:** 9-2-16

FILE NAME: TOCAQ 090216 **METHOD(S):** TOC DOC TIC

- (•) EPA 415.1 • EPA 415.1 • EPA 415.1
- (•) SM5310B • SM5310B • SM5310B
- (•) SW9060 • _____ • _____

CALIBRATION DATE: 8-24-16 **CALIBRATION ANALYST:** ZF

Calibration standards were prepared by performing dilutions of the following standard on the day of calibration:

Calibration Source Standard ID	Prep Date	Expiration Date	Standard Conc. (mg/L)
LCS4935	8-24-16	11-24-16	2000

Note: Calibration must be performed quarterly or whenever a change in analysis conditions warrant. A copy of the associated calibration data is attached to this run.

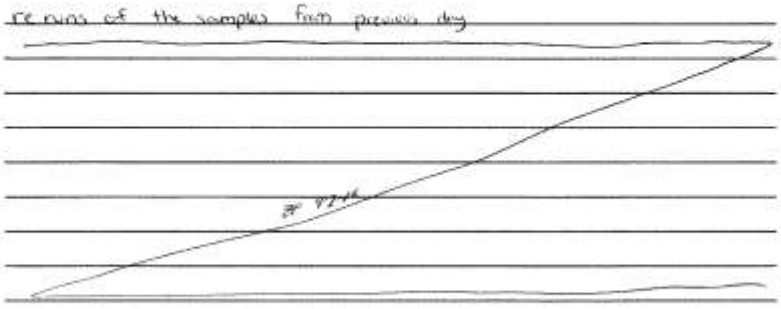
STANDARDS USED:

Standard Name	Standard ID	Prep Date	Expiration Date	Standard Conc.
CCV	LCS4936	8-24-16	11-24-16	100 mg C/L
LCS	LCS4937	8-17-16	11-17-16	50 mg C/L
MS Stock Std. *	LCS4935	8-24-16	11-24-16	2000 mg C/L
Alkalinity Check	LCS4933	8-24-16	11-24-16	15 mg/L (Inorg. C)

* Matrix spikes are prepared by adding 2.0 mL of MS Stock Std. to 38 mL of sample.

Additional Comments and Notes:

re runs of the samples from previous day



WL-067 – Revision 1 – 01/06/2016

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Ryan Oliver

Review Date: 2/21/19

SOP Number: CA-763-10

SOP Title: Analysis of TOC, DOC, & TIC in Aqueous Samples Using the Shimadzu Carbon Analyzer

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: R. Brewer **Date:** 02/21/19

QAO Signature: Leslie Dimond **Date:** 02.21.19

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 Brewer Date: 03/11/08

Operations Manager: Deborah J. Nadeau Date: 3.11.08

QA Officer: Leslie Dimond Date: 3.11.08

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added definitions to section 1.1. 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: DoD QSM 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, LLC. to KAS throughout. Updated Method references. Sect 7.6 - added note for NO MS recovery. Table 3 - added reagent modification. Updated Figure 5.	LAD	09/17	09/17

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-772-04, 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-772-04, 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.

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 standardsand 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. Discard when the solution becomes discolored. Also may be purchased from EST.

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.

7.0 PROCEDURES

7.1 Refer to Konelab operation SOP (CA-769) for instrument operation and maintenance procedures.

7.2 Load the 10% sulfuric acid and the hexavalent chromium color reagent onto the reagent wheel as described in the Konelab operation SOP.

7.3 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.

7.4 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.

7.5 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.

7.6 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 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, 10/06/2010.

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.

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

LIST OF TABLES AND FIGURES

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

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 $\leq$ 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.	

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-772-04	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		

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 1

EXAMPLE OF CALIBRATION CURVE

Calibration results AquaKem 6.5 Page: 1

KONELAB #1
KATAHDIN ANALYTICAL SERVICES

01.02.2008 09:47

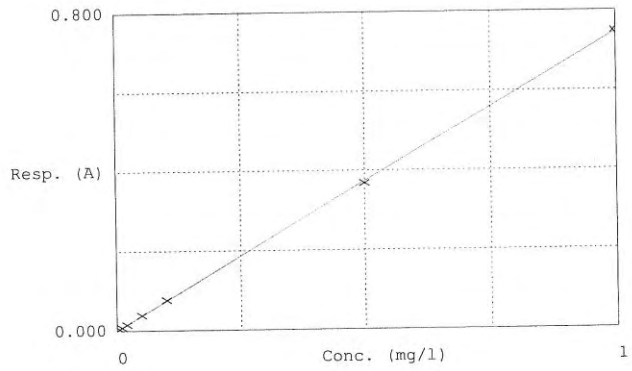
Test CHROMIUM

Accepted 01.02.2008 09:47 ICK K 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 2.1.09

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: CP 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:

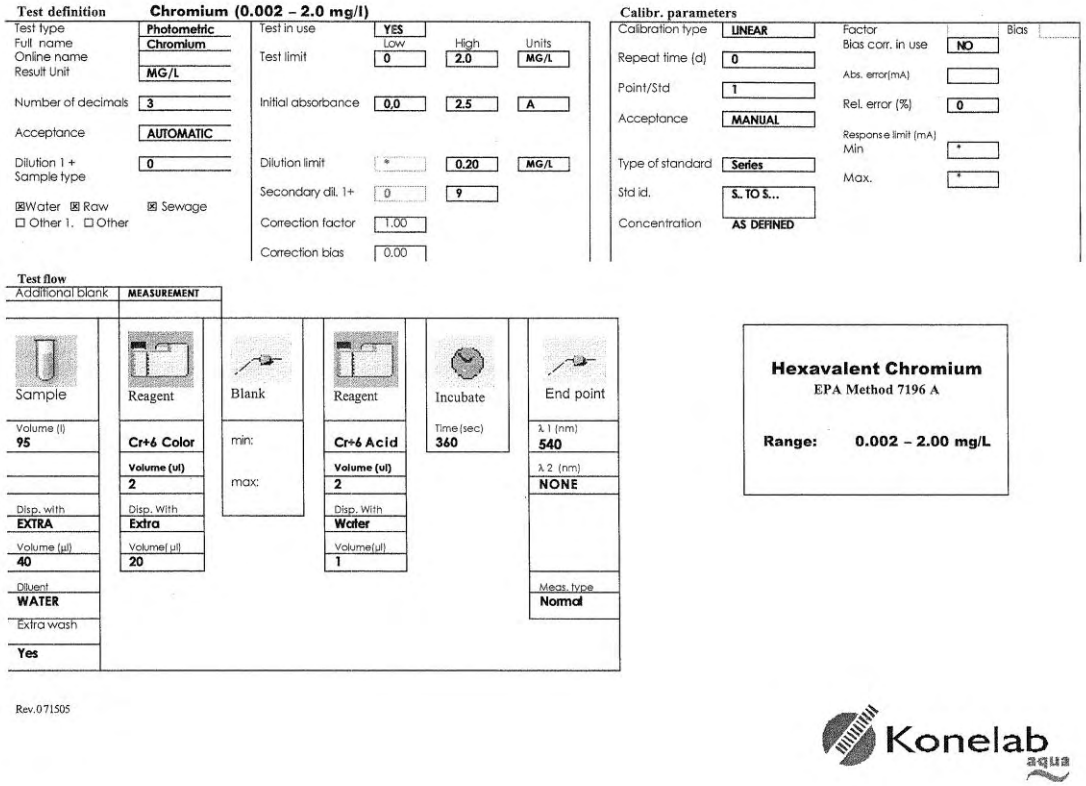
1) Additional calibration standards (0.50, 0.10, 0.050, 0.020, 0.010 mg/L) are automatically prepared by the instrument during analysis through dilution of the 1.0 mg/L Calibration Standard.
2) Matrix spikes are prepared prior to analysis by adding 0.5 mL of LCS / MS Intermediate to 25 mL of sample. Matrix spike added = 0.50 mg/L as Cr6.

Comments:

TITLE: COLORIMETRIC ANALYSIS OF HEXAVALENT CHROMIUM USING THE AUTOMATED KONELAB MULTIWAVELENGTH PHOTOMETRIC ANALYZER

FIGURE 6

TEST FLOW DIAGRAM



ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Zachary Fuller

Review Date: 7/24/19

SOP Number: CA-372-04

SOP Title: Colorimetric Analysis of Hexavalent Chromium using the Automated Konelab Multiwavelength Photometric Analyzer

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: C. Brewer **Date:** 07/24/19

QAO Signature: Lucie Dimond **Date:** 07.25.19

SOP CHANGE FORM

Cr6+ by IC
SOP Code: GEN-7199
SOP Revision No.: 7
SOP Date: 10/23/17
SOP Section(s) Affected by Change: 12.3.3

Description of Change:
Add a note to section 12.3.3

The EPA method requires that the RSD between the double injections is <20RSD. The laboratory uses a more stringent limit of <20RPD.

Reason(s) for Change(s):

NCAR RC0793 NY 2018 audit pg23

Change(s) Submitted by: Vicky Collom	Date: 3/5/18

Approvals:

Technical Reviewer Signature:	Date:
QA Signature: V. Collom	Date: 3/22/18
Operations Manager Signature:	Date: 3/18/18

Change(s) Effective Date: current practice

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

HEXAVALENT CHROMIUM BY ION CHROMATOGRAPHY FOR WATER AND SOIL EXTRACTS

DOCUMENT I.D. GEN-7199

Approved By: _____ Date: 10/13/17
Department Supervisor, Christopher Woods

Approved By: _____ Date: 10/13/17
Inorganics Manager, Christine Kutzer

Approved By: _____ Date: 10/13/17
Operations Manager, Michael Cymbal

Approved By: _____ Date: 10/13/17
Quality Assurance Manager, Vicky Collom

Approved By: _____ Date: 10/15/17
Laboratory Director, Carlton Beechler

Doc Control ID:	17-GEN-01	Archived Date:	

10/16/17

TABLE OF CONTENTS

1) Scope & Applicability

1.1 This SOP uses the following methods for the determination of Hexavalent chromium: EPA Method 7199 for water samples and soil extracts; 218.6 for drinking water and other waters; 218.7 for drinking water; NIOSH 7605 for air filter extracts, and 0061 for air impingers.

1.2 Although testing for Hexavalent chromium is not currently regulated in drinking water (except through total chromium limits), the EPA recommended monitoring (Dec 2010) of Hexavalent chromium using 218.6, modified to reach a MDL of 0.02 ug/L (Jan 2011). The EPA released method 218.7 in January of 2012. Method 218.7 has been included in UMCR3 and is to be used for testing of certain public water systems designated by EPA starting January 2013. The UCMR3 MRL is 0.03 ug/L.

1.3 This method may not be useful for the analysis of samples containing high levels of anionic species such as sulfate and chloride, since these species may cause column overload.

1.4 Samples containing high levels of organics or sulfides cause rapid reduction of soluble Cr(VI) to Cr(III).

1.5 This method should be used by analysts experienced in the use of ion chromatography and in the interpretation of ion chromatograms.

1.6 Current reporting limits:

	Drinking Water	Water	Soils
218.7	0.03 ug/L	NA	NA
218.6	0.02 ug/L	0.02 ug/L (LL) 0.01 mg/L (RL)	NA
7199	NA	0.01 mg/L	0.40 mg/kg
NIOSH 7605	see GEN-N7605		

1.7 Current MDLs:

	Drinking Water	Water	Soils
218.7	0.01 ug/L	NA	NA
218.6	0.01 ug/L	0.01 ug/L (LL) 0.001 mg/L (RL)	NA
7199	NA	0.001 mg/L	0.026 mg/kg
NIOSH 7605	see GEN-N7605		

2) Summary of Procedure

- Waters by 218.6 and 218.7 are preserved with a combined buffer/dechlorinating reagent which complexes free chlorine and increases the pH to a specified value (see Preservation Section). Water samples by 7199 are not chemically preserved with the buffer. Soils are digested by EPA 3060A (GEN-3060A). Air filters are digested by NIOSH 7605 (GEN-N7605).
- A measured volume of the sample is introduced into an ion chromatograph. CrO42- is separated from other matrix components on an anion exchange column. CrO42- is derivatized with 1,5-diphenylcarbazide in a post-column reaction and is detected spectrophotometrically at a wavelength of 530 nm. Cr(VI) is qualitatively identified via retention time, and the concentration of CrO42- in the sample is calculated using the integrated peak area and the external standard technique.

3) Definitions

3.1 Initial Calibration - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the system.

3.2 Matrix Spike (MS) - In the matrix spike analysis, a predetermined quantity of standard solution is added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recovery is calculated for the analyte detected. EPA methods 218.6 and 218.7 call the matrix spike a Laboratory Fortified Sample Matrix (LFM or LFSM).

3.3 Soluble Matrix Spike (MS-Sol) - Only applicable to soil preparation batches digested by GEN-3060A. In the soluble matrix spike analysis, a predetermined quantity of a soluble standard solution of the analyte is added to a sample matrix prior to sample digestion and analysis. The purpose of the soluble matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recovery is calculated for the analyte detected

3.4 Insoluble Matrix Spike (MS-Insol) - Only applicable to soil preparation batches digested by GEN-3060A. In the insoluble matrix spike analysis, a predetermined quantity of an insoluble standard of the analyte is added to a sample matrix prior to sample digestion and analysis. The purpose of the insoluble matrix spike is to evaluate the dissolution during the digestion process and effects of the sample matrix on the dissolution. Percent recovery is calculated for the analyte detected.

3.5 Duplicate Sample (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision. EPA methods 218.6 and 218.7 use the abbreviation LD (Laboratory Duplicate)

3.6 Relative Percent Difference (RPD) – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.7 Method Blank (MB) - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire preparation and analytical procedure. For waters by 218.7, the MB is the Laboratory Reagent Blank (LRB) or CCB and contains the method preservative.

3.8 Laboratory Control Sample (LCS) - In the LCS or blank spike analysis, a predetermined quantity of standard solution is added to a blank prior to sample analysis. Percent recovery is calculated for the analyte detected. This LCS is a check on the calibration only and has not undergone digestion. The LCS-Insol is a check on the preparation batch.

3.9 Blank Spike (LCS-Insol) – Only applicable to soil preparation batches digested by GEN-3060A. In the LCS-Insol analysis, a predetermined quantity of an insoluble standard solution is added to a blank prior to sample digestion and analysis. Percent recoveries are calculated for the analyte detected.

3.10 Post Verification Spike (PVS) – The PVS (sometimes referred to as PDS for Post Digestion Spike by New Jersey) analysis is designed to verify that neither a reducing nor a chemical interference is affecting the analysis. In the PVS analysis, a predetermined quantity of a soluble standard solution is added to a sample after sample digestion and analysis. Percent recoveries are calculated for the analyte detected.

3.11 Independent Calibration Verification/ Continuing Calibration Verification (ICV / CCV) – ICV/CCV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization. This is sometimes referred to by New Jersey as CCS (Calibration Check Standard), or QCS (Quality Control Sample). Method 218.7 calls the ICV a QCS and the CCV a CCC (Continuing Calibration Check).

3.12 Initial Calibration Blank (ICB) - A blank run immediately after calibration to determine if the instrument is adequately zeroed.

3.13 Continuous Calibration Blank (CCB) - A blank run periodically (every 10 samples after the CCV) to ensure the instrument is still zeroed adequately. The CCB may be used for the MB for waters.

3.14 Preparation Batch - Samples digested together as a unit, not to exceed 20 investigative samples. The Digested QC (PB, LCSs, MSs, DUP) are associated with the preparation batch and may be analyzed in separate analytical batches. See ADM-BATCH for further discussion.

3.15 Analytical Batch - Samples analyzed together as a unit, not to exceed 10 injections for 218.7 or 20 injections for 7199 and 218.6. This batch must contain all of the undigested or instrument QC and may not necessarily contain all of the digested QC associated with the samples in the analytical batch. Typically, the preparation batch QC is analyzed prior to the analysis of client samples to verify that the associated samples are valid for use. See ADM-BATCH for further discussion. The analytical sequence for 218.7 is attached.

3.16 Method Detection Limit (MDL): a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.17 Method Reporting Limit (MRL): The minimum amount of a target analyte that can be measured and reported quantitatively.

3.18 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 and may be laboratory-dependent. For DOD, 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%). At the LOD, the false negative rate (Type II error) is 1%.

3.19 Limit of Quantitation (LOQ)/Reporting Limit: The minimum levels, concentrations, or quantities of a target that can be reported with a specified degree of confidence. For DOD, the lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ shall be set at or above the concentration of the lowest calibration standard.

4) Health and Safety Warnings

4.1 All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

4.2 Chemicals, reagents and standards must be handled as described in the company safety policies, approved methods and in MSDSs where available. Refer to the Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

4.3 Refer to the Safety Manual for further discussion of general safety procedures and information.

4.4 Waste Management and Pollution Prevention

Hexavalent chromium solutions should be dumped in the red inorganic carboys which will later be emptied by qualified personnel. All other waste can be flushed down the drain with large amounts of water. See SMO-SPLDIS for further information on sample disposal.

It is the laboratory's practice to minimize the amount of acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the EH&S Manual.

5) Cautions

- See SOP GEN-300.0 for preventive maintenance of the instrument.
- Clean labware according to GEN-GC. NOTE: Chromic acid must not be used for the cleaning of labware.

6) Interferences

6.1 Contamination - A trace amount of Cr is sometimes found in reagent grade salts. Since a concentrated buffer solution is used in this method to adjust the pH of samples, reagent blanks are analyzed to assess for potential Cr(VI) contamination. Contamination can also come from improperly cleaned glassware or contact or caustic or acidic reagents of samples with stainless steel or pigmented material.

6.2 OXIDATION-REDUCTION (REDOX) CONCERNS – To ensure sample integrity, Cr(VI) must be protected from reduction, and Cr(III), if present, must not oxidize to Cr(VI) during sample storage or processing.

6.2.1 Within the normal pH range in drinking water, Cr(VI), present as a result of pollution or oxidation of Cr(III) in source water during treatment, forms oxyanions, which are typically represented as $HCrO_4^-$ and CrO_4^{2-}. The very stable CrO_4^{2-} anion dominates above pH 8; therefore, the method preservative is designed to buffer samples to at least pH 8. Chromate compounds are quite soluble, mobile and stable, particularly in an oxidizing

environment. In contrast, soluble Cr(III) species oxidize to Cr(VI) in the presence of free chlorine, although natural organic matter in surface water sources may complex Cr(III), slowing its oxidation even in a highly oxidizing environment. The rate of Cr(III) oxidation increases with chlorine concentration and is pH-dependent. For these reasons, the preservation includes ammonium ions to complex free chlorine. The resulting formation of chloramines minimizes, but does not completely prevent, the oxidation of Cr(III). EPA experiments have demonstrated the ability of the method preservative to minimize the oxidation of Cr(III) and to prevent the reduction of Cr(VI) for at least 14 days in drinking water from ground and surface water sources.

6.2.2 A reducing tendency of the sample matrix may change Cr (VI) to Cr(III). The reducing/oxidizing tendency of each sample may be characterized using additional analytical parameters, such as pH, ferrous iron, sulfides, and oxidation/reduction potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biochemical Oxygen Demand (BOD). Analysis of these additional parameters establishes the tendency of Cr(VI) to exist in the unspiked sample(s) and may be necessary to assist in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria. Reduction of Cr(VI) to Cr(III) can occur in an acidic medium. However, at a pH of 6.5 or greater, CrO42- (which is less reactive than the HCrO4-) is the predominant species.

6.3 Sample ionic strength may enhance or suppress Cr(VI) response; however, the 4-mm column systems used tolerate typical concentrations of common anions in drinking water in combination with method preservative. Acceptable method performance has been demonstrated by EPA for samples with hardness up to 350 mg/L as CaCO3 and total organic carbon content of 3 mg/L.

6.4 Overloading of the analytical column capacity with high concentrations of anionic species, especially chloride and sulfate, will cause a loss of Cr(VI). The column can handle samples containing up to 5% sodium sulfate or 2% sodium chloride. Poor recoveries from fortified samples and tailing peaks are typical manifestations of column overload.

7) Personnel Qualifications and Responsibilities

- Personnel must be trained according to CE-QA003.
- It is the responsibility of the Project Manager to obtain information from the client before sampling. The laboratory needs to know whether the client will require additional investigation of the sample matrix in the case of matrix spike failures. A reducing condition in the sample matrix will reduce Cr(VI) to Cr(III) causing low bias. Additional parameters – sulfide, pH, REDOX, ferrous irons, BOD, COD, TOC may be used to demonstrate a reducing condition in the sample matrix. If any or all of these parameters are to be analyzed, additional aliquots are to be sampled and the tests are to be scheduled when hexavalent chromium is scheduled due to holding time limitations. If pH and REDOX are to be analyzed, they are to be analyzed from the same DI extract. It is the responsibility of the Project Manager to appropriately flag the data and make notes in the case narrative about the nature of the matrix, if applicable (see the attached flowchart).
- It is the responsibility of the analyst to perform the analysis according to this SOP and

to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

8) Sample Containers, Collection, Preservations, and Storage

8.1 Bottleware - If the lab provides the bottleware, waters are sampled in certified clean 250-mL narrow-mouth, high-density polypropylene containers, or equivalent. Soils are sampled in certified clean 4 or 8 oz. glass soil jars. The client must provide additional aliquots of soil sample if further investigation into the reducing/oxidizing nature of the sample is to be done.

8.2 Pretesting – Pre-test each lot of the the preservation solution (buffer). Add 2.5 mL of the buffer to a 250 mL sample bottle and fill with DI. Preservative is acceptable if result is <MDL of the low level analysis.

8.3 Temperature Requirements - Samples are typically shipped and stored at 0-6°C. Drinking water samples for UCMR3 method 218.7 received within 48 hours of collection must be ≤10ºC or they must be rejected. If UCMR3 samples are received after 48 hours of collection they must be ≤6ºC or they must be rejected. The project manager is to notify the client that a new sample must be collected.

8.4 pH Measurements (laboratory) – Use a calibrated pH meter (see GEN-pHSupport). Triple rinse the probe between client samples by first rinsing with DI, then with dilute nitric acid, then with DI.

8.5 Preservation and Holding Time:

	Drinking Water 218.7	**Drinking Water 218.6**	**Non-Potable Water 218.6**	**Waters 7199**	**Soil 7199**	**Air filters NIOSH 7605**	**Air (impingers) collected by 0061**
Filter	No	Required see 6.4.1	Required see 6.4.1	Optional – Field or Lab	NA	NA	Field
Preservative	NH4OH/ (NH4)2SO 4 liquid	NH4OH/ (NH4)2SO4 liquid	NH4OH/ (NH4)2SO4 liquid	None	None	None	None
pH	>8.0	9.0-9.5	9.3-9.7	unpreserved	NA	NA	NA
Residual Chlorine	<0.1 mg/L	NA	NA	NA	NA	NA	NA
Holding Time	14 days	5 days	28 days	24 hours	7 days from extraction	28 days (recommend ed)	14 days

- Sample pH and residual chlorine (if applicable) is measured and recorded upon receipt. The procedure for checking the residual chlorine is in SMO-GEN. The preservation sheet and associated steps may be completed by SMO or Wetchem personnel. An example Preservation sheet is attached to this SOP. SMO also records the lot number of the bottle and preservative upon receipt.
- For 218.7, the sample is only valid if received pH>8.0 and Residual chlorine <0.1 mg/L. The sample must be rejected if it does not meet receipt requirements. It may not be reported for UCMR3.

- For 218.6, the holding times listed only apply if the sample is properly filtered and buffered within 24 hours of sampling. If the buffering requirements (pH) are not met, and the sample is still within 24 hours of collection, the pH is adjusted with the appropriate buffer or ammonium hydroxide.

8.5.1 For 218.6, samples shall be filtered and preserved with buffer solution within 24-hours of collection (preferably in the field at the time of collection). Procedures are as follows, listed in order of preference

8.5.1.1 Filter and Buffer in the Field during time of collection:

Filter sample aliquot using an in-line filter or plastic syringe filtration unit equipped with a 0.45um membrane filter (mfr. Gelman, Millipore, or equivalent).

- Non-Potable Water - Adjust the pH of the filtered sample to pH 9.3 - 9.7 using buffer solution provided by the laboratory. pH meter should be capable of ±0.03 SU. Holding time is 28-days from collection.
- Drinking Water - Adjust the pH of the filtered sample to pH 9.0 - 9.5 using buffer solution provided by the laboratory. pH meter should be capable of ±0.03 SU. Holding time is 5-days from collection.

The bottle set shall include unpreserved 125 or 250 mL plastic bottles. A minimum of 25 mL filtered sample should be provided for analysis.

8.5.1.2 Filter and Buffer in the Field during time of collection –No pH meter available:

Filter sample aliquot using an in-line filter or plastic syringe filtration unit equipped with a 0.45um membrane filter (mfr. Gelman, Millipore, or equivalent). Filtered sample shall be collected in a pre-preserved sample bottle containing buffer solution. Samples must be received by the laboratory within 24-hr of collection to ensure proper pH has been achieved by the buffer solution. The lab will adjust the pH, if necessary, within 24-hr of collection. Holding time is 28-days.

The bottle set shall include pre-preserved 125-ml plastic bottles with 1 mL buffer solution, or equivalent. A minimum of 25 mL filtered sample should be provided for analysis.

8.5.1.3 Filter and Buffer at the Laboratory –No filtration unit or pH meter available:

Collect sample in an un-preserved plastic bottle. Samples must be received by the laboratory within 24-hr for immediate analysis, or in-lab filtration and pH adjustment with buffer solution. Holding time is 28-days for non-potable water and 5 days for drinking water if properly filtered and buffered.

The bottle set shall include unpreserved 125 or 250-mL plastic bottles.

8.6 Method 7199 water sample: If it is impossible to complete 2 simultaneous injections of all samples within the 24 hour holding time, perform the injections separately in order to meet one injection within holding time of as many samples as possible.

8.7 If investigation is requested, the investigative tests (such as sulfide, pH, REDOX and Fe2+) must be scheduled with the Cr6+ test because some of the investigative test have shorter holding times than hexavalent chromium. These tests must be completed prior to the evaluation of the Cr6+ QC so that the results are available if needed.

8.8 Sample handling, storage, receipt, and custody procedures are discussed in SMO-GEN and SMO-ICOC.

9) Equipment and Supplies

Instrument ID	Instrument Configuration	Manufacturer Part	Serial Number
IC # 5 (R-IC-05)	Ion Chromatograph	Dionex ICS-1000	7090145
	Gradient Pump	GP40	
	Conductivity Detector	DS6	7081071
	Autosampler	ASDV	12090422
	Computer Workstation	Dell Optiplex 745	1441DAA99
	Analytical Software	Chromeleon 6.80	56276
IC #8 (R-IC-08)	Ion Chromatograph	Dionex ICS-2100	12030901
	Heated Conductivity Cell	DS6	12030664
	Reagent Pump	AXP	20045075
	Variable Wavelength Detector	ICS Series VWD	12031294
	Autosampler	AS-DV	12111147
	Loop	1000 uL	
	Analytical Column	Dionex AS-7 2x250mm	
	Computer Workstation	Dell Optiplex 790	15105322945
	Analytical Software	Chromeleon 7.0	151838

9.1.1 Reaction Coil: Dionex P/N 042631 750 uL

9.1.2 Column Heater - integrated

9.1.3 Pressurized eluent container, plastic, two liter size.

9.1.4 Nitrogen Tanks

9.2 Labware

9.2.1 Class A volumetric flasks, and graduated cylinders.

9.2.2 Assorted pipettes - of acceptable precision and accuracy – calibrated according to ADM-PCAL.

9.2.3 Disposable syringes - 50-mL, with male luer-lock fittings.

9.2.4 Dionex ONGuard-P sample pretreatment cartridges - p/n 39597

9.2.5 Syringe filters - 0.45-µm, Millipore, p/n SLHV 025 NK.

9.2.6 Storage bottles - high density polypropylene or amber glass, 1-L capacity.

9.2.7 Orion Model 720A pH meter or Orion SA 520 pH meter, or equivalent, calibrated according to ADM-phSupport, with accuracy ± 0.05 pH.

9.2.8 Analytical and TopLoading Balances – calibrated according to ADM-DALYCK.

10) Standards and Reagents

10.1 All standards must be traceable using the laboratory lot system (CE-QA007)

10.2 All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request.

10.3 Purchased Reagents and Standards - store at room temperature and expire per Expiration Policy (CE-QA012) unless otherwise indicated.

10.3.1 Ammonium hydroxide, NH4 OH. EM Science cat. #AX1303-14, or equivalent, sp.gr. 0.902, CAS RN 1336-21-6.

10.3.2 Ammonium sulfate, (NH4)2SO4 . EM Science cat #SX0597-1, or equivalent, CAS RN 7783-20-2

10.3.3 1,5-Diphenylcarbazide. EM Science cat. #DX2205-1, or equivalent, CAS RN 140-22-7.

10.3.4 Methanol, HPLC grade, EM Science cat #MX0475-1, or equivalent

10.3.5 Sulfuric acid, concentrated, EM Science, Omnitrace, cat #SX1247-2 or equivalent.

10.3.6 Cr(VI) Standards Stock Solution (1000 mg/L)

10.3.7 Cr(VI) Reference Stock Solution (1000 mg/L) – the Reference Stock is to be from a separate manufacturer of the Standard Stock.

10.3.8 Chlorine Residual Test Strips capable of reading to 0.1 mg/L. HF Scientific Micro Check Test Strips.

10.3.9 Nitric Acid, HNO3, Metals grade.

10.4 Prepared Reagents

10.4.1 Buffer Solution. Dissolve 3.3 g of ammonium sulfate in 75 mL of reagent water in a 100 mL volumetric flask. Add 6.5 mL of ammonium hydroxide. Dilute to volume (100 mL) with reagent water. Degas the solution with helium gas for 5-10 minutes prior to use. Store at 0-6°C for up to one year.

10.4.2 Buffered DI (Dilution Water). A batch of reagent grade water must be prepared by adjusting the pH within the range of 9-9.5 using the buffer solution. Use this solution for diluting working standards and high level samples. Prepare fresh before use. pH range of 9.3-9.7 shall be used for Method 218.6 waters as per Footnote 20 of EPA Method Update Rule and FAQ-Cr6.

10.4.3 Dilute Nitric Acid Rinse – First prepare a 1:1 dilution of concentrated HNO3. To a 500 mL rinse bottle, add 1 mL of 1:1 Nitric Acid and dilute to ~500 mL with DI.

10.4.4 Eluent.- Dissolve 33 g of ammonium sulfate in 500 mL of DI and add 6.5 mL of ammonium hydroxide. Dilute to one liter with reagent water. Degas the solution with helium gas for 5-10 minutes prior to use. Expires 1 month from preparation.

10.4.5 Post-column reagent. Dissolve 0.5 g of 1,5 diphenylcarbazide in 100 mL of HPLC grade methanol in a 1000 mL volumetric flask. In a separate container, add about 500 mL DI, then add 28 mL of 98% sulfuric acid, mix and degas with helium gas for 5-10 minutes. Carefully combine the degassed acid with the diphenylcarbazide/methanol solution, introducing as little air as possible. This method of preparation reduces the frequency and intensity of air spikes in the chromatography. Dilute to volume with reagent water. Store in amber glass at 0-6°C. This reagent should be made fresh for the low level analysis, and may be kept no longer than 5 days.

10.5 Standards: 7199/218.6 Regular Level. Prepare fresh before use.

10.5.1 Regular Level Intermediate Standards Working Stock (10 mg/L). Do two 1/10 serial dilutions of the 1000 ppm standard stock solution, using buffered DI.

10.5.2 Initial Calibration Standards: Prepare a series of standards and a blank by pipetting suitable volumes of the Intermediate Standards Working Stock and buffered DI into a dispo cup.

The typical calibration for regular level (7199 and 218.6) is as follows:

Standard #	Volume (mL) of 10.0 mg/L Standard	Volume buffered DI (mL)	Final Concentration (mg/L)
6	1.00	9.0	1.00
5	0.70	9.3	0.70
4	0.50	9.5	0.50
3	0.10	9.9	0.10
2	1/10 of #3	-	0.01
1	0.0	10	0.000

*Note – if 1ppb MRL is requested, this curve is prepared, but an additional standard is added (1 ppb)

10.5.3 Regular Level Intermediate Reference Working Stock (10 mg/L) - Do two 1/10 serial dilutions of the 1000 ppm reference stock solution, using buffered DI (as above).

10.5.4 Regular Level ICV/CCV (0.5 mg/L): In a 10mL dispo cup add 9.5 mL buffered DI (as above) and 0.5 mL Intermediate Reference Working Stock.

10.5.5 ICB / CCB / Method Blank / LRB (waters): DI preserved as a sample.

10.5.6 LCS - Regular Level Waters (0.20 mg/L): Add 0.20 mL of 10 mg/L Intermediate Standards Working Stock to 9.8 mL buffered DI. Analyze as a sample.

10.5.7 MS Regular Level Waters (0.2 mg/L): Add 0.20 mL of 10 mg/L Intermediate Standards Working Stock to 10 mL sample.

10.5.8 MB/LCS/MS Soils:prepared and digested according to GEN-3060A.

10.5.9 Post Verification Spike (PVS) – soil extracts only - after digestion, filtration, pH adjustment to 9.0-9.5, and dilution to 100 mLs (as described in GEN-3060), add 0.45 mL of 100 mg/L Cr(VI) standard stock solution to a 45 mL aliquot (this is equal to 40 mg/Kg) OR a concentration twice the original sample, whichever is higher.

10.6 Standards: 218.7 Prepare fresh before use.

10.6.1 1000 ug/L Standards Working Stock - Add 75 mL of DI to a 100 mL volumetric flask. To this add 1.0 mL Buffer Solution. Add 0.1 mL 1000 mg/L Cr(VI) Standard Stock Solution. Bring to volume with DI. Prepare fresh before use. Mix thoroughly.

10.6.2 100 ug/L Standards Working Stock - Add 75 mL of DI to a 100 mL volumetric flask. To this add 1.0 mL Buffer Solution. Add 10 mL 1000 ug/L Cr(VI) Standard Working Stock Solution. Bring to volume with DI. Prepare fresh before use. Mix thoroughly.

10.6.3 Initial calibration standards: For each standard, add 75 mL DI to a 100 mL volumetric flask. To this add 1/0 mL Buffer Solution. Then follow the recipe below:

Standard #	Volume (mL) of 1000 µg/L Standard	Final Volume with DI (mL)	Final Concentration (µg/L)
8	0.5	100	5.00
7	0.1	100	1.00
6	0.7 mL of 100 ug/L Std	100	0.70
5	0.5 mL of 100 ug/L Std	100	0.50
4	0.3 mL of 100 ug/L Std	100	0.30
3	0.1 mL of 100 ug/L Std	100	0.10
2	10 mL Std#4	100	0.030
1	10 mL Std#3	100	0.010

10.6.4 MS Spiking Stock (10 µg/L). Perform two 1/10 serial dilutions of the 1000 µg/L Standard Stock using DI.

10.6.5 1000 ug/L Reference Working Stock - Add 75 mL of DI to a 100 mL volumetric flask. To this add 1.0 mL Buffer Solution. Add 0.1 mL 1000 mg/L Cr(VI) Reference Stock Solution. Bring to volume with DI. Prepare fresh before use. Mix thoroughly.

10.6.6 ICV/CCVs:

10.6.6.1 Low Level (LL-CCV): 0.03 ug/L –

- 100 ug/L Reference Working Stock - Add 75 mL of DI to a 100 mL volumetric flask. To this add 1.0 mL Buffer Solution. Add 10 mL 1000 ug/L Reference Stock Solution. Bring to volume with DI. Prepare fresh before use. Mix thoroughly.
- 0.30 ug/L Reference Working Stock - To a 100 mL volumetric flask add about 75 mL DI. To this add 1.0 mL Buffer Solution and 0.3 mL of the 100 ug/L Reference stock. Bring to volume with DI. Prepare fresh before use. Mix thoroughly.
- To a separate 100 mL volumetric flask, add about 75 mL DI. To this add 1.0 mL Buffer Solution and 10 mL of the 0.30 ug/L Reference Working Stock. Bring to volume with DI. Prepare fresh before use. Mix thoroughly.

10.6.6.2 Mid Level CCV (ML-CCV) / ICV: 2.5 ug/L – To a 100 mL volumetric flask, add about 50 mL DI. To this add 1.0 mL Buffer Solution and 0.25 mL of 1000 ug/L Reference stock. Bring to volume with DI and mix. Prepare fresh before use.

10.6.6.3 High Level CCV (HL-CCV): 4.0 ug/L - to a 100 mL volumetric flask, add about 50 mL DI. To this add 1.0 mL Buffer Solution and 0.4 mL of 1000 ug/L Reference. Bring to volume with DI and mix. Prepare fresh before use.

10.6.7 ICB / CCB / Method Blank / LRB (waters): In a 250 mL volumetric flask, add 2.5 mL liquid preservative to about 100 mL DI. Bring to volume with DI and transfer to a 250 mL HDPE sample bottle, received from SMO.

10.6.8 MS/LFSM - Add 0.20 mL of 10 µg/L Low Level LCS/MS Spiking Stock to 10 mL pH adjusted sample.

10.7 Standards: 218.6 Low Level –prepare fresh before use

10.7.1 1000 ug/L Standards Working and Reference Stocks – same as 218.7.

10.7.2 100 ug/L Standard Working Stock – make a 1/10 dilution of the 1000 ug/L Standard Working stock.

10.7.3 ICAL standards –

Standard #	Volume (mL) of 1000 µg/L Standard	Final Volume with buffered DI (mL)	Final Concentration (µg/L)
6	0.1	100	1.00
5	0.7 mL of 100 ug/L Std	100	0.70
4	0.5 mL of 100 ug/L Std	100	0.50
3	0.2 mL of 100 ug/L Std	100	0.20
2	10 mL of Std#6	100	0.10
1	10 mL Std#3	100	0.020
0	0	100	0

10.7.4 LCS/MS Spiking Stock (10 µg/L). Perform two 1/10 serial dilutions of the 1000 µg/L Standard Stock.

10.7.5 ICV/CCV (0.5 µg/L): To 5 mL buffered DI (as above) add 5 mL 1.0 µg/L Low Level Intermediate Reference Working Stock.

10.7.6 LCS (0.20 µg/L): Add 0.20 mL of 10 µg/L Low Level LCS/MS Spiking Stock to 9.8 mL buffered DI. Analyze as a sample.

10.7.7 Matrix Spike(0.20 µg/L): Add 0.20 mL of 10 µg/L Low Level LCS/MS Spiking Stock to 10 mL pH adjusted sample.

10.7.8 ICB/CCB/MB – same as 218.7

11) Method Calibration

11.1 Follow policies in ADM-ICAL unless otherwise specified in this SOP.

11.2 Initial Calibration

11.2.1 For New Jersey - Inject a calibration blank. Be sure this calibration blank is less than the MDL before continuing.

11.2.2 Number of Standards - Calibrate the instrument using the standards prepared as per the Standards and Reagents Section. Six or more standards must be used for 218.7. Three or more standards must be used for 218.6. A blank and three or more standards must be used for 7199. If a quadratic fit is to be used, analyze at least 6 standard levels plus blank.

11.2.3 Frequency – Initially and whenever continuing calibration verification criteria cannot be met.

11.2.4 Calibration Fit– Construct a calibration curve of analyte response (peak area) versus analyte concentration. The curve may be first order (y=mx+b), or second order (quadratic). Weighting may be used (1/x).

11.2.5 Limits –

11.2.5.1 For first order polynomial, the coefficient of correlation must be 0.999 or greater. File the printout of the linear regression with the ICAL.

11.2.5.2 For second order, mark the linear regression printout so that it is clear that the linear was for verification only. The accuracy of the quadratic calibration is verified by assessing the recovery of each standard. The recovery of each standard above the LOQ must be within 10% for 218.6 and 7199.

11.2.5.3 The LOQ standard must be within ±50% of the true value.

11.2.5.4 For 218.7, all standards above the LOQ must be within ±15% of the true value (regardless of curve type).

11.2.6 ICV(QCS) - Analyze an ICV immediately after the calibration standards. The ICV must be within limits to use the curve. The limits are 90-110% for 7199, 95-105% for 218.6, and 85-115% for 218.7. If the correctly prepared ICV is not compliant, the second source standard does not verify the curve. Fix the problem and recalibrate. If the ICV was prepared incorrectly, the curve may be used if a correctly prepared ICV verifies the curve.

11.3 Continuing Calibration Verification and Blank

11.3.1 Frequency - run a CCV and CCB set to start a daily run, every ten injections (or 24 hours, whichever is more frequent), and at the close of the run. For water samples, one blank counts as both the CCB and the MB. For 218.7, the CCV concentration varies (see attached Analytical Sequence and Standards and Reagents Section).

11.3.2 Limits –

Method	CCV % Recovery
7199	90-110
218.6	95-105
218.7	85-115
NIOSH 7605	85-115

Method	ICB/CCB/MB Limit
7199 7605	<MRL
7199 NJ	<MDL
218.6	<LOD
218.7	<0.01 ug/L
NIOSH 7605	<MRL
DOD	<1/2 LOQ

11.3.3 CCV Corrective Action - If a CCV fails, corrective actions must be performed. If routine corrective action fails to produce a second consecutive (immediate) acceptable CCV, then either the lab has to demonstrate acceptable performance after corrective action with two consecutive CCVs or a new ICAL must be performed.

11.3.4 Blank Corrective Action - Sample results greater than 10 times the result of the contaminated blank may be reported without qualification. Samples <10x blank contamination must be reanalyzed with a compliant blank whenever possible. If samples associated with a contaminated blank are not repeated (due to holding time restrictions, etc.), the data must be qualified on the report.

12) Sample Preparation and Analysis

12.1 Sample preparation.

12.1.1 Dilutions – Document the preparation of dilutions on the Responsibility Report in real-time as the dilutions are being made. Also write the dilution on the autosampler vial. Transfer the dilution information to the analytical software queue. When the samples come off of the autosampler, remove one-by-one and check the entry in the software against the notation on the vial. If historical results are provided to the analyst, the analyst must use the historical results to determine an initial dilution. If the instrument result does not compare well (within 2x) with the historical, the sample must be repeated to verify the dilution. The secondary reviewer is to compare the dilution written on the responsibility report to the dilution entered to LIMS.

12.1.2 Waters - Allow pH-adjusted samples to equilibrate to ambient temperature prior to analysis. Samples that have not been pH adjusted should be adjusted to pH 9-9.5 by dropwise addition of buffer solution. Record the pH adjustment and the pH meter ID on the benchsheet. If salts are formed as a result of the pH adjustment, the filtrate must be filtered again prior to analysis. pH range for 218.6 non-potable waters shall be 9.3-9.7 due to footnote 20 of EPA method update rule and FAQ-Cr6. All drinking waters are

to be pH 9.0-9.5. Triple rinse the pH probe between samples per Section 8 when measuring pH.

12.1.3 Soils – Digest according to GEN-3060A except pH adjust the sample to 9.0-9.5 instead of 7.5±0.5.

12.1.4 Air Filters – Prepare according to GEN-7605.

12.2 Establish Operating Conditions:

	IC #5	IC #8
Warm up	45-60 minutes	45-60 minutes
Eluent flow rate	1.0 mL/min	0.36 mL/min
Post-column flow rate	~0.33 mL/min	~0.12 mL/min
Column heater	30ºC	30ºC
Sample loop	2000 uL (LL) or 100 uL (RL)	1000 uL
Wavelength	530 nm	530 nm

Check flow rate of waste after the flow cell prior to calibration and sample analysis.

12.3 Sample Analysis.

12.3.1 Standards and samples are injected onto the column using an autosampler.

12.3.2 Color Interference - The guard column should be removing any inherent color in samples. If, however, a sample appears colored after elution through the columns, it is possible that not all sample color/organic material was removed and a false positive due to color could occur. At this point, pass a sample aliquot through Dionex ONGuard-P syringe filters and re-analyze. Alternatively, re-analyze a sample aliquot, but replace the post-column reagent with the matrix match reagent and subtract the matrix-match reagent result from the post-column reagent result.

12.3.3 Double Injection – 7199 requires that samples are injected twice (double injections are not required for 218.6 nor 218.7). The RPD between the samples must be less than 20 if the sample concentration is ≥ four times the reporting limit. A control limit of ± the reporting limit is used when the sample concentration is < four times the reporting limit. If it is impossible to meet holding times for both injections, it is best to inject samples once within holding time and make the second injection out of holding time. Report both results. Label the comments field R1, R2, R3, R4, etc.

12.3.4 Salinity – High salinity can cause the Cr6+ peak to shift outside of the established retention time window. Dilute all ocean water samples at least 50X and run on the low level curve. Spike (MS/MSD) one ocean water sample in each Folder containing ocean water samples.

12.4 Sample Evaluation –

12.4.1 Each chromatogram is reviewed for compliance and initialed by the reviewer. All chromatographic baselines should be examined by a knowledgeable analyst to ensure that proper integrations have been made by the analytical software. Especially for the low level analysis, care must be taken to ensure proper identification and integration of all low level peaks approaching a signal to noise ration of 3:1. When the data system incorrectly quantitates or identifies analytes, manual integration is necessary. Data must be integrated consistently between standards, samples, and QC. See CE-QA002 for

integration requirements and manual integration documentation.

12.4.2 Samples or extracts exceeding the highest calibration standard (including PVS) must be diluted using buffered DI and re-analyzed.

12.4.3 Sample must be bound by acceptable analytical QC and from a batch with acceptable batch QC. Double injection must meets limits.

13) Troubleshooting

13.1 See Instrument manual or maintenance log for help with solving specific analytical or instrument problems.

13.2 Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by laboratory staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

14) Data Acquisition

- Data is uploaded electronically from the instrument to LIMS. Data is uploaded from the instrument into LIMS. All chromatograms are visually inspected by both a primary and secondary reviewer. The IC Checklist serves as an attestation by both reviewers that all chromatograms were reviewed. Any manual integrations and/or changes to chromatogram(s) are to be documented on said chromatogram(s) and signed off by both parties, as outlined in the SOP for manual integration (CE-QA002).

15) Calculation and Data Reduction Requirements

15.1 Determine the concentration of the injected sample from the calibration curve.

15.1.1 For waters, multiply the injected concentration by dilution (use 1 if there is no dilution). Report in mg/L except report in µg/L for low level 218.6 and 218.7.

15.1.2 For digested soils:

$$\text{Concentration (mg/Kg dry wt.)} = \frac{A \times D \times E}{B \times C}$$

where: A = Concentration observed in the digest (mg/L)
B = Initial moist sample weight (g)
C = % Solids/100
D = Dilution factor
E = Final digest volume (mL)

15.1.3 For air strips (NIOSH 7605) – see GEN-N7605.

15.2 Report both of the results from the double injection (if applicable).

15.3 For NJ - Data is "R" flagged if the MS is outside of 50-150%, if CCV or LCS is out of

control, if calibration CC<0.999, if calibration blank >MDL, if PB >MDL for samples >MDL, if a water sample is run beyond 48 hours from sampling, if required QC is not performed, or if a soil sample is not redigested as required.

15.4 For NJ - Data is "J" flagged if the result is run between 24 and 48 hours from sampling (waters only), if QC is not performed at the correct frequency, if the MS is outside of limits but within 50-150%, if the RPD of a double injection or a DUP is greater than 20, if digested QC fails the initial and the redigestion, or if PDS <85%.

15.5 If samples are redigested, report as replicates. Both original and redigested data is reported.

15.6 All sample data and QC data, including calibration verification must reference the name (date or filename) of the ICAL on the raw data report. The current system lists the ICAL under the heading of "Quantif. Method" and the convention is to use the IC# and date in the name. For Example: "5-121610" is IC#5 on 12/16/10.

15.7 Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV.

16) Quality Control, Acceptance Limits and Corrective Action

16.1 ICV/CCV and ICB/CCB/MB requirements are in the Calibration Section.

16.2 For Water Samples

16.2.1 Matrix Spike –

16.2.1.1 Frequency - A minimum of one matrix spike sample per sample batch (1/10 for 218.6) must be analyzed to check for matrix interference.

16.2.1.2 Limits - The recovery of the matrix spike must be within limits in the Data Quality Objectives Table. For 218.7 – the MS must be within 15%.

Method	MS Limit
7199	DQO Table
7199 NJ	DQO Table
218.6	90-110
218.7	85-115

16.2.1.3 Corrective Action - If the matrix spike recovery fails these limits, report with appropriate qualifiers.

16.2.2 DUP/MSD

16.2.2.1 Frequency - A minimum of one duplicate sample per sample batch must be analyzed to check for precision. Alternatively, a Matrix Spike Duplicate may be used instead of a Duplicate. The MSD is required for UCMR3.

16.2.2.2 Limits:

	RPD	Other
7199	<20	+/-MRL if <4xMRL
218.6	<20	+/-MRL if <4xMRL
218.7	<15	NA

16.2.2.3 Corrective Action - If the RPD is out of limits, repeat the sample and duplicate unless there is assignable matrix interference, historical failures, or lack of volume. If an out of control duplicate is not repeated, note the reason on the data quality checklist. If, at the time when the problem is discovered, the sample exceeds twice the holding time, discuss with supervisor or Project Manager prior to repeating the samples. Report all of the replicates and explain in the checklist for the case narrative.

16.2.3 LCS -

16.2.3.1 Frequency –An LCS must be analyzed with every batch for 7199 and 218.6. An LCS is not required for 218.7 (performance measured with CCV). See GEN-N7605 for air strips.

16.2.3.2 Limits –

Method	Limits
7199	DQO Table
218.6	90-110%
218.7	NA

16.2.3.3 Corrective Action - If the LCS fails these limits, correct the problem and reanalyze the affected samples or flag the associated data.

16.3 For Samples Digested by GEN-3060A – see also the attached flowchart. Undigested QC (LCS, MB, DUP, MS) is not required to be analyzed with the digested QC in a 7199 run which only has digested samples.

16.3.1 MB - A preparation blank must be prepared and analyzed with each digestion batch. Detected Cr(VI) concentrations must be less than the reporting limit (less than the MDL for New Jersey) or the batch must be redigested and reanalyzed. If the samples are out of holding time, redigest and reanalyze and both sets of data will be reported. If insufficient sample volume necessitate the use the data, flag the data associated with the non-compliant PB (the entire preparation batch – not just those in the analytical batch).

16.3.2 LCS Insoluble – An insoluble LCS must be prepared and analyzed with each digestion batch. Recovery must be within 80-120% or the entire sample batch must be redigested and reanalyzed. If the samples are out of holding time, redigest and reanalyze and both sets of data will be reported. If insufficient sample volume necessitate the use the data, flag the data associated with the non-compliant LCS (the entire preparation batch – not just those in the analytical batch)

16.3.3 DUP - A separately prepared duplicate soil sample must be analyzed at a frequency of one per batch. Duplicate samples must have a Relative Percent Difference (RPD) of ≤ 20%, if the sample concentration is ≥ four times the reporting limit. A control limit of ± the reporting limit is used when the sample concentration is < four times the reporting limit. If the RPD of the duplicates is out of limits, repeat the sample and duplicate unless there is assignable matrix interference, historical failures, or lack of volume. If an out of control duplicate is not repeated, note the reason on the data quality checklist. If, at the time when the problem is discovered, the sample exceeds twice the holding time, discuss with supervisor or Project Manager prior to repeating the samples. Report all of the replicates and explain in the

checklist for the case narrative.

16.3.4 MS- Both soluble an insoluble digested matrix spikes must be analyzed at a frequency of one each per batch. Both matrix spike recoveries must be within 75-125% of the true value. If either of the matrix spike recoveries are not within these recovery limits the entire batch must be redigested and reanalyzed. If the reanalysis also fails the 75-125% limit, sample data is flagged. Exception – if the sample concentration is greater than 4 times the spike concentration, the spike is "diluted out" and is not used to evaluate the batch (redigestion is not required). The client should be notified of the possible condition of the sample and further investigation is needed using the oxidation/reduction parameters discussed in 4.1. If the samples are out of holding time for the reanalysis, redigest and reanalyze and both sets of data will be reported. If insufficient sample volume necessitate the use the data, flag the data associated with the non-compliant LCS (the entire preparation batch – not just those in the analytical batch).

16.3.5 A post-digestion Cr(VI) matrix spike must be analyzed per batch, whether or not the MS passed or failed. Use the attached PVS Calculation sheet. The criteria for the post digestion matrix spike recovery is 85-115% recovery. If spike recovery is outside limits, and the matrix spike also failed, no further action is needed apart from the corrective action for the MS. The corrective actions will show whether these digestates may contain soluble reducing agents for Cr(VI). If the PVS fails and the MS passes, reanalyze the PVS.

17) Data Records Management

- See CE-GEN003 and ADM-ARCH

18) Contingencies for Handling Out of Control Data

- If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, data is flagged with the appropriate data qualifiers.

19) Method Performance

19.1 Reporting limits are based upon an MDL study performed according to CE-QA011 and filed in the MDL binders in the QA office.

19.2 Demonstration of Capability is performed according to CE-QA003.

19.3 From the EPA Method 7199:

- Single Laboratory Precision and Accuracy is available in Table 3
- Single Analyst Precision, overall precision and Recovery from Multilaboratory Study is available in Table 4.

20) Summary of Changes

- Incorporated change forms for DOD requirements, Ocean Water, signing of chromatograms, and triple rinsing pH probe.
- 12.1.1 - Incorporated change form for dilution factor verification. Further expanded on requirements for dilution factor records, review, and historical verification.

- Added IC#5
- Added 0.5ppb option to Regular level curve

21) References and Related Documents

- Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW-846.
- NJDEP Standard Operating Procedure (SOP No.5.A.10) Dated August 15, 2005: Standard Operating Procedure for Analytical Data Validation of Hexavalent Chromium.
- Methods for the Determination of Metals in Environmental Samples, Supplement I. EPA/600/R-94/111. May 1994. Method 218.6 revision 3.3.
- Method 218.7: Determination of Hexavalent Chromium in Drinking Water by Ion Chromatography with Post-Column Derivatization and UV-Visible Spectroscopic Detection. EPA Document Number EPA 815-R-11-005. Version 1.0, November 2011.
- NIOSH Manual of Analytical Methods, Fourth Edition, Method 7605 Issue 1, March 15, 2003.
- Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.

22) Attachments

- Table 3 Single Laboratory Precision and Accuracy
- QC Flowchart
- PVS Calculation Sheet
- PH Adjustment Sheet – controlled separately on the Controlled Forms section of the Rochester Intranet.
- eH/pH diagram
- Analytical Sequence

TABLE 3
SINGLE-LABORATORY PRECISION AND ACCURACY

Sample Type	Cr(VI) (µg/L)[a]	Percent Mean Recovery	RPD[b]
Reagent Water	100	100	0.8
	1000	100	0.0
Drinking Water	100	105	6.7
	1000	98	1.5
Ground Water	100	98	0.0
	1000	96	0.8
Primary Sewage	100	100	0.7
Wastewater	1000	104	2.7
Electroplating	100	99	0.4
Wastewater	1000	101	0.4

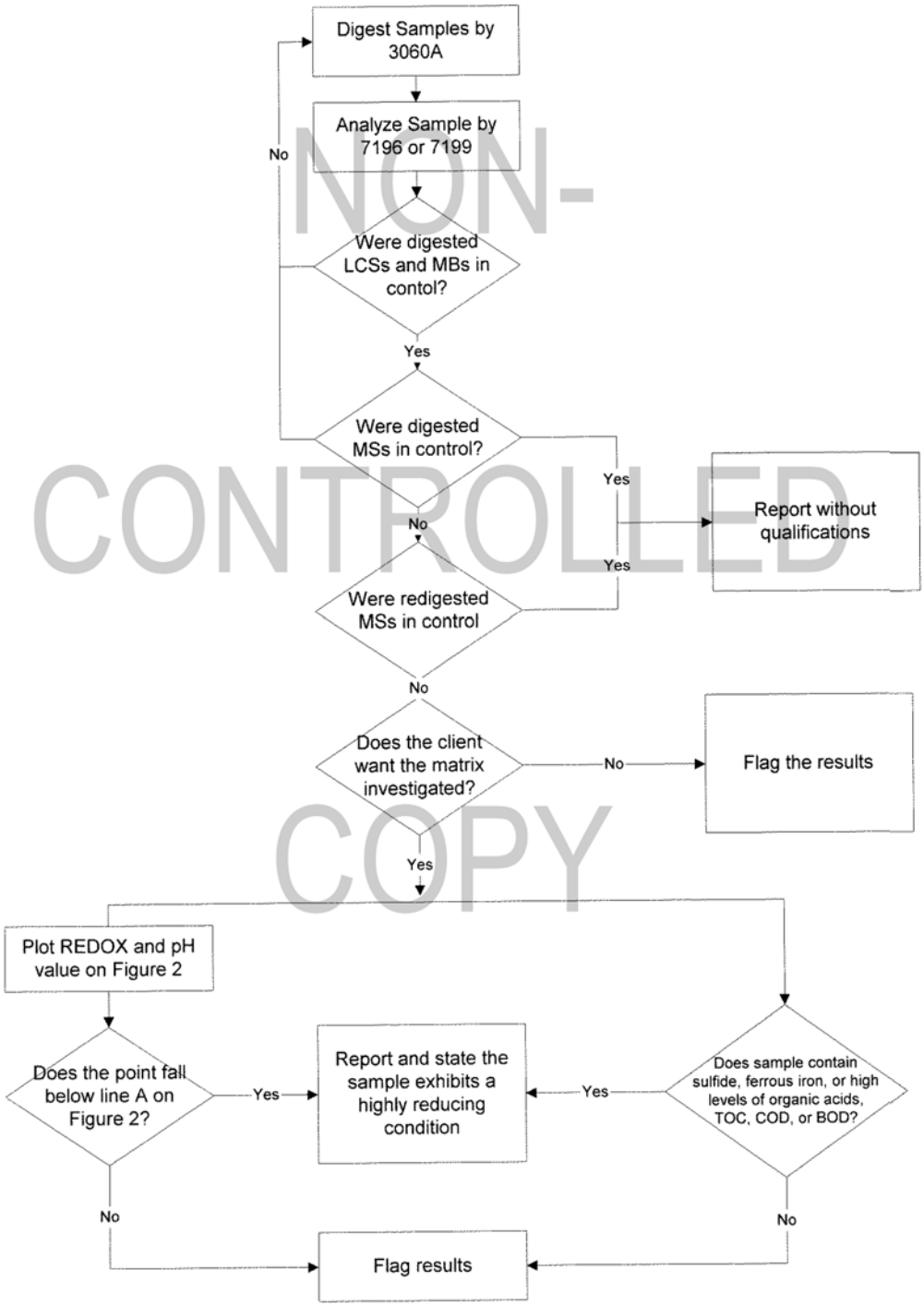
[a] Sample spiked at this concentration level.
[b] RPD - relative percent difference between duplicates.

TABLE 4
SINGLE-ANALYST PRECISION, OVERALL PRECISION AND RECOVERY
FROM MULTILABORATORY STUDY

	Reagent Water (6-960 µg/L)	Matrix Water (6-960 µg/L)
Mean Recovery	X = 1.020C + 0.592	X = 0.989C - 0.411
Overall Standard Deviation	S_R= 0.035X + 0.893	S_R= 0.059X + 1.055
Single-Analyst Standard-Deviation	S_R= 0.021X + 0.375	S_R= 0.041X + 0.393

X = Mean concentration; µg/L, exclusive of outliers.
C = True value, µg/L.
S_R= Overall standard deviation.
S_R= Single-Analyst standard deviation.

HEXAVALENT CHROMIUM
QUALITY CONTROL FLOW CHART



ALS Environmental
Rochester, NY

Analyst: _____
Date: _____
Pipet ID: _____

Hexavalent Chromium

Method: _____EPA 7196A, Manual Colorimetric
_____ 7199, IC
_____218.6, IC

Post-Verification Spike (PVS) Calculations

True Value = $\frac{A \times B}{C}$

Note: Calculations based on sample result and PVS expressed in mg/L before dilution or digest factors

Order #	Original Result mg/L on curve	Vol. (mL) of Spk Added (A)	Conc. (mg/L) of Spk Sol'n (B)	Digestate Vol. (mL) spk'd (C)	PVS True Value (mg/L)	Spike Result (mg/L)	Percent Recovery

Date/Time Received	Sample ID	Analysis	Matrix	Date/Time Sampled	Sample Filtered	Filter Lot ID	Chlorine Residual (mg/L) 218.7 only	pH at Receipt	pH Adjustment	Analyst/ Date/ Time pH Adjustment	Solution Used For PH Adjust	Solution Lot ID
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	
		7199 218.6 RL 218.6 LL 218.7	Water Drinking Water		Yes No Field						Buffer 10%H2SO4 10%NH4OH NH4OH(conc)	

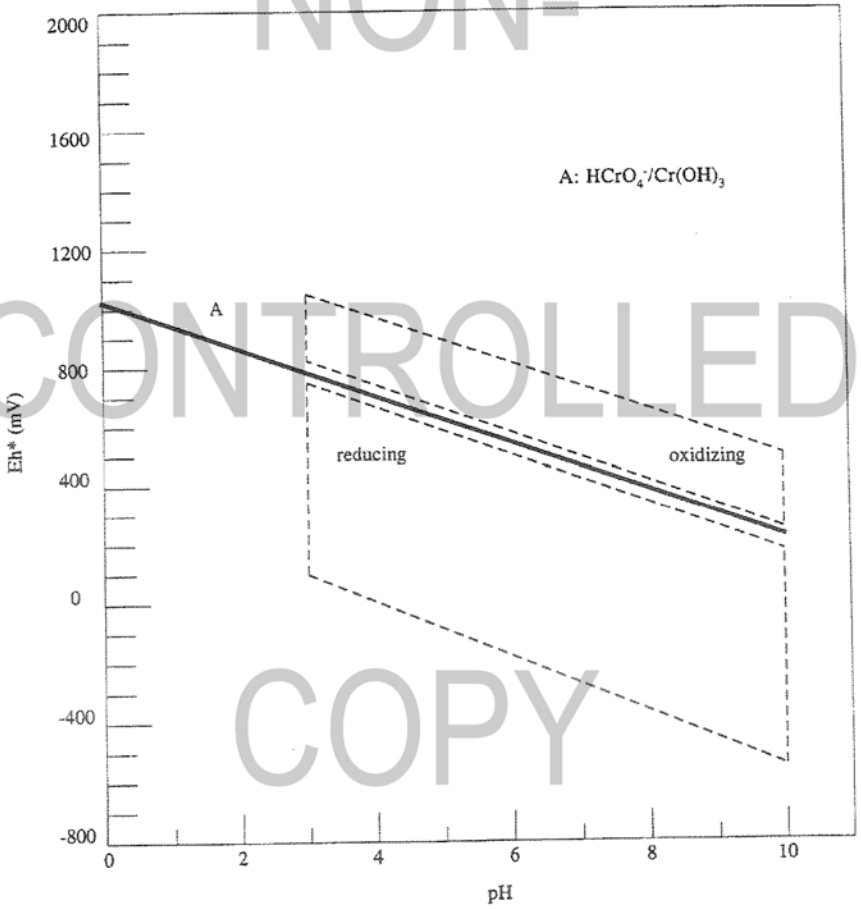
	Drinking water 218.7	Drinking water 218.6	Non-Pot Water 218.6	Water 7199
Filter	No	Required	Required	Optional
pH	>8.0	9.0-9.5	9.3-9.7	unpreserved - adjust to 9.0-9.5
Res Chlorine	<0.1 mg/L	NA	NA	NA
Holding Time	14 days	5 days	28 days	24 hours

P:\INTRANET\QAQC\Forms Controlled\SMO Cr6+ preservation log r1
SMO Cr6+ preservation log r1 1/10/13

Page 1

FIGURE 2
Eh/pH PHASE DIAGRAM

The dashed lines define Eh-pH boundaries commonly encountered in soils and sediments.



* Note the Eh values plotted on this diagram are corrected for the reference electrode voltage: 244 mV units must be added to the measured value when a separate calomel electrode is used, or 199 mV units must be added if a combination platinum electrode is used.

Analytical Sequence for Method 218.7

Injection Number	Sample ID
1	Calibration Standard 1
2	Calibration Standard 2
3	Calibration Standard 3
4	Calibration Standard 4
5	Calibration Standard 5
6	Calibration Standard 6
7	Calibration Standard 7
8	LL-CCV
9	CCB
10	Analytical Sample 1
11	Analytical Sample 2
12	Analytical Sample 3
13	Analytical Sample 4
14	Analytical Sample 5
15	Analytical Sample 6
16	Analytical Sample 7
17	Analytical Sample 8
18	Analytical Sample 8 Spike
19	Analytical Sample 8 Spike Dup
20	ML-CCV
21	CCB

Injection Number	Sample ID
22	Analytical Sample 9
23	Analytical Sample 10
24	Analytical Sample 11
25	Analytical Sample 12
26	Analytical Sample 13
27	Analytical Sample 14
28	Analytical Sample 15
29	Analytical Sample 16
30	Analytical Sample 16 Spike
31	Analytical Sample 16 Spike Dup
32	HL-CCV
33	CCB
34	Analytical Sample 17
35	Analytical Sample 18
36	Analytical Sample 19
37	Analytical Sample 20
38	Analytical Sample 20 Spike
39	Analytical Sample 20 Spike Dup
40	ML-CCV
41	CCB

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 w/ current 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 radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes. Added section concerning locking of coolers. Added more detail to 7.18 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 login 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 shorts 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 field 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

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
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 / 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,N+N	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 (AHS)	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 unuseuable, 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 #: FK 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		

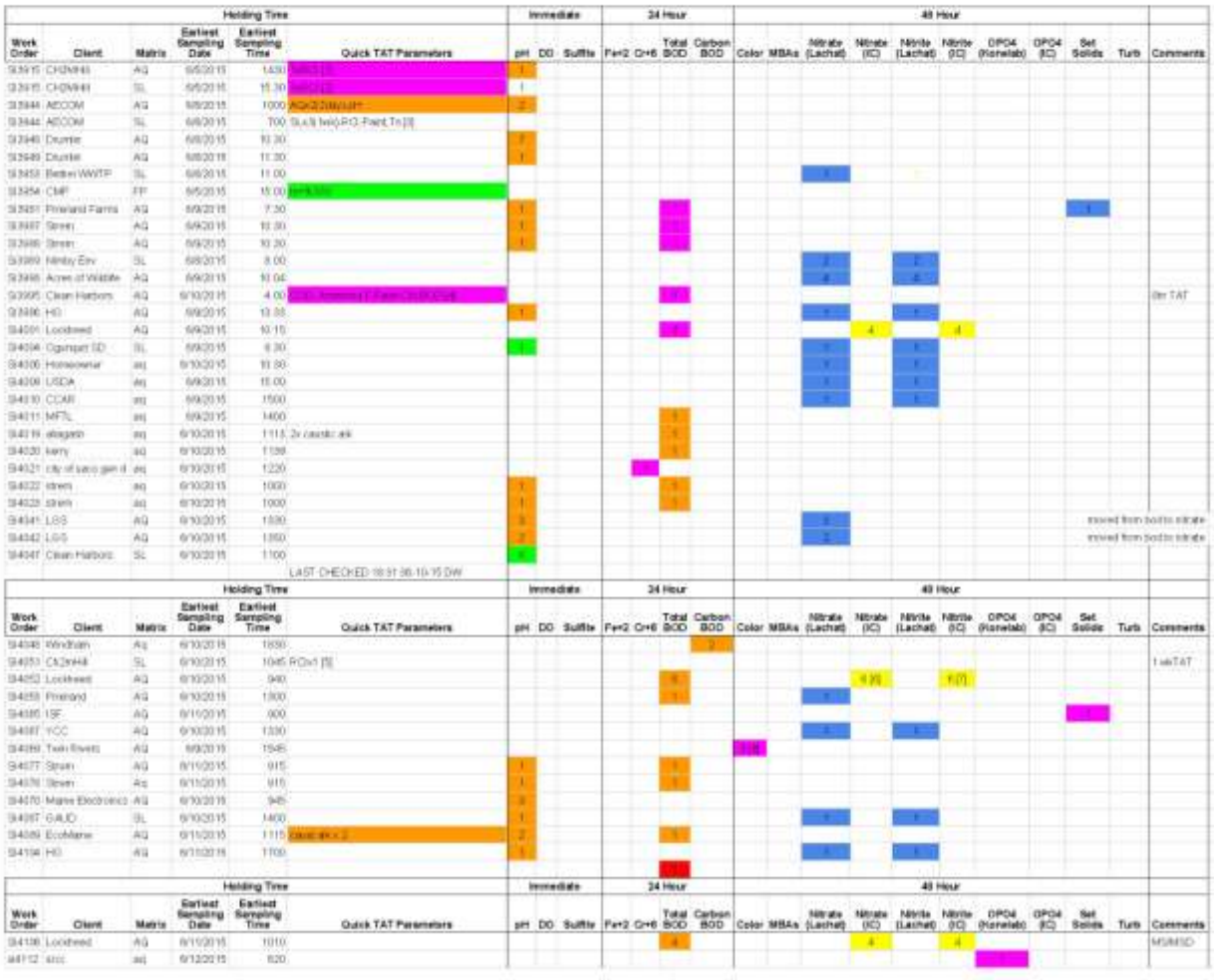
QA-032 - Revision 1 - 12/30/2009 Updated: 04/26/2016

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:					

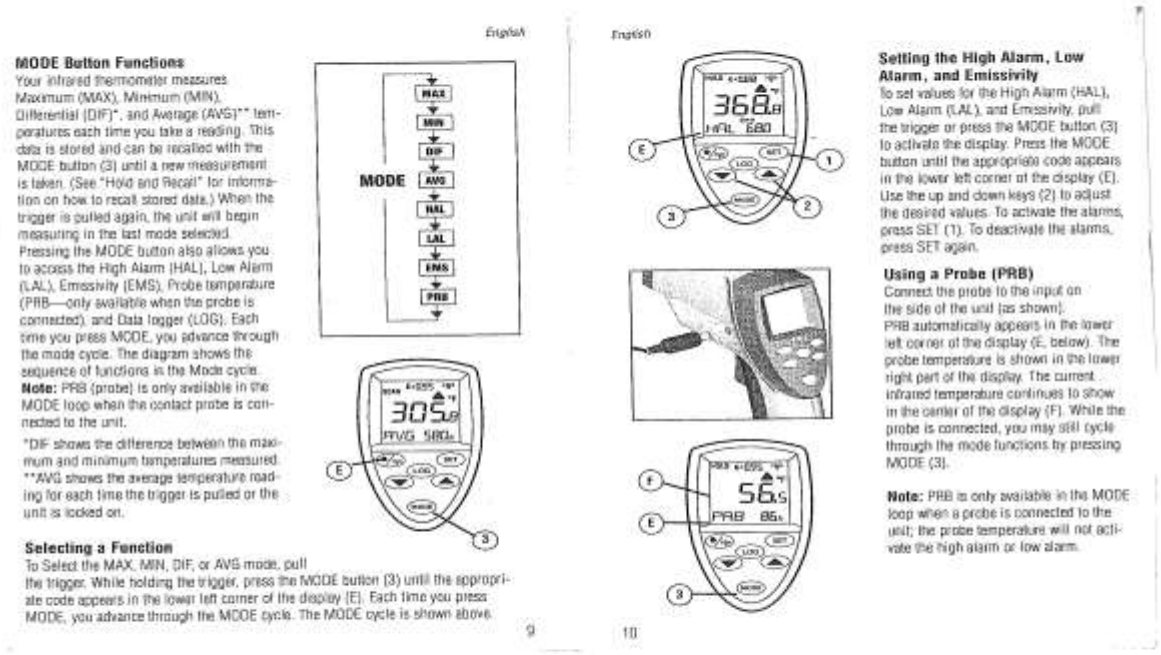
* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments.

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.

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.

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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

Reviewed and Approved by: _____ **Date:** _____

ME-005 - Revision 1 - 09/23/2010

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
REVIEWED BY:			DATE:			

QA-068 – Revision 1 – 09/23/2010

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] Quote Date: 29-MAR-10 Expires:
Name: [redacted] Date Expected: 28-SEP-40
Address: [redacted] Email: [redacted]

Notes: 8-aqueous VOA, 4-MEE,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 tsnarr@elmllc.com, Mail rpt and EDD on CD, no HC, metals need to be rpt mg/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: 13 Terms: Reg List: Edd: KAS064-XLS

Product	Matrix	Quant	STD or Special Lists	Short	Unit Price	Total Price
E325.2-CHLORIDE	AQ	1	STD		40	40
E353.2-NITRATE	AQ	1	STD	SHORT	0	0
E353.2-NITRITE	AQ	1	STD	SHORT	0	0
E375.4-SULFATE	AQ	1	STD		0	0
RSKSOP175-MEE	AQ	1	STD		85	85
SM5310B-TOC	AQ	1	STD		25	25
SW3010-PREP	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_LD	AQ	1	STD		115	115
					$	$85.00

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

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	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

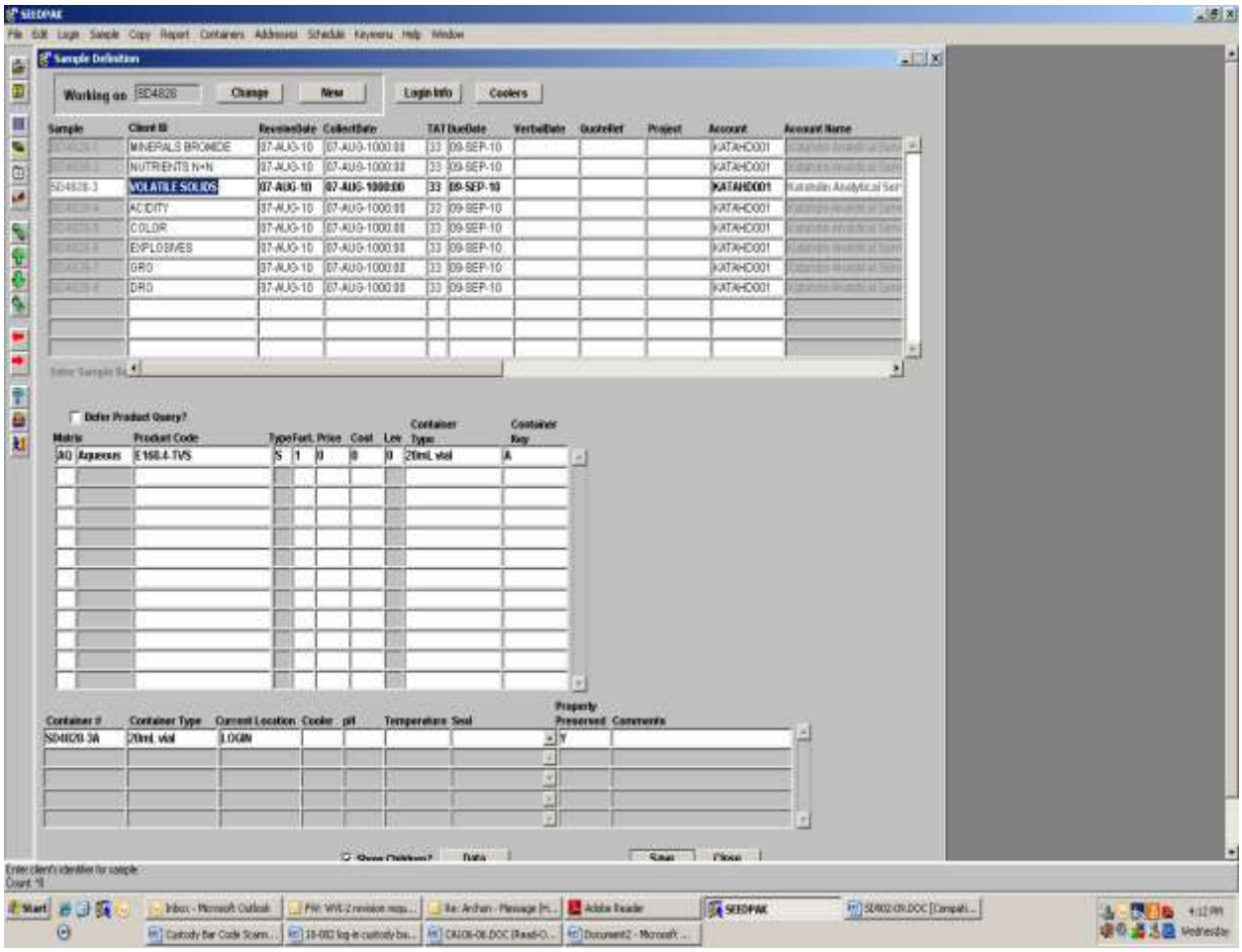
Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULLMS	09-FEB-07			3		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULLMS	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: _____ Date: 2/01

QA Officer: _____ 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 greater 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.	LMD	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

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

QA-022 - Revision 1 - 11/09/2009

QAQC645

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									↓	↓

0000012

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: GW
# 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: GW/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 G

Risk Assessment Work Plan

AECOM Imagine it. Delivered.

Human Health and Ecological Risk Assessment Work Plan

Former Nike PR-79 Control Area

U.S. Army Corp of Engineers

DERP FUDS Project and Property Number: D01RI0063/02

April 2020

Prepared for:

U.S. Army Corp of Engineers
New England District
696 Virginia Road
Concord, MA 01741-275

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Julie AF Kabel, MPH	Christine Archer	Robert Shoemaker	Gregory Hencir

Table of Contents

Figures

Tables

1. Introduction

This Risk Assessment Work Plan (RAWP) is provided as Appendix G of the Quality Assurance Project Plan (QAPP) for the Comprehensive Remedial Investigation (RI) of the Former Nike PR-79 Control Area (property) in Foster, Rhode Island (AECOM, 2019). The area where contamination associated with the property has come to be located is referred to, throughout the QAPP and this RAWP, as the "Site". A Site Plan is provided in **Figure 1**.

The objective of the RI sampling event is to amend the existing Site dataset and determine the presence and/or absence of constituents of potential concern (COPCs) associated with former Department of Defense (DoD) activities in Site media using a biased sampling approach to identify potential source areas. The biased sampling results will be evaluated in comparison to risk-based human health and ecological screening levels and Site-specific background levels to determine whether COPCs are present in Site media, for which exposure-based, unbiased sampling should be performed in a second RI phase for the purposes of evaluation in a human health and ecological risk assessment.

The objectives of this RAWP are the following:

- To provide the approach that will be followed to perform the evaluation of biased sampling results collected during the first RI phase in comparison to risk-based human health and ecological screening levels and Site-specific background levels (referred to in this RAWP as the "Phase I risk screening assessment"). The approach for evaluation of the first RI phase of data is provided in Section 2 of this RAWP; and
- To provide the approaches that will be followed in the human health risk assessment (HHRA) and screening-level ecological risk assessment (SLERA) performed as part of the second RI phase (if determined to be necessary following evaluation of biased sampling results collected during the first RI phase), including the human and ecological receptors and exposure pathways to be evaluated, and the associated exposure assumptions that will be used. The risk assessment approach, which is provided in Section 3 of this RAWP, was developed in accordance with the United States Environmental Protection Agency (USEPA) Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and United States (U.S.) Army Corp of Engineers (USACE) risk assessment guidance, as available/applicable.

A detailed description of the Site is provided in Worksheet #10 of the QAPP.

2. Phase I Risk Screening Assessment

The planned field investigation discussed in the QAPP is designed to amend the existing Site dataset and determine the presence and/or absence of COPCs associated with former DoD activities in Site media using a biased sampling approach to identify potential source areas. It is understood that this biased sampling design does not provide representative exposure-based samples for statistical analysis as necessary for a risk assessment. However, in understanding the limitations of that dataset, biased dataset will be evaluated in comparison to risk-based human health and ecological screening levels and Site-specific background levels to determine which constituents, if any, exceed the risk-based screening levels and background levels, and in which areas. If biased sample results exceed risk-based screening levels and background levels, then a second RI phase will be designed with an unbiased and exposure-based sampling approach and decision units and exposure areas to be further evaluated in a HHRA and SLERA, using the approach discussed in Section 3. If biased sample results do not exceed risk-based screening levels or Site-specific background levels, using the approach discussed in this section, then no further risk assessment will be performed.

This section provides further information on how the risk screening evaluation will be performed.

2.1 Data Selection

The Phase I risk screening assessment will include analytical data collected as part of the RI field program in addition to data collected from previous investigations, as appropriate. Environmental data that will be evaluated in the risk screening assessment will include Site soil, groundwater, porewater, sediment, and surface water samples. Data will be selected for use in the risk screening assessment based on consideration of the location of samples, potential exposure media, exposure points, exposure pathways, land uses, and receptors.

As discussed in the QAPP Worksheet #10, previous Site investigations have identified the following areas of concern (AOCs), from which existing soil analytical data are available:

- AOC-1 Radar Area
- AOC-2 Operations and Maintenance Area
- AOC-3 Southern Leach Field
- AOC-4 Western Sewage Disposal Area

The RI field investigation, discussed in the QAPP Worksheet #17, focuses soil and groundwater sampling locations in areas determined to contain spatial or analytical data gaps and in areas which have not previously been investigated. In addition, data will be collected from additional off-property locations to evaluate potential migration pathways to groundwater and potential discharge into Winsor Brook (located approximately 0.25 miles west of the property) and other downgradient wetlands and streams to the south and east.

The primary objective of the RI sampling is to resolve the remaining questions regarding the conceptual Site model (CSM) by selecting discrete sampling locations targeting potential release areas across the property (and in targeted off-property locations) rather than an unbiased sampling approach. Specific sampling locations and/or intervals have been incorporated into the QAPP in order to augment the existing dataset for evaluation in comparison to risk-based human health and ecological screening levels and Site-specific background levels. It is recognized that this sampling plan, which specifically targets areas of known former activities and potential release, instead of an unbiased sampling approach within an exposure area, may over-estimate exposure and the associated potential risk to human and ecological receptors. However, this design allows for the team to evaluate the magnitude and extent of potential impacts and determine whether further evaluation of potential risk using unbiased data collected using an exposure-based sampling approach is recommended in an additional phase of investigation.

Table 1 summarizes the media and areas for which data have been and/or will be collected, which will be evaluated in the risk screening assessment.

Table 1. Media and Areas Included in the Risk Screening Assessment Dataset

Media	On-Property (Associated with AOCs-1, -2, -3, and -4)	Off-Property
Soil	X	
Groundwater (a)	X	X
Sediment		X
Surface Water		X
Porewater		X

X – Indicates analytical data for the associated media/area will be evaluated in the risk screening assessment.

(a) Includes monitoring wells, piezometers, and water supply wells.

In general, data collected from the more recent sampling event(s) are preferred for use in the risk screening assessment over historical data. However, where the use of recent data alone is not considered sufficient to provide a measure of concentrations within environmental media that are appropriate to evaluate potentially complete exposure scenarios, historical data will be used. If data collected during previous investigations are determined not to be representative of current conditions (i.e., results are from excavated or inaccessible soils), these data will be excluded from evaluation, as applicable.

Soil

Soil data will be divided into two different depth intervals for evaluation in the risk screening assessment, as applicable. Surface soil will be defined as soil collected at a depth within 0 to 2 feet (ft) below ground surface (bgs), consistent with the surface soil interval defined in the previously collected dataset. Subsurface soil will be defined as soil collected at a depth greater than (>) 2 to 10 ft bgs. Consistent with standard CERCLA risk assessment practice (USEPA *Supplemental Risk Assessment Guidance for the Superfund Program* [1989]), soils deeper than 10 ft bgs will not be included in the risk assessment because receptors are not likely to contact these deeper soils within the predicted future uses of this Site.

Groundwater

Groundwater samples will be collected from on-property monitoring wells, piezometers, and bedrock wells and one off-property bedrock well installed during the RI sampling event (discussed in QAPP Worksheet #17, Section 17.2). The potentially complete exposure scenarios for these groundwater data are discussed in Section 4.2.1.

Existing analytical groundwater data from previous Site investigations are available from on-property temporary overburden piezometers, on-property bedrock water supply wells, and off-property bedrock residential water supply wells. Groundwater data were collected from temporary piezometers during a previous Site investigation conducted in 2016. Potential issues related to the data collection methods used during the 2016 investigation that have the potential to have affected the analytical groundwater results were identified, including but not limited to insufficient groundwater purge volumes for water supply well sampling (The Johnson Company, 2018). In addition, high turbidity was noted at multiple piezometer locations that was attributed to possible silting within the wells (USAPHC, 2016). Therefore, groundwater results from the 2016 sampling event will be reviewed (for data quality [whether they are biased by turbidity, etc.], location within the plume [if present], representative groundwater depths, and whether they are representative of most current conditions) to determine whether these data will be included in the risk screening assessment.

The existing water supply wells on and in the vicinity of the property include two on-property bedrock water supply wells (NIKE-1 and NIKE-2), four off-property residential water supply wells (ROU-1, ROU-2, ROU-3, and ROU-4) within 200 ft of the property, and approximately 30 residential water supply wells located within a one-mile radius of the property. The RI sampling event does not include sampling of water supply wells due to the presence of an adequate existing dataset from previous sampling events; however, additional groundwater wells will be established as part of the RI sampling event to provide representative groundwater monitoring across the Site and to assess

potential continuing sources. Results for the on-property bedrock water supply wells (NIKE-1 and NIKE-2) and the four off-property residential water supply wells located within 200 ft of the property (ROU-1, ROU-2, ROU-3, and ROU-4) from previous investigations will be reviewed to select the appropriate dataset for evaluation in the risk screening assessment. The groundwater dataset will be selected to represent current Site conditions, while accounting for potential variations in concentration over time. Groundwater results from samples collected prior to the carbon filtration will be used for evaluation in the risk screening assessment.

Existing groundwater data from off-property residential water supply wells located further away from the property ("DW-" wells) will not be evaluated within the scope of the risk screening assessment. However, recommendations for further assessment of these wells during a later RI phase may be made following assessment of groundwater results from the on-property area and adjacent off-property area.

Groundwater data will be summarized separately, as follows, for evaluation in the risk screening assessment, so that COPCs can be identified separately, since these represent different groundwater exposure points, as further discussed in Section 4.2:

- shallow groundwater (defined as groundwater samples collected less than or equal to 15 ft bgs);
- groundwater from monitoring wells (overburden and bedrock) and piezometers;
- groundwater from water supply wells will be evaluated on a well-by-well basis using existing groundwater data collected during previous investigations.

The risk screening assessment will utilize only the total inorganic groundwater data, consistent with HHRA guidance. Groundwater data will not be evaluated in comparison to ecological screening levels given there are no complete exposure pathways to ecological receptors.

Sediment, Surface Water, and Porewater

Analytical sediment, surface water, and porewater data from previous Site investigations in 2013 are available from off-property locations. Sediment, surface water, and porewater samples will be collected from streams, seeps, and delineated wetlands in the off-property area during the RI sampling event. The risk screening assessment will utilize data from both the previous Site investigations and the RI sampling event. Porewater data will not be evaluated in comparison to human health screening levels given there are no complete exposure pathways to human receptors.

2.2 Risk Screening Assessment

The risk screening assessment may consider low frequency of detection (FOD), low toxicity and low concentration, consistency with Site-specific background, essential nutrient status, and whether the constituent is likely to be present due to laboratory contamination. COPCs will be selected based on a comparison of the maximum detected concentration within each media and depth interval (as appropriate) to risk-based human health and ecological screening levels selected as described in Sections 4.1.1.2 and 5.2.2, respectively. Essential nutrients, including magnesium, calcium, potassium, and sodium will not be selected as COPCs, consistent with USEPA guidance (USEPA, 1989). COPCs detected at concentrations above the risk-based screening levels will be identified as preliminary COPCs for further evaluation in comparison with Site-specific background levels (background threshold values [BTVs]).

Constituents within a medium/depth interval that are not detected at a concentration above the associated BTV will not be identified as Site-related COPCs and will be excluded from further quantitative risk assessment (e.g., in later RI phases). Background-related COPCs may be qualitatively discussed in the RI or risk characterization section of a risk assessment performed in a later phase of the RI (if applicable). Preliminary COPCs that are not identified as being consistent with background will be identified as Site-related COPCs and will be further quantitatively evaluated in a risk assessment performed based on unbiased and exposure-based sampling results in a second phase of the RI.

2.2.1 Background Threshold Values

The background dataset for each medium/analyte will be evaluated for the presence of outliers using the default outlier test in USEPA's ProUCL software (USEPA, 2016) prior to calculation of BTVs. Outliers identified will be eliminated from the background dataset, as appropriate. Following the elimination of outliers, medium-specific BTVs will be derived for constituents identified as preliminary COPCs. BTVs will be calculated in USEPA's ProUCL software (USEPA, 2016) as the 95 percent (%) upper tolerance limit (95UTL). The 95UTL statistic will be selected based on the distribution of the raw dataset (e.g., if the detected concentrations follow a normal, lognormal, or gamma distribution, then the normal, lognormal, or gamma 95UTL will be selected, respectively). In cases of no discernible distribution, the nonparametric 95UTL statistic will be selected. If the dataset includes non-detects, the Kaplan-Meier BTV statistics will be selected on the basis of the distribution of the detected concentrations.

2.2.2 Screening-Level Vapor Intrusion Evaluation

A screening-level vapor intrusion evaluation will be completed in which shallow groundwater results will be compared to screening levels protective of the vapor intrusion pathway. The following groundwater screening levels will be used to select preliminary groundwater COPCs associated with the vapor intrusion pathway:

- USEPA Vapor Intrusion Screening Levels (VISLs) for groundwater based on a target excess lifetime cancer risk (ELCR) of $1x10^{-6}$ and a target HQ of 0.1 (to account for potential cumulative effects of multiple chemicals on the same target organ) (USEPA, 2019b). VISLs for a residential and industrial exposure scenario will be used to select groundwater COPCs for the associated exposure scenario.

If groundwater COPCs are identified in the well adjacent to the current on-property building (the former Mess Hall; PR79-MW-006S), recommendations for further investigation and assessment of the potential vapor intrusion pathway, such as the collection and evaluation of soil vapor and/or indoor air samples in a second RI phase, will be made. If groundwater COPCs are identified in wells elsewhere on-Site, recommendations may be made to further evaluate the potential vapor intrusion pathway in the event that, in the future, additional on-Site buildings are planned.

3. Phase II Human Health Risk Assessment and SLERA Approach

This section presents the approach to be used for performing an HHRA and SLERA in a later phase of the RI if deemed necessary based on the risk screening assessment discussed in Section 2.

3.1 Data Evaluation

The HHRA and SLERA will evaluate Site soil, groundwater, porewater, sediment and/or surface water samples collected during a second RI phase (if applicable) using an unbiased and exposure-based sampling approach. The specific media, decision units, and exposure areas that will be evaluated in the HHRA and SLERA will be determined following the risk screening evaluation, as appropriate. Biased sampling results collected during the first RI Phase or other historical sampling events will not be included in the HHRA or SLERA.

3.2 Summary Statistics

For each dataset, the data will be compiled into summary statistics as discussed below for evaluation in the HHRA and SLERA. For each constituent detected at least once within an exposure area/media/depth interval (i.e., surface soil, subsurface soil, etc.), the summary statistics will include the minimum and maximum detected concentrations, location of maximum detected concentration, FOD, and the range of detection limits calculated in accordance with USEPA *Risk Assessment Guidance for Superfund* (RAGS; USEPA, 1989). All summary statistics will be calculated following data treatment, as described below.

- Treatment of Duplicates: For sample locations in which a field duplicate sample is also collected (e.g. parent sample and corresponding field duplicate), the combined sample results for each constituent/medium/area/ depth interval combination will be processed for use in the calculation of summary statistics. Parent and field duplicates will be resolved as follows: 1) where both the parent sample and the duplicate are not detected, the lower of the limits of detection (LODs) will be used, 2) where both the parent sample and the duplicate are detected, the average of the detected results will be used, and 3) where one of the pair is reported as not detected and the other is detected, the detected concentration will be used.
- Calculation of Totals: Groups of constituents including polycyclic aromatic hydrocarbons (PAHs), low molecular weight PAHs, high molecular weight PAHs, and xylenes (i.e., o-, m-, and p-xylenes) will be totaled per sample prior to calculating summary statistics by summing only the detected individual concentrations within each group. For samples without any detection of individual constituents, the maximum LOD within the sample will be used as a non-detect value.

3.3 Background Evaluation

Representative soil, sediment, surface water, and porewater background chemistry data will be collected for comparison to Site-specific data within the risk assessment. As part of the risk assessment and data evaluation process, relevant available background data will be compiled and compared with Site-specific data to distinguish Site-related impacts from non-Site-related background conditions (naturally occurring or anthropogenic). The background evaluation will consist of a tiered process and will be conducted in accordance with the *Tri-Service Position Paper on Background Levels in Risk Assessment* (USACE, 2011), as follows:

- The background dataset will be evaluated for the presence of outliers using the default outlier test in USEPA's ProUCL software (USEPA, 2016) prior to conducting the background evaluation. Outliers identified will be eliminated from the background dataset, as appropriate, prior to conducting the following tiers of evaluation.
- Derivation of BTVs, as described in Section 2.2.1.
- Hypothesis testing - For each medium and constituent for which preliminary COPCs are detected at concentrations above BTVs, hypothesis testing for centrality will be conducted to compare the mean background concentration with Site-related concentrations. This analysis will be conducted prior to the selection

of final COPCs to focus the HHRA and SLERA on Site-related COPCs. The null hypothesis (Ho) and alternative hypothesis (Ha) are defined as:

– Ho = Mean/Median of the Site data >= Background data + S

– Ha = Mean/Median of the Site data < Background data + S

*Whereas the substantial difference (S) will be the standard deviation of the background data set.

For media/constituents for which a sufficient background dataset is not available or where requirements for conducting a statistical evaluation are not met, a qualitative/graphical comparison (using histograms, quantile plots, and/or cumulative frequency plots, as appropriate) will be performed.

The results of the background evaluation will be used in the HHRA and SLERA as discussed in Sections 4 and 5, respectively. Constituents identified as being detected at concentrations consistent with background (i.e., non-Site related COPCs) will not be further quantitatively evaluated in the HHRA and SLERA.

4. Human Health Risk Assessment

An HHRA will be conducted to evaluate whether current or future potential exposures to COPCs attributable to past operations at the former Nike PR-79 Control Area may pose a risk and/ hazard to human health above USEPA target levels. The evaluation will perform quantitative estimation of potential cancer risk and noncancer hazard to current and potential future human receptors that may come into contact with Site-related constituents in soil, groundwater, sediment, and surface water. The HHRA will be conducted in accordance with USEPA CERCLA risk assessment guidance (USEPA, 1989, 2004, 2005a, and 2009a), with consideration of the Rhode Island Department of Environmental Management (RIDEM) risk assessment guidance (RIDEM, 2019). Where differences between USEPA and RIDEM guidance occur, USEPA methods will be followed. References cited herein are based on the most current versions of sources available as of the date of this RAWP. In the case that updates to references become available, the most current version of the references available at the time the HHRA is initiated will be used and appropriately referenced in the HHRA.

USEPA's four step HHRA paradigm (USEPA, 1989) will be followed to conduct the HHRA. Each of the tiers of evaluation is discussed in further detail in the following subsections.

- Data Evaluation
- Exposure Assessment
- Toxicity Assessment
- Risk Characterization
- Uncertainty Analysis

4.1 Data Evaluation

The data evaluation step involves identification and summarization of analytical data appropriate for use in the HHRA, and the selection of COPCs for quantitative evaluation in the HHRA.

4.1.1 Selection of Constituents of Potential Concern

COPCs are a subset of the complete list of constituents detected in Site media that are carried through the quantitative risk assessment process. The HHRA COPC selection process may consider low FOD, low toxicity and low concentration, consistency with Site-specific background, essential nutrient status, and whether the constituent is likely to be present due to laboratory contamination. For this HHRA, Site-related COPCs will be established per exposure area, medium, and depth interval (as appropriate) as described in the following sub-sections.

4.1.1.1 Frequency of Detection and Essential Nutrients

Essential nutrients, including magnesium, calcium, potassium, and sodium will not be selected as COPCs for further evaluation in the HHRA, consistent with USEPA guidance (USEPA, 1989). Constituents will not be eliminated as COPCs on the basis of low FOD alone.

4.1.1.2 Comparison to Human-Health Screening Levels

Preliminary COPCs will be identified based on a comparison of the maximum detected concentrations of each detected constituent per media/exposure area/depth interval (as applicable) to human health screening levels (which may include regulatory values, if applicable). Constituents detected at concentrations above the USEPA screening levels will be identified as preliminary COPCs for further evaluation in comparison with background, as discussed in the following section. A comparison to RIDEM screening levels will also be performed for informational purposes, but RIDEM screening levels will not be used to select COPCs. Constituents either not detected in a particular medium/exposure area or detected at concentrations below the USEPA screening levels will not be identified as COPCs for the associated exposure scenario and will not be evaluated further in the HHRA. For constituents lacking a screening level, a conservative surrogate constituent may be identified, and its screening level selected for use in the COPC selection. A constituent will be eliminated as a COPC if there are no associated toxicity values available with which quantitative evaluation in the HHRA may be performed, an appropriate surrogate cannot be identified, and the investigation provides no other evidence of an apparent threat.

Screening levels that will be used in the HHRA are discussed by media below.

Soil

The following soil screening levels will be used in the HHRA:

- USEPA Regional Screening Levels (RSLs) (USEPA, 2019a) based on a target ELCR of $1x10^{-6}$ and a target hazard quotient (HQ) of 0.1. The RSLs based on a target HQ of 0.1 will be used to account for potential cumulative effects of multiple constituents on the same target organ. The residential soil RSLs will be used in the selection of preliminary COPCs for evaluation of all receptors. The residential soil RSLs are based on USEPA's default residential exposure frequency of 350 days/year and exposure duration of 26 years. Use of the residential soil RSLs is conservative for non-residential scenarios because the exposure frequency and duration associated with non-residential exposure scenarios are less than those for a residential scenario.
- RIDEM Method 1 Direct Contact Exposure Criteria for soil (RIDEM, 2019). The residential criteria will be used for comparison purposes only, but will not be used to select COPCs for further evaluation in the HHRA.

A comparison to screening levels protective of the soil to groundwater pathway will not be included in this HHRA. Analytical groundwater data will be available and evaluated directly.

Groundwater

Groundwater data will be summarized as follows for evaluation in the HHRA, (1) shallow groundwater (defined as groundwater samples collected less than or equal to 15 ft bgs), (2) groundwater from monitoring wells and piezometers, and (3) groundwater from water supply wells. The following screening levels will be used for all groundwater datasets:

- USEPA tap water RSLs (USEPA, 2019a) based on a target ELCR of $1x10^{-6}$ and a target HQ of 0.1 (to account for potential cumulative effects of multiple chemicals on the same target organ).
- USEPA Maximum Contaminant Levels (MCLs) (USEPA, 2018a).
- RIDEM Method 1 GA Groundwater Objectives (RIDEM, 2019).

Constituents detected at a concentration greater than the RSL will be selected as preliminary groundwater COPCs. These levels are protective of a residential drinking water exposure scenario. Therefore, use of these screening levels is conservative for selecting COPCs for non-drinking water exposure scenarios (such as incidental ingestion by a construction worker). A comparison to the MCLs and RIDEM criteria will be used for comparison purposes only, but will not be used to select COPCs for further evaluation in the HHRA.

Sediment

Published human health screening levels are not available for sediment. Therefore, soil screening levels will be used for selecting preliminary COPCs in sediment. The use of soil screening levels for evaluation of sediment is very conservative due to the lower exposure frequency for sediment as compared to soil.

Surface Water

The following surface water screening levels will be used in the HHRA:

- USEPA National Recommended Ambient Water Quality Criteria (AWQC) for human health consumption of water and organisms (USEPA, 2019c).
- RIDEM AWQCs for human health consumption of water and organisms (RIDEM, 2018).

If screening levels are not available for a constituent from the above sources, the screening level will be equal to the following:

- USEPA tap water RSLs based on a target ELCR of $1x10^{-6}$ and a target HQ of 0.1 (USEPA, 2019a). The tap water RSLs are protective of a residential drinking water exposure scenario. Therefore, use of these screening levels is overly conservative for selecting surface water COPCs.

Constituents detected at a concentration greater than the USEPA AWQC (or RSL if AWQCs are not available) will be selected as preliminary surface water COPCs. A comparison to the RIDEM AWQCs will be used for comparison purposes only, but will not be used to select COPCs for further evaluation in the HHRA.

4.1.1.3 Comparison to Background Levels

Preliminary COPCs identified based on the comparison to human health screening levels will be further evaluated in conjunction with background data, as available. The background evaluation will be conducted as described in Section 3.3. Constituents within a medium/exposure area/depth interval that are either (1) not detected at a concentration above the associated BTV, or (2) for which the hypothesis testing for centrality concludes that Site-specific concentrations are consistent with background, will not be identified as Site-related COPCs and will be excluded from further quantitative evaluation in the HHRA. Background-related COPCs may be qualitatively discussed in the risk characterization section of the HHRA. Preliminary COPCs that are not identified as being consistent with background will be identified as Site-related COPCs and will be further quantitatively evaluated in the HHRA.

4.2 Exposure Assessment

The purpose of the exposure assessment is to provide a quantitative estimate of the magnitude and frequency of potential exposure to COPCs by current and future receptors. Potentially exposed individuals, and the pathways through which those individuals may be exposed to COPCs are identified based on the physical characteristics as well as the current and reasonably foreseeable future uses of the area. As discussed further in the following section, the property is currently zoned as “municipal” and the current land use is non-residential (i.e., used for municipal administration). The area to the south of the property is currently residential use. Future use of the property and the surrounding area is anticipated to remain consistent with current use. However, due to the residential areas located in proximity to the property, residential and recreational use of the Property are considered reasonable future use scenarios. Therefore, the HHRA will evaluate an unlimited use and unrestricted exposure (UU/UE) scenario to provide information for making risk-management decisions. The extent of a receptor's exposure is estimated by constructing exposure scenarios that describe the potential pathways of exposure to COPCs and the activities and behaviors of individuals that might lead to contact with COPCs in the environment. This information is identified based on the Site-specific CSM, which is presented in the QAPP Worksheet #10. A summary of the information related to human health exposure is provided in this section.

4.2.1 Potential Human Exposure Scenarios

The property, which is located on top of Oak Hill, was originally developed for agricultural use, namely as an apple orchard. The US Government acquired the subject property between 1955 and 1957 and developed the Site for radar missile tracking as part of the NIKE Missile Defense System. The property is currently zoned as “municipal” and the current land use is non-residential (i.e., used for municipal administration). There is only one building currently present on the property, which is the former Mess Hall building that is used by the Foster-Glocester Regional School District, Northwest Special Education Region for administrative purposes.

The area surrounding the property is comprised of northern hardwood forest and rural development. The area located at the base of Oak Hill to the south of the property is currently occupied by residences and farms, and to the southeast and northeast of the property are solar panel arrays. Three residences are located within 300 to 400 ft of the property with approximately 68 residences located within a one-mile radius in Foster and North Scituate, Rhode Island (USAPHC, 2010).

Local potable water is supplied with private bedrock drinking water supply wells, not municipal water. Groundwater in the area of the Site is classified by RIDEM as ‘GA’, known or presumed to be suitable for drinking water use without treatment. There are two on-property bedrock water supply wells (NIKE-1 and NIKE-2). There are four off-property residential water supply wells (ROU-1, ROU-2, ROU-3, and ROU-4) within 200 ft of the property, and approximately 30 residential water supply wells located within a one-mile radius. Point of use duel carbon filtration systems have been installed at one on-property water supply well (NIKE-1) and three off-property residential water supply wells (ROU-1, ROU-2, and ROU-3) due to trichloroethene (TCE) reported above the associated USEPA MCL during previous sampling events (discussed in detail in QAPP Worksheet #10 Section 10.6). USACE New England District (CENAE) continues to monitor these four carbon filtration systems.

The nearest surface water bodies include three streams and a 16-acre wetland complex located approximately 0.25 mile to the south; a 0.15-acre wetland followed by Winsor Brook located approximately 0.25 miles to the west; and a 0.07 acre open water body to the north of the property.

Future use of the property is assumed to remain consistent with current use for the foreseeable future. However, due to the residential areas located in proximity to the property, residential or recreational use of the property are considered reasonable future use scenarios.

The HHRA will evaluate potentially complete exposure pathways for the following receptors under a current and/or reasonably foreseeable future land-use scenario:

- Current/Future On-Property Trespasser (Adolescent) – Exposure to surface soil (or combined surface and subsurface soil assuming soils become mixed during potential future redevelopment activities) via incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles.
- Current On-Property (Commercial/Industrial) Worker - Exposure to surface soil via incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles; and exposure to shallow groundwater via inhalation of indoor air within the current on-property building (Former Mess Hall Building) (screening-level evaluation). (Exposure to groundwater via ingestion of drinking water is not complete under a current scenario due to the presence of a carbon filtration system on one existing on-property water supply well (NIKE-1) and NIKE-2 has been inactive since 2003. The evaluation of the future on-property (commercial/industrial) worker scenario below will provide information on a hypothetical scenario in which the carbon filtration system on NIKE-1 fails).
- Future On-Property (Commercial/Industrial) Worker - Exposure to surface soil (or combined surface and subsurface soil assuming soils become mixed during potential future redevelopment activities) via incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles; exposure to shallow groundwater via inhalation of indoor air within hypothetical on-property buildings that may be constructed in the future (i.e., potential vapor intrusion pathway) (screening-level evaluation); and exposure to groundwater (from on-property monitoring wells and piezometers and on-property water supply wells [NIKE-1 and NIKE-2]) via ingestion of drinking water.
- Current/Future On-Property Construction/Utility Worker - Exposure to combined surface and subsurface soil (to a maximum depth of 10 ft) via incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles; and exposure to shallow groundwater via incidental ingestion, dermal contact, and inhalation of volatiles in an excavation trench.
- Current/Future Off-Property Resident (Adult/Child) - Exposure to shallow groundwater via inhalation of volatiles in indoor air (i.e., potential vapor intrusion pathway) (screening-level evaluation); and exposure to groundwater (from off-property water supply wells [ROU-1, ROU-2, and ROU-3]) via ingestion of drinking water, and dermal contact and inhalation while bathing/showering. (The drinking water pathway is currently incomplete in residences with carbon filtration systems but may be complete under a future scenario in which carbon filtration systems are removed or become non-operational).
- Future Recreational User (Adult/Child) – Exposure to surface soil (or combined surface and subsurface soil assuming soils become mixed during potential future redevelopment activities) via incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles; and exposure to sediment and surface water via dermal contact while wading (incidental ingestion while wading is considered negligible). (Scenario conservatively assumes recreational users may walk or hike through the area and access on-property soils in addition to off-property sediment and surface water).
- Hypothetical Future On-Property Resident (Adult/Child) - Exposure to surface soil (or combined surface and subsurface soil assuming soils become mixed during potential future redevelopment activities) via incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles; exposure to shallow groundwater via inhalation of volatiles in indoor air (i.e., potential vapor intrusion pathway) (screening-level evaluation); and exposure to groundwater (from on-property monitoring wells and piezometers and on-property water supply wells [NIKE-1 and NIKE-2]) via ingestion of drinking water, and dermal contact and inhalation while bathing/showering. There are currently no residential receptors on the property. However, due to the residential areas located in proximity to the property, the HHRA will evaluate an UU/UE scenario to provide information for making risk-management decisions.

The quantitative exposure assumptions that will be used in the HHRA were selected in accordance with USEPA guidance. Exposure assumptions may differ from default values to factor in Site-specific considerations. **Table 2** summarizes the receptors, exposure media, and exposure pathways to be evaluated in the HHRA. **Tables 3 through 7** present the exposure assumptions that will be used to evaluate each human receptor under a reasonable maximum exposure (RME) scenario. A central tendency exposure (CTE) scenario will only be evaluated for receptors for which the RME scenario yields a potential risk/hazard above USEPA target levels, as defined in Section 4.4.

For each receptor and exposure area, the exposure dose will be estimated for each COPC via each exposure pathway by which the receptor is assumed to be exposed. Exposure doses for oral and dermal exposure will be calculated following USEPA guidance (1989, 2004). Exposure doses for inhalation exposures are calculated following USEPA guidance (USEPA, 2009a).

4.2.2 Exposure Point Concentrations

Exposure point concentrations (EPCs) for evaluation of soil, groundwater, surface water, and sediment COPCs will be calculated per exposure medium and exposure area, as discussed in this section. EPCs will generally be equal to the 95% upper confidence limit (UCL) on the arithmetic mean concentration (USEPA, 2002a). When a sufficient sample size or minimum frequency of detection is not available, or if the UCL is greater than the maximum detected concentration, the maximum detected concentration will be used as the EPC for evaluation of the RME scenario, and the arithmetic mean concentration will be used as the EPC for evaluation of the CTE scenario. The most current version of USEPA's ProUCL software available at the time the HHRA is started will be used to calculate the UCL (USEPA, 2016).

Groundwater EPCs will be derived in accordance with USEPA guidance (2014), as follows. Groundwater results from multiple sampling events will be reviewed to select the dataset to most appropriately represent current conditions, while accounting for potential variations in concentration over time. Groundwater EPCs will be calculated using data from monitoring wells (and piezometers) within the core of the plume (or area of higher groundwater concentrations). On-property and off-property water supply wells will be evaluated on a well-by-well basis using existing groundwater data collected during previous investigations.

If lead is identified as a COPC, EPCs for evaluation of lead will be equal to the arithmetic mean concentration (i.e., rather than the UCL on the mean) in accordance with USEPA guidance (USEPA, 2003a and 2007).

EPCs for COPCs in fugitive dust (outdoor air), excavation trench air, and shower air will be modeled based on soil or groundwater EPCs (as appropriate), calculated (as described above) based on measured concentrations. These EPCs will be derived as follows:

- EPCs for COPCs in fugitive dust (outdoor air) will be estimated by combining soil EPCs with a particulate emission factor (PEF) calculated in accordance with USEPA guidance (2002b).
- Excavation trench air concentrations of volatile COPCs in groundwater infiltrating an excavation trench will be modeled by the use of the method recommended by USEPA (1994a) for estimating volatilization from standing water.
- For evaluation of the bathing/showering pathway, concentrations of volatiles in shower air will be estimated based on groundwater EPCs using the Foster and Chrostowski (2003) shower model.

4.3 Toxicity Assessment

The purpose of the toxicity (or dose-response assessment) is to identify the types of adverse health effects a constituent may potentially cause, and to define the relationship between the dose of a constituent and the likelihood or magnitude of an adverse effect (response) (USEPA, 1989). The USEPA's guidance regarding the hierarchy of sources of human health dose-response values in risk assessment will be followed (USEPA, 2003b; USEPA, 2019a), as follows:

- Tier 1: USEPA's Integrated Risk Information System (IRIS) (USEPA, 2019d).
- Tier 2: Provisional Peer-Reviewed Toxicity Values (PPRTVs) obtained from USEPA via the USEPA National Center for Environmental Assessment (NCEA) in Cincinnati, Ohio (USEPA, 2019e).

- Tier 3: Other sources of dose-response values include, but are not limited to, California EPA's Office of Environmental Health and Hazard Assessment (OEHHA) Toxicity Criteria Database (CalEPA, 2018), Minimal Risk Levels (MRLs) published by the Agency for Toxic Substances and Disease Registry (ATSDR, 2019), and the Health Effects Assessment Summary Tables (HEAST) (USEPA, 1997a).

Where published dose-response values are not available for a constituent, dose-response values will be selected based on surrogate constituents (i.e., constituents with structural similarities), if available.

4.3.1 Evaluation of Mutagenic Mode of Action

USEPA guidance for early life exposure to carcinogens (USEPA, 2005a) requires that potential risks from constituents that act by a mutagenic mode of action be calculated differently than constituents that do not act via a mutagenic mode of action. Therefore, the age-dependent adjustment factors (ADAFs) will be applied to all COPCs that have been identified by USEPA to act via a mutagenic mode of action unless otherwise specified in accordance with the guidance, as follows:

- Age 0 to 2 years (2-year interval from birth until 2nd birthday) – ADAF = 10.
- Ages 2 to 16 years (14-year interval from 2nd birthday to 16th birthday) – ADAF = 3.
- Ages 16 and up (after 16th birthday) – no adjustment - ADAF = 1.

ADAFs will be used to quantify potential cancer risks for the trespasser (adolescent), resident, and recreational user exposure scenarios. The worker receptors are assumed to be adults (i.e., over 16 years of age); therefore, adjustment factors are not applicable, and will not be used for the worker exposure scenarios.

4.3.2 Constituent-Specific Information

The following constituent-specific approaches will be followed in the HHRA:

- Chromium – Toxicity values for total chromium are not available from USEPA sources of toxicity values. There is no historical evidence that chromium was specifically related to a particular process or material in past DoD operations at this Site. Therefore, total chromium results will be evaluated using toxicity values for trivalent chromium.
- Lead - If lead is identified as a COPC, the following models will be used to further evaluate lead in the HHRA:
  - USEPA's Integrated Exposure Uptake Biokinetic (IEUBK) Model (USEPA, 1994b, 2007, 2010, 2017a) will be used to assess exposure to lead for the residential child exposure scenario. As children are more sensitive to the effects of lead than adults, this evaluation will also be protective of the residential adult exposure scenario.
  - USEPA's Adult Lead Model (ALM) (USEPA, 2003a, 2017b, 2017c) will be used to assess exposure to lead in soils/sediment for non-resident adult exposures.
  - The Bowers Model will be used to assess exposure to lead in groundwater for non-residential adult exposures. The Bowers Model, which is available from peer reviewed literature (Bowers et al., 1994), is based upon a biokinetic slope factor approach conceptually similar to the ALM.
  - USEPA's model for evaluating Intermittent or Variable Exposures at Lead Sites (USEPA, 2003c) Equation 8 will be used to derive a screening level for lead protective of a recreational child's exposure. As children are more sensitive to the effects of lead than adults, the screening level will also be protective of recreational adult's exposure. The use of this equation is appropriate for situations in which children are exposed to a background level of lead at home and a different concentration of lead at a Site. The equation allows for the modification of the default residential lead screening level of 400 milligrams per kilogram (mg/kg) by using a weighted exposure time and frequency to account for the varying exposures.

  The determination of whether there is unacceptable risk associated with lead will be based on the results of the modeling. USEPA's default residential lead soil screening level of 400 mg/kg derived based on a target blood lead level (PbB) of 10 microgram per deciliter (µg/dL) will be used to evaluate lead as described above.

  Since the toxicokinetics of lead are well understood, lead is regulated based on a PbB concentration. USEPA has determined that childhood PbB concentrations at or above 10 µg/dL present risks to children's health. The

modeled PbB concentration is compared to the PbB level of concern of 10 µg/d (USEPA, 2007). USEPA's risk reduction goal for contaminated sites is to limit the probability of a child's PbB concentration exceeding 10 µg/dL (the P10) to 5% or less after cleanup. Therefore, the USEPA Office of Solid Waste and Emergency Response (OSWER) has established a health protection goal that young children exposed to lead at their residences should not encounter a risk of more than 5% of exceeding a PbB level of 10 µg/dL (USEPA, 1994b, 1998a). Based on USEPA's current approach, PbB concentrations less than 10 µg/dL do not require further management of the risk associated with exposure to lead.

Up until May 2012, the Centers for Disease Control and Prevention (CDC) had defined an elevated PbB level as equal to or greater than 10 µg/dL for children under 6 years of age. However, more recent scientific evidence on adverse effects of PbB levels below 10 µg/dL in children have been noted and CDC has adopted a "reference value" for lead based on the 97.5th percentile of the PbB level distribution in U.S. children aged 1-5 years, which currently is 5 µg/dL. If the CDC reference value were adopted by the USEPA in the future, the potential health effects associated with exposure to lead may be underestimated should the calculated maximum estimated PbB levels exceed 5 µg/dL.

- PAHs - PAHs (acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, indeno(1,2,3-cd)pyrene, fluoranthene, fluorene, naphthalene, phenanthrene, and pyrene) identified as COPCs will be evaluated individually in the HHRA and in accordance with USEPA guidance (USEPA, 1993a).
- Xylenes - Xylenes (i.e., o-, m-, and p-xylenes) will be evaluated as total xylenes in the HHRA.

4.4 Risk Characterization

Risk characterization combines estimates of exposure with toxicity data to develop estimates of the probability that an adverse effect will occur under the specified conditions of exposure. Estimates of carcinogenic risks are expressed as probabilities of developing cancer reported as ELCR. Current HHRA practice considers carcinogenic risks to be additive when assessing exposure to a mixture of hazardous substances. Non-carcinogenic hazards are reported as pathway-specific hazard indices (HIs), which are the sum of individual COPC HQs for that pathway. A total HI is calculated for each receptor by summing the pathway-specific HIs within each media (e.g., summing dermal and ingestion soil HI estimates). As a first approximation, all COPCs will be conservatively assumed to have additive effects. If the total HI assuming additive effects is greater than USEPA and RIDEM's target levels, HIs will be calculated separately for COPCs that have similar systemic effects (i.e., per target organ).

USEPA (1991) states that where the cumulative incremental current or future potential ELCR to an individual is less than 10^{-4}, action generally is not warranted unless there are adverse environmental impacts. USEPA also considers noncancer hazards by using a target HI per target organ of 1 (USEPA, 1991). For each associated exposure scenario (i.e., receptor/medium) with a potential ELCR or HI above USEPA risk targets, constituents of concern (COC) will be selected from those Site-related COPCs significantly contributing to an individual ELCR > 10^{-4} or target organ HI > 1 (at one significant figure). Constituents significantly contributing to an ELCR > a target level of 10^{-5}, which is the midpoint of the USEPA acceptable risk range, will also be identified in the HHRA to facilitate risk management decisions based on the Site-specific CSM, if warranted.

4.5 Uncertainty Analysis

Within any of the steps of the HHRA process, assumptions must be made due to a lack of absolute scientific knowledge. Some of the assumptions are supported by considerable scientific evidence, while others have less support. Every assumption introduces some degree of uncertainty into the HHRA process. Regulatory guidance on HHRA methodology requires that conservative assumptions be made throughout the HHRA to ensure that public health is protected. Therefore, when all of the assumptions are combined, it is much more likely that risks are overestimated rather than underestimated.

The assumptions that introduce the greatest amount of uncertainty in the HHRA, both Site-specific and those inherent to the HHRA process, will be discussed qualitatively in the Uncertainty Analysis section of the HHRA. Examples of Site-specific uncertainties are those associated with sampling/analysis methods, the COPC selection process, estimation of EPCs, representativeness of the exposure scenarios and input parameters, the availability of toxicity values, etc. Examples of uncertainties inherent to the HHRA process are the extrapolation of toxicity from animal

studies to humans, from high to low doses, and the specific models used to develop dose-response values; the combination of upper-bound exposure estimates with upper-bound toxicity estimates, etc. The uncertainties associated with the HHRA will be discussed in qualitative terms, because for most of the assumptions there is not enough information to assign a numerical value to the uncertainty that can be factored into the calculation of risk.

5. Ecological Risk Assessment

A SLERA will be conducted to evaluate potential ecological effects of chronic exposures to constituents detected in soil, porewater, sediment, and surface water collected within the property and/or adjacent off-property areas, and to determine whether Site-related constituents detected in these areas pose a potential hazard above target levels to the environment. The SLERA will be conducted in accordance with USEPA CERCLA and RIDEM risk assessment guidance. However, where differences between USEPA and RIDEM guidance occur, USEPA guidance will be followed.

The SLERA is considered the first tier of the ecological risk assessment (ERA) process (i.e., Steps 1 and 2 of the USEPA eight-step process for ERA [USEPA, 1997b]). The purpose of the SLERA will be to evaluate the potential adverse environmental effects of Site-related constituents on ecological receptors and resources at or near the Site, including off-property wetland and aquatic habitat associated with Winsor Brook and other downgradient streams. If the results of the SLERA indicate sufficient potential ecological risk, further ecological risk assessment will be conducted in a sub-tier of Step 3 (referred to as Step 3a or a refined SLERA) of the USEPA eight-step process.

The framework for the SLERA will be consistent with USEPA methodology based on the following key guidance documents:

- Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessment, Interim Final (USEPA, 1997b);
- Guidelines for Ecological Risk Assessment (USEPA, 1998b);
- The Role of Screening-Level Risk Assessments and Refining Contaminants of Concern in Baseline Ecological Risk Assessments (USEPA, 2001); and
- A Guide to Screening Level Ecological Risk Assessment (Tri-Services Environmental Risk Assessment Work Group [TSERAWG], 2008).

The SLERA will include the following components:

- Problem Formulation
- Risk Analysis including:
  - Exposure Assessment
  - Effect Assessment
- Risk Characterization

These components are described in the following subsections.

5.1 Problem Formulation

The Problem Formulation provides the framework for the SLERA and serves to define the risk assessment objectives and identify the ecological receptors, exposure pathways and endpoints to be evaluated. These components are presented in the following sections and are compiled into an ecological CSM. It is anticipated that the SLERA will evaluate an upland exposure area (surface soil associated with the AOCs) and a wetland/stream exposure area (sediment, surface water, and porewater collected from downgradient aquatic habitats). Exposure areas may be further divided following the SLERA, if warranted based on the Phase I RI sampling results or based on identification of additional ecological exposure areas.

5.1.1 Selection of Specific Receptors and Exposure Pathways

Potential ecological receptors occurring within the property and/or adjacent off-property areas and potentially complete ecological exposure pathways will be evaluated. Each exposure pathway includes a potential source of COPC, an environmental medium, and a potential exposure route. Incomplete routes of exposure will not be evaluated in the SLERA. This approach is used to focus the risk evaluation on exposure pathways that are

considered to be potentially complete and for which there are adequate data pertaining to the receptors, exposure, and toxicity.

Exposure pathways for several groups of ecological receptors were identified as potentially relevant. The available data suggest that the primary receptors at the Site are terrestrial plants, invertebrates, small mammals, and birds exposed to surface soils. In addition, the SLERA will also evaluate:

- Benthic/wetland invertebrates exposed to surface sediment and/or porewater associated with downgradient wetlands and streams;
- Fish, aquatic invertebrates, and amphibians exposed to surface water associated with downgradient wetlands and streams;
- Birds and mammals exposed through incidental ingestion of sediment, ingestion of surface water, and/or by ingestion of contaminated prey items impacted by sediment or surface water.

The presence/absence of standing water in the streams and wetlands will be documented during the field sampling. Fish are not expected to be present in intermittent streams or wetlands. Amphibians are likely to be exposed to surface water and sediment within streams and wetlands; however, due to a lack of amphibian-specific screening values, amphibians will be considered qualitatively in the SLERA.

5.1.2 Selection of Biological Endpoints

The following assessment and measurement endpoints will be included in the SLERA:

- For terrestrial plants and soil invertebrates exposed to surface soils:
  - Assessment endpoint – Sustainability (survival, growth, reproduction) of local populations of terrestrial plants and invertebrates exposed to surface soils.
  - Measurement endpoint – Comparison of surface soil concentrations against soil screening benchmarks that are protective of soil invertebrates and terrestrial plants.
- For small mammals and birds exposed to surface soils:
  - Assessment endpoint – Sustainability (survival, growth, reproduction) of local populations of small mammals and birds exposed to surface soils.
  - Measurement endpoint – Comparison of calculated total daily dose (TDD) for avian and mammalian receptors from exposure to chemicals in soil and/or ingestion of contaminated prey items to constituent-specific toxicity reference values (TRVs) (compiled from the literature or derived as described below). Doses above the TRVs are considered indicative of a potential for ecological risks.
- For benthic/wetland invertebrates exposed to surface sediment and/or porewater:
  - Assessment endpoint – Sustainability (survival, growth, reproduction) of local populations of benthic/wetland invertebrates exposed to sediment associated with downgradient wetlands and streams.
  - Measurement endpoint – Comparison of surface sediment concentrations against benthic invertebrate-based sediment screening benchmarks.
  - Measurement endpoint – Comparison of porewater concentrations against aquatic life-based surface water screening benchmarks.
- For fish, aquatic invertebrates, and amphibians exposed to surface water:
  - Assessment endpoint – Sustainability (survival, growth, reproduction) of local populations of fish, aquatic invertebrates, and amphibians exposed to surface water associated with downgradient wetlands and streams.
  - Measurement endpoint – Comparison of surface water concentrations against aquatic life-based surface water screening benchmarks.

- For birds and mammals exposed to sediment associated with downgradient wetlands and streams:
  - Assessment endpoint – Sustainability (survival, growth, reproduction) of local populations of birds and mammals exposed to sediment and/or associated with downgradient wetlands and streams.
  - Measurement endpoint – Comparison of calculated TDD for avian and mammalian receptors from exposure to chemicals in sediment, surface water and/or ingestion of contaminated prey items to constituent-specific TRVs (compiled from the literature or derived as described below). Doses above the TRVs are considered indicative of a potential for ecological risks.

The end product of the problem formulation step is the development of an ecological CSM, which describes the COPC origin, fate, transport, exposure pathways, and receptors of concern. The preliminary Site-specific ecological CSM is presented in the QAPP (Worksheet #10).

5.2 Risk Analysis

The risk analysis phase of the SLERA is based on the CSM developed in problem formulation and addresses the characterization of potential ecological exposure and corresponding effects. The ecological exposure assessment involves the identification of potential exposure pathways and an evaluation of the magnitude of exposure of identified ecological receptors. The ecological effects assessment describes the potential adverse effects to ecological receptors from exposure to COPCs in environmental media. The data and methods that will be used to identify and characterize ecological exposure and effects are described in the following subsections.

5.2.1 Data Evaluation and Identification of COPCs

Analytical data will be grouped for evaluation by media in the SLERA. The SLERA will evaluate surface soil associated with the AOCs and sediment, surface water, and porewater collected from potentially impacted downgradient aquatic habitats (i.e., wetlands and streams).

COPCs will be identified based on the complete list of constituents detected in on-property and off-property media that are carried through the quantitative risk assessment process and will be established per exposure area, medium, and depth interval (as appropriate). Consistent with the HHRA COPC selection process, COPCs will be selected for the SLERA based on a comparison of the maximum detected concentrations of each detected constituent per media/area/depth interval (as applicable) to ecological screening values (ESVs). The ESVs identified are based on conservative endpoints and sensitive ecological effects data and they represent tools useful for a preliminary ecological screening of surface soil, porewater, surface water and sediment concentrations.

Constituents detected at concentrations above the ESVs will be identified as preliminary COPCs for further evaluation in comparison with background, as discussed in Section 1.3. The COPC selection process may consider low FOD, low toxicity and low concentration, consistency with Site-specific background, essential nutrient status, and whether the constituent is likely to be present due to laboratory contamination. Essential nutrients, including magnesium, calcium, potassium, and sodium will not be selected as COPCs for further evaluation in the SLERA, consistent with USEPA guidance (USEPA, 1989). Constituents will not be eliminated as COPCs on the basis of low FOD alone. In addition, a constituent may be retained as a COPC if there is no ESV available. A constituent will not be retained as a COPC if it is consistent with background based on the BTV comparison, hypothesis testing, or qualitative/graphical background comparisons.

5.2.2 Ecological Effects Assessment

Receptor- and media-specific ESVs will be selected in order to identify COPCs and evaluate the potential for ecological risks in the vicinity of the Site. These ESVs will be based on conservative endpoints and sensitive ecological effects data and will be used for a preliminary screening of Site constituent levels to determine if there is a need to conduct further analyses or investigations at the Site.

Literature-derived soil ESVs will be selected to evaluate potential impacts to terrestrial invertebrates and plants using the following hierarchy:

- USEPA Ecological Soil Screening Levels (Eco-SSLs) for plants and invertebrates developed according to USEPA guidance (USEPA, 2005b); and

- USEPA Region 4 soil screening levels (USEPA, 2018b).

Surface water and porewater analytical chemistry analysis results will be compared to risk-based surface water screening values. Sources for surface water screening values will be considered in this order:

- Chronic federal Ambient Water Quality Criteria (AWQC) for aquatic life (USEPA, 2018c);
- RIDEM WQS (RIDEM, 2018); and
- USEPA Region 4 surface water screening levels (USEPA, 2018b).

As appropriate, hardness-dependent screening values will be adjusted based on the average Site-specific hardness.

Sediment analytical results will be compared to low effect risk-based sediment screening values derived by USEPA Region 4 (USEPA, 2018b).

If screening values are not available from the above sources, then other sources will be considered. These may include benchmarks presented in the Los Alamos National Laboratory (LANL) EcoRisk Database (LANL, 2017) or other literature sources.

Risks to mammals and birds from exposure to constituents in surface soil and sediment will be evaluated using food chain models to estimate the TDD which will be compared to TRVs representing acceptable daily doses in mg/kg-day. Detected constituents will be evaluated using a food chain model if the chemical is identified as an 'important bio-accumulative compound' by USEPA (USEPA, 2000).

TRVs incorporated into the quantitative evaluation of potential ecological risks to wildlife will be obtained from the following sources: TRVs derived according to USEPA guidance (USEPA, 2005b) during the development of Eco-SSLs, ORNL publication Toxicological Benchmarks for Wildlife: 1996 Revision (Sample et al,. 1996), and the LANL EcoRisk Database (LANL, 2017). When TRVs are not derived in these documents, the literature will be reviewed for relevant data and TRVs will be derived using the methodology of ORNL (Sample et al., 1996). TRVs used in the SLERA will be based on No Observed Adverse Effect Levels (NOAELs).

Constituent concentrations in food items will be calculated using bioaccumulation factors (BAFs) and other uptake factors from published sources or regression equations from the USEPA Eco-SSL Guidance (USEPA, 2007b) and other literature sources (e.g., Bechtel Jacobs, 1998a,b). The USEPA's Biota-Sediment Accumulation Factor (BSAF) database will be used as a primary source to identify sediment-to-invertebrate uptake factors for organic constituents. Soil-based uptake factors may be used when sediment-specific uptake factors are not identified. Given the lack of uptake factors for amphibians, surface water-to-fish uptake factors will be used to estimate amphibian tissue concentrations.

5.2.3 Invertebrate and Plant Ecological Exposure Assessment

Step 2 of the of the eight-step process is the screening level exposure estimate and risk calculation in which risk is estimated by comparing maximum detected concentrations in the relevant media against associated ESVs. Data treatment and calculation of summary statistics for the SLERA will be consistent with the rules described in Section 1.2. Maximum detected concentrations will be identified by per exposure area and medium

5.2.4 Wildlife Ecological Exposure Assessment

Exposure assumptions (e.g., body weights, relative consumption of food items, foraging range, exposure duration, etc.) for wildlife species will generally be obtained from the USEPA's Wildlife Exposure Factors Handbook (USEPA, 1993b). **Table 8** provides the exposure parameters for birds and mammals to be evaluated in the food web models. Allometric equations (Nagy, 2001) were used to estimate food ingestion rates in **Table 8**. It is anticipated that separate food web models will be conducted for the stream/wetland and upland areas.

Wildlife receptors to be modeled will include:

- American robin (*Turdus migratorius*) – insectivore foraging in upland areas
- Bobwhite quail (*Colinus virginianus*) – herbivore foraging in upland areas
- Marsh wren (*Cistothorus palustris*) – insectivore foraging in stream/wetland areas

- Raccoon (*Procyon lotor*) – omnivore foraging in stream/wetland and upland areas
- Meadow vole (*Microtus pennsylvanicus*) – herbivore foraging in stream/wetland and upland areas
- Short-tailed shrew (*Blarina brevicauda*) –insectivore foraging in stream/wetland and upland areas

5.2.5 SLERA Risk Calculations

To estimate risks to plants, soil invertebrates, aquatic invertebrates, and benthic invertebrates, HQs will be calculated for each analyte in each medium (i.e., surface soil, sediment, surface water, porewater) by dividing the maximum detected concentration by the relevant ESV using the following formula:

HQ = Maximum detected concentration/ESV

For higher trophic level wildlife receptors, the risk estimate is also based on the HQ, defined as the ingested dose divided by the species-specific TRV:

HQ = TDD/TRV

Analytes which exceed their respective screening benchmarks (i.e., HQs > 1) and analytes without screening benchmarks will be retained as preliminary COPCs. Essential nutrients (calcium, magnesium, potassium, and sodium) will not be retained as COPCs. Preliminary COPCs will be evaluated as described in Section 1.3 to determine whether concentrations in exposure areas are consistent with background. Preliminary COPCs will be retained as final COPCs if they are not identified as being consistent with background.

5.3 Risk Characterization

The results of the risk analysis step of the SLERA will be reviewed to determine the likelihood of adverse environmental effects, and to determine whether a conclusion of no significant risk can be reached for each assessment endpoint evaluated. The ecological risk characterization will summarize the results of the risk analysis step and will provide interpretation of the ecologically significant findings. Aspects of ecological significance that will be considered to help place the Site into a broader ecological context include: the nature and magnitude of effects, the spatial and temporal patterns of effects, results of the background analyses, and the potential for recovery once a stressor has been removed.

The documentation of the risk characterization will include a summary of assumptions, uncertainties (both generic and Site-specific), strengths and weaknesses of the analysis phase of work, and justification of conclusions regarding the ecological significance of the estimated (i.e., risk of harm) or actual (i.e., evidence of harm) risks. Some uncertainties bias the results of the risk assessment towards overestimating risk, while others underestimate the potential risks. Discussions of uncertainty will include several aspects of the SLERA including, but not limited to, sampling, data quality, study design, selection of indicator species, estimates of exposure, and selection of ESVs. The uncertainty section will identify limitations and assumptions of the SLERA and will relate them to the overall conclusions of the SLERA.

5.4 SLERA Scientific/Management Decision Point

A scientific/management decision point (SMDP) will be determined based on the outcome of the SLERA to conclude that (1) the available data indicate the potential for ecological risk and further ERA evaluation is warranted, (2) the available data indicate either no or low potential for ecological risk and no further work is warranted, or (3) there are data gaps that must be addressed before the presence or absence of risk can be concluded (e.g., additional sampling or analysis).

If the decision is made that further investigation is warranted for any specific receptors/pathways, a sub-tier of Step 3 (Step 3a) of the USEPA's eight-step ERA process may be conducted. USEPA (2001) and Department of Defense (DoD) guidance (TSERAWG, 2008) provide the basis to introduce sub-tiers into the SLERA process and the approach is described in the following sub-section.

5.5 Step 3a COPC Refinement

Step 3a, a sub-tier of Step 3 (referred to as a refinement of the SLERA by TSERAWG [2008]), serves to refine the list of COPCs identified in the conservative evaluation conducted in Steps 1 and 2 by considering additional Site-specific factors. The refinement step may include further evaluation against background concentrations or re-evaluation of parameters considered in the SLERA (e.g., assumption of 100% bioavailability). Only COPCs, pathways, and receptors retained in Step 3a would be considered for additional risk assessment. In many cases, the Step 3a refined risk estimate provides the basis for defining potential risk drivers which may be further evaluated for remedial decisions, or alternatively a complete baseline ERA (BERA) may be initiated, which applies USEPA Step 3b through Step 8 of the ERA process. It is currently unknown if Step 3a will be warranted for any ecological receptors, but the following discussion provides the proposed approach.

The purpose of Step 3a is to reevaluate COPCs that were retained in the SLERA for further evaluation, and to identify and eliminate from further consideration those COPCs that were retained because of the use of conservative exposure scenarios (e.g., maximum concentrations). Using more realistic Step 3a assumptions, the SLERA risk estimates described above will be recalculated for the pathways and COPCs retained at the end of the SLERA.

It is anticipated that the Step 3a re-evaluation/refinement process will follow these steps:

- Calculate alternative EPCs based on UCLs (rather than the maximum detected concentrations).
- Consider whether upland and stream/wetland areas can be divided into smaller exposure areas.
- Revise food web exposure assumptions and calculate Step 3a doses and HQ risk estimates. This includes considering alternative EPCs, uptake factors, exposure durations, area use factors, and both NOAEL- and lowest observed adverse effect level (LOAEL)-based TRVs.
- Revise benchmarks screens using alternative EPCs and alternative screening levels (e.g., probable effect concentrations).
- Identify COPCs with HQ less than 1 in the Step 3a food web model and benchmark screens and eliminate from further evaluation.
- For COPCs with an HQ greater than 1, compare media concentrations to background levels, if not already conducted. Identify COPCs present at concentrations below or consistent with background levels and propose these for elimination from further evaluation.
- For COPCs with an HQ greater than 1, consider bioavailability, identify COPCs likely to be biologically unavailable, and propose these for elimination from further evaluation.
- Review FOD to identify COPCs with low detection frequencies (and sufficient data for acceptable Site characterization). If a COPC was detected in only a very small percentage of the samples collected (5 percent or less), the risk identified in the SLERA may be overestimated and further evaluation of the COPC is not warranted.

After the re-evaluation/refinement, the decision criteria for Step 3a include:

- If the re-evaluation of the conservative exposure assumptions used in the SLERA supports an acceptable risk determination for all COPCs, then a no further action designation for ecological risk is warranted, and the Site exits the ERA process.
- If the re-evaluation of the conservative exposure assumptions does not support an acceptable risk for all constituents, the BERA process continues to Step 3b and subsequent steps, or to remedial decisions.

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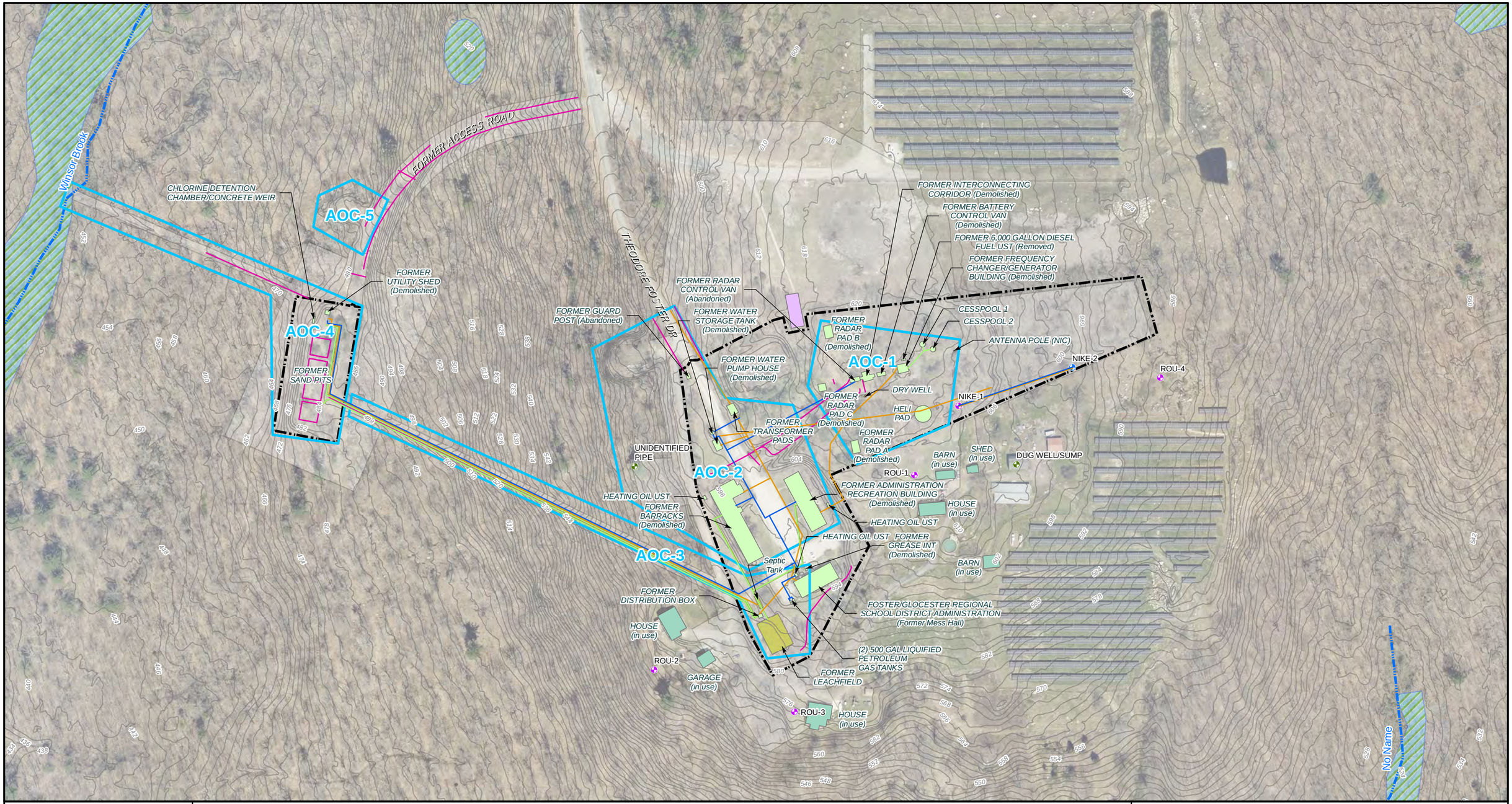

AECOM

Drawn:	JB	09/24/2019
Approved:	GH	09/24/2019
Project #:	60608703	

Legend

- Drinking Water - Individual
- Drinking Water - Not in Use
- Monitoring
- Stream
- Perimeter Fence
- Former U.S. Army Property
- Non-Government Property
- RI/FS Area of Concern (AOC)
- Wetlands
- Extent of Former NIKE PR-79 Control Area (HPA/USAGC, 2015)
- Cemetery
- Contour (2 ft)
- Electrical Line
- Sewer Line
- Water Line
- Drainage

Sources
Orthoimagery from RIGIS, 2019.
Buildings, Utilities and Contours from The Johnson Company (RI/FS) Work Plan, February 2018.
HPA/USAGC, 2015.

N

0 100 200

Scale in Feet

FIGURE 1
SITE PLAN

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: \\uschl1fp001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO 0001 NIKE PR79 RIFS\900_GIS\Projects\QAPP\MXD\Fig_10_1_Site_Plan.mxd

TABLE 2

SELECTION OF EXPOSURE PATHWAYS

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe	Medium	Exposure Medium	Exposure Point	Receptor Population	Receptor Age	Exposure Route	Type of Analysis	Rationale for Selection or Exclusion of Exposure Pathway
Current/ Future	Soil	Surface Soil (0 - 2 feet)	On-Site	Trespasser	Adolescent (ages 7-<16)	Ingestion	Quant	Incidental ingestion of surface soil impacted by Site-related releases may occur.
						Inhalation	Quant	Exposure to fugitive dust and volatiles originating from surface soil may occur.
						Dermal	Quant	Dermal contact with surface soil impacted by Site-related releases may occur.
				Commercial/ Industrial Worker	Adult	Ingestion	Quant	Assumes full time workers, including maintenance workers or landscapers, etc. may become exposed to soil impacted by Site-related releases. Future development of Site could include commercial/industrial development, leading to potential exposure to soil.
						Inhalation	Quant	Exposure to fugitive dust and volatiles originating from surface soil may occur.
						Dermal	Quant	Assumes full time workers, including maintenance workers or landscapers, etc. may become exposed to soil impacted by Site-related releases. Future development of Site could include commercial/industrial development, leading to potential exposure to soil.
		Surface and Subsurface Soil (0 - 10 feet)	On-Site	Construction Worker	Adult	Ingestion	Quant	Construction workers may incidentally ingest soils impacted by Site-related releases.
						Inhalation	Quant	Construction workers may inhale fugitive dusts and volatiles originating from soils impacted by Site-related releases.
						Dermal	Quant	Construction workers may come into contact with soils impacted by Site-related releases.
	Groundwater	Groundwater	Off-Site Water Supply Wells (ROU-1, ROU-2, ROU-3, and ROU-4)	Resident	Adult	Ingestion	Quant	Exposure to groundwater as drinking water from off-Site supply wells will be evaluated to assess potential risk under a scenario in which existing carbon filtration systems are turned off.
						Inhalation	Quant	Exposure to groundwater from off-Site supply wells via inhalation of volatiles while showering/bathing will be evaluated to assess potential risk under a scenario in which existing carbon filtration systems are turned off.
						Dermal	Quant	Exposure to groundwater from off-Site supply wells via contact while showering/bathing will be evaluated to assess potential risk under a scenario in which existing carbon filtration systems are turned off.
					Child	Ingestion	Quant	Exposure to groundwater as drinking water from off-Site supply wells will be evaluated to assess potential risk under a scenario in which existing carbon filtration systems are turned off.
						Inhalation	Quant	Exposure to groundwater from off-Site supply wells via inhalation of volatiles while showering/bathing will be evaluated to assess potential risk under a scenario in which existing carbon filtration systems are turned off.
						Dermal	Quant	Exposure to groundwater from off-Site supply wells via contact while showering/bathing will be evaluated to assess potential risk under a scenario in which existing carbon filtration systems are turned off.
	Shallow Groundwater	Indoor Air	On-Site	Commercial/ Industrial Worker	Adult	Inhalation	Semi-Quant	Workers in the current on-Site building (Former Mess Hall) or future buildings constructed on-Site may inhale volatiles originating from shallow groundwater via the vapor intrusion pathway. A screening-level evaluation and qualitative discussion of this pathway is performed. Recommendations will be made for further quantitative evaluation if deemed appropriate.
			Off-Site	Resident	Adult	Inhalation	Qual	Residents may inhale volatiles originating from Site groundwater via the vapor intrusion pathway. A screening-level evaluation and qualitative discussion of this pathway will be performed for the on-Site area. Recommendations for further evaluation of the off-Site area will be made if deemed appropriate.
					Child	Inhalation	Qual	
		Trench Air	On-Site	Construction Worker	Adult	Inhalation	Quant	Construction workers may inhale volatiles originating from shallow groundwater that accumulates in an excavation trench.
		Shallow Groundwater	On-Site	Construction Worker	Adult	Ingestion	Quant	Construction workers may incidentally ingest shallow groundwater while working in an excavation trench during construction related activities.
						Dermal	Quant	Construction workers may contact shallow groundwater while working in an excavation trench during construction related activities.

TABLE 2

SELECTION OF EXPOSURE PATHWAYS

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe	Medium	Exposure Medium	Exposure Point	Receptor Population	Receptor Age	Exposure Route	Type of Analysis	Rationale for Selection or Exclusion of Exposure Pathway
Future	Soil	Surface Soil (0 - 2 feet)	On-Site	Resident	Adult	Ingestion	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, incidental ingestion of surface soil may occur.
						Inhalation	Quant	Exposure to fugitive dust and volatiles originating from surface soil may occur.
						Dermal	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, dermal contact with surface soil may occur.
					Child	Ingestion	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, incidental ingestion of surface soil may occur.
						Inhalation	Quant	Exposure to fugitive dust and volatiles originating from surface soil may occur.
						Dermal	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, dermal contact with surface soil may occur.
				Recreational User	Adult	Ingestion	Quant	Incidental ingestion of surface soil impacted by Site-related releases may occur.
						Inhalation	Quant	Exposure to fugitive dust and volatiles originating from surface soil may occur.
						Dermal	Quant	Dermal contact with surface soil impacted by Site-related releases may occur.
					Child	Ingestion	Quant	Incidental ingestion of surface soil may occur in the future.
						Inhalation	Quant	Exposure to fugitive dust and volatiles originating from surface soil may occur.
						Dermal	Quant	Dermal contact with surface soil may occur in the future.
		Surface and Subsurface Soil (0 - 10 feet)	On-Site	Trespasser	Adolescent (ages 7-<16)	Ingestion	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
						Inhalation	Quant	Exposure to fugitive dust and volatiles may occur.
						Dermal	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
				Commercial/ Industrial Worker	Adult	Ingestion	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
						Inhalation	Quant	Exposure to fugitive dust and volatiles may occur.
						Dermal	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
				Resident	Adult	Ingestion	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property.
						Inhalation	Quant	Exposure to fugitive dust and volatiles may occur.
						Dermal	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property.
					Child	Ingestion	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property.
						Inhalation	Quant	Exposure to fugitive dust and volatiles may occur.
						Dermal	Quant	Under a hypothetical scenario in which the Site is redeveloped for future residential use, exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property.
				Recreational User	Adult	Ingestion	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
						Inhalation	Quant	Exposure to fugitive dust and volatiles may occur.
						Dermal	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
					Child	Ingestion	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.
						Inhalation	Quant	Exposure to fugitive dust and volatiles may occur.
						Dermal	Quant	Exposure to surface and subsurface soil may occur assuming soil is mixed during re-development of the property for future use.

TABLE 2

SELECTION OF EXPOSURE PATHWAYS

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe	Medium	Exposure Medium	Exposure Point	Receptor Population	Receptor Age	Exposure Route	Type of Analysis	Rationale for Selection or Exclusion of Exposure Pathway
Future	Groundwater	Groundwater	On-Site (a)	Resident	Adult	Ingestion	Quant	Exposure to Site groundwater as drinking water will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use.
						Inhalation	Quant	Exposure to Site groundwater via inhalation of volatiles while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use.
						Dermal	Quant	Exposure to Site groundwater via contact while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use.
					Child	Ingestion	Quant	Exposure to Site groundwater as drinking water will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use.
						Inhalation	Quant	Exposure to Site groundwater via inhalation of volatiles while showering/bathing will be evaluated to under a hypothetical scenario in which the Site is redeveloped for future residential use.
						Dermal	Quant	Exposure to Site groundwater via contact while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use.
				Commercial/ Industrial Worker	Adult	Ingestion	Quant	Exposure to Site groundwater as drinking water may occur if additional water supply wells are installed in the future.
			On-Site Water Supply Wells (NIKE-1 and NIKE-2)	Resident	Adult	Ingestion	Quant	Exposure to Site groundwater as drinking water from on-Site supply wells will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use and the existing carbon filtration system is turned off.
						Inhalation	Quant	Exposure to Site groundwater via inhalation of volatiles while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use and the existing carbon filtration system is turned off.
						Dermal	Quant	Exposure to Site groundwater via contact while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use and the existing carbon filtration system is turned off.
					Child	Ingestion	Quant	Exposure to Site groundwater as drinking water from on-Site supply wells will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use and the existing carbon filtration system is turned off.
						Inhalation	Quant	Exposure to Site groundwater via inhalation of volatiles while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use and the existing carbon filtration system is turned off.
						Dermal	Quant	Exposure to Site groundwater via contact while showering/bathing will be evaluated under a hypothetical scenario in which the Site is redeveloped for future residential use and the existing carbon filtration system is turned off.
				Commercial/ Industrial Worker	Adult	Ingestion	Quant	Exposure to Site groundwater as drinking water from on-Site supply wells will be evaluated under a future use scenario to assess potential risk under a scenario in which the existing carbon filtration system is turned off.
	Shallow Groundwater	Indoor Air	On-Site	Resident	Adult	Inhalation	Qual	Under a hypothetical scenario in which the Site is redeveloped for future residential use, future residents may inhale volatiles originating from shallow groundwater via the vapor intrusion pathway. A screening-level evaluation and qualitative discussion of this pathway will be performed.
					Child	Inhalation	Qual	
	Surface Water	Surface Water	Off-Site Streams and Wetlands	Recreational User	Adult	Ingestion	None	Exposure to off-Site surface water may occur under a wading scenario. Ingestion of surface water is considered to be negligible.
						Dermal	Quant	Dermal contact with off-Site surface water may occur under a wading scenario.
					Child	Ingestion	None	Exposure to off-Site surface water may occur under a wading scenario. Ingestion of surface water is considered to be negligible.
						Dermal	Quant	Dermal contact with off-Site surface water may occur under a wading scenario.
	Sediment	Sediment	Off-Site Streams and Wetlands	Recreational User	Adult	Ingestion	Quant	Incidental ingestion of sediment may occur while wading in the off-Site stream/wetland area.
						Dermal	Quant	Dermal contact with sediment may occur while wading in the off-Site stream/wetland area.
					Child	Ingestion	Quant	Incidental ingestion of sediment may occur while wading in the off-Site stream/wetland area.
						Dermal	Quant	Dermal contact with sediment may occur while wading in the off-Site stream/wetland area.

Notes:

(a) Measured via monitoring wells and piezometers.

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Soil
Exposure Medium:	Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Incidental Ingestion	On-Site Commercial/	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
	Industrial Worker			IR	Ingestion Rate	100	mg/day	USEPA, 2014	$\frac{CS \times IR \times EF \times ED \times CF \times FI \times RBA}{BW \times AT}$
	(Current/Future)			EF	Exposure Frequency	250	days/yr	USEPA, 2014	
				ED	Exposure Duration	25	year	USEPA, 2014	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	9,125	days	USEPA, 1989	
	Off-Site Resident	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
	(Hypothetical Future)			IR	Ingestion Rate	100	mg/day	USEPA, 2014	$\frac{CS \times IR \times EF \times ED \times CF \times FI \times RBA}{BW \times AT}$
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	20	years	USEPA, 2014	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	200	mg/day	USEPA, 2014	$\frac{CS \times IR \times EF \times ED \times CF \times FI \times RBA}{BW \times AT}$
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	6	years	USEPA, 2014	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
	Trespasser	Adolescent	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
	(Current/Future)	(7 to <16 years)		IR	Ingestion Rate	100	mg/day	USEPA, 2014 (2)	$\frac{CS \times IR \times EF \times ED \times CF \times FI \times RBA}{BW \times AT}$
				EF	Exposure Frequency	52	days/yr	(3)	
				ED	Exposure Duration	9	years	(4)	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	44	kg	USEPA, 2011 (5)	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	3,285	days	USEPA, 1989	

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Soil
Exposure Medium:	Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Incidental Ingestion	Recreational User (Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	100	mg/day	USEPA, 2014 (2)	CS x IR x EF x ED x CF x FI x RBA
				EF	Exposure Frequency	78	days/yr	(6)	BW x AT
				ED	Exposure Duration	20	years	USEPA, 2014 (7)	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	200	mg/day	USEPA, 2014 (9)	CS x IR x EF x ED x CF x FI x RBA
				EF	Exposure Frequency	78	days/yr	(6)	BW x AT
				ED	Exposure Duration	6	years	USEPA, 2014 (8)	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
	Construction Worker (Current/Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	330	mg/day	USEPA, 2002	CS x IR x EF x ED x CF x FI x RBA
				EF	Exposure Frequency	125	days/yr	(17)	BW x AT
				ED	Exposure Duration	1	year	USEPA, 2002	
				FI	Fraction Ingested from Site	1	unitless	(1)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	365	days	USEPA, 1989	

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe: Current/Future
Medium: Soil
Exposure Medium: Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Dermal	On-Site Commercial/ Industrial Worker (Current/Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	3,527	cm^2	USEPA, 2014 (10)	CS x SA x AF x ABS x EV x EF x ED x CF / BW x AT
				AF	Adherence Factor	0.12	mg/cm^2-event	USEPA, 2014 (11)	
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	250	days/yr	USEPA, 2014	
				ED	Exposure Duration	25	year	USEPA, 2014	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	9,125	days	USEPA, 1989	
	Off-Site Resident (Hypothetical Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	6,032	cm^2	USEPA, 2014 (12)	CS x SA x AF x ABS x EV x EF x ED x CF / BW x AT
				AF	Adherence Factor	0.07	mg/cm^2-event	USEPA, 2014 (13)	
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	20	years	USEPA, 2014	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	2,373	cm^2	USEPA, 2014 (14)	CS x SA x AF x ABS x EV x EF x ED x CF / BW x AT
				AF	Adherence Factor	0.2	mg/cm^2-event	USEPA, 2014 (15)	
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	6	years	USEPA, 2014	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
	Trespasser (Current/Future)	Adolescent (7 to <16 years)	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	3,624	cm^2	USEPA, 2011 (16)	CS x SA x AF x ABS x EV x EF x ED x CF / BW x AT
				AF	Adherence Factor	0.07	mg/cm^2-event	USEPA, 2014 (13)	
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	52	days/yr	(3)	
				ED	Exposure Duration	9	years	(4)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	44	kg	USEPA, 2011 (5)	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	3,285	days	USEPA, 1989	

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe: Current/Future
Medium: Soil
Exposure Medium: Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Dermal	Recreational User (Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	6,032	cm^2	USEPA, 2014 (12)	CS x SA x AF x ABS x EV x EF x ED x CF
				AF	Adherence Factor	0.07	mg/cm^2-event	USEPA, 2014 (13)	BW x AT
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	78	days/yr	(6)	
				ED	Exposure Duration	20	years	USEPA, 2014 (7)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	2,373	cm^2	USEPA, 2014 (14)	CS x SA x AF x ABS x EV x EF x ED x CF
				AF	Adherence Factor	0.2	mg/cm^2-event	USEPA, 2014 (15)	BW x AT
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	78	days/yr	(6)	
				ED	Exposure Duration	6	years	USEPA, 2014 (8)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
	Construction Worker (Current/Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	3,527	cm^2	USEPA, 2014 (10)	CS x SA x AF x ABS x EV x EF x ED x CF
				AF	Adherence Factor	0.3	mg/cm^2-event	USEPA, 2004 (18)	BW x AT
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(20)	
				EF	Exposure Frequency	125	days/yr	(17)	
				ED	Exposure Duration	1	year	USEPA, 2002	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	365	days	USEPA, 1989	

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Soil
Exposure Medium:	Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation of fugitive dust and volatiles	On-Site Commercial/ Industrial Worker (Current/Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	8	hrs/day	USEPA, 2014	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	250	days/yr	USEPA, 2014	(PEF + VF) x AT x CF1
				ED	Exposure Duration	25	year	USEPA, 2014	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	9,125	days	USEPA, 1989	
	Off-Site Resident (Hypothetical Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	24	hrs/day	USEPA, 2014	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	(PEF + VF) x AT x CF1
				ED	Exposure Duration	20	years	USEPA, 2014	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	24	hrs/day	USEPA, 2014	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	(PEF + VF) x AT x CF1
				ED	Exposure Duration	6	years	USEPA, 2014	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
	Trespasser (Current/Future)	Adolescent (7 to <16 years)	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	2	hrs/day	(19)	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	52	days/yr	(3)	(PEF + VF) x AT x CF1
				ED	Exposure Duration	9	years	(4)	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1,000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	3,285	days	USEPA, 1989	

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Soil
Exposure Medium:	Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Inhalation of fugitive dust and volatiles	Recreational User (Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	2	hrs/day	(19)	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	78	days/yr	(6)	(PEF + VF) x AT x CF1
				ED	Exposure Duration	20	years	USEPA, 2014 (7)	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1,000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	2	hrs/day	(19)	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	78	days/yr	(6)	(PEF + VF) x AT x CF1
				ED	Exposure Duration	6	years	USEPA, 2014 (8)	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1,000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
	Construction Worker (Current/Future)	Adult	On-Site	CS	Chemical Concentration in Soil	Chemical Specific	mg/kg	--	Intake (ug/m^3) =
				ET	Exposure Time	8	hrs/day	USEPA, 2014	CS x ET x EF x ED x CF2
				EF	Exposure Frequency	125	days/yr	(17)	(PEF + VF) x AT x CF1
				ED	Exposure Duration	1	year	USEPA, 2002	
				PEF	Particulate Emission Factor	1.36E+09	m^3/kg	USEPA, 2002	
				VF	Volatilization Factor	Chemical Specific	m^3/kg	USEPA, 2019	
				CF1	Conversion Factor 1	24	hrs/day	--	
				CF2	Conversion Factor 2	1000	µg/mg	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	365	days	USEPA, 1989	

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Soil
Exposure Medium:	Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name

Notes:

< = Less than.

RME = Reasonable Maximum Exposure.

USEPA = United States Environmental Protection Agency.

(1) Professional judgment; conservatively assumes 100 percent of soil ingested is from the Site.

(2) Default value for adult resident.

(3) Assumes access to Site soils may occur 2 days per week for 6 months (i.e., 26 weeks) of the year, based on professional judgment.

(4) Exposure duration reflects age-range of 7 to <16 years.

(5) Weighted average body weight for adolescent ages 6 to 16 years old.

(6) Assumes access to Site soils may occur 3 days per week for 6 months (i.e., 26 weeks) of the year, based on professional judgment.

(7) Default value for adult resident; assumes an adult recreational user accesses the Site for 20 years.

(8) Default value for child resident; assumes a child recreational user accesses the Site for 6 years.

(9) Default value for child resident.

(10) Represents the weighted mean surface area for males and females ages 21+, including head, hands, and forearms (USEPA, 2011; Table 7-2).

(11) Represents the arithmetic mean of weighted average of body-specific (hands, forearms, and face) mean adherence factors for adult commercial/industrial activities (USEPA, 2011; Table 7-20).

(12) Represents the weighted mean surface area for male and female adults, including head, hands, forearms, and lower legs (USEPA, 2011; Table 7-2).

(13) Represents the geometric mean (50th percentile) of weighted average body-specific (hands, forearms, lower legs and face) adherence factors for gardeners (USEPA, 2004; Exhibit C-2).

(14) Represents the weighted mean surface area for males and females ages 0 to <6 years old, including head, hands, forearms, lower legs, and feet (USEPA, 2011; Table 7-2).

(15) Represents the geometric mean (50th percentile) of weighted average body-specific (hands, forearms, lower legs and face) adherence factors for children playing (wet soil) (USEPA, 2004; Exhibit C-2).

(16) Represents the weighted mean surface area for males and females ages 6 to <16 years old, including hands, forearms, lower legs, and head (USEPA, 2011; Table 7-2).

(17) Construction activities are assumed to occur for 125 days over the course of a 1 year period (for RME scenario), based on professional judgment.

(18) Represents the geometric mean (50th percentile) of weighted average body-specific (face, forearms, and hands) adherence factors for construction workers (USEPA, 2004; Exhibit C-2).

(19) Professional judgement; assumes an exposure time of 2 hours per event.

(20) Professional judgement; assumes one event per day.

Sources:

USEPA, 1989. Risk Assessment Guidance for Superfund. Vol 1: Human Health Evaluation Manual, Part A. EPA/540/1-86/060.

USEPA, 2002. Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites.

USEPA, 2004. Risk Assessment Guidance for Superfund. Part E, Supplemental Guidance for Dermal Risk Assessment. Final. EPA/540/R/99/005.

USEPA, 2011. Exposure Factors Handbook. September 2011.

USEPA, 2012. Recommendations for default value for relative bioavailability of arsenic in soil. OSWER Directive 9200.1-113. December 2012.

USEPA, 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. February 6, 2014. Revised September 2015.

USEPA, 2019. Regional Screening Level Tables and Calculator. May 2019.

TABLE 3

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE SOIL AND COMBINED SURFACE/SUBSURFACE SOIL

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Soil
Exposure Medium:	Surface soil; Combined Surface/Subsurface Soil

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name

Unit Intake Calculations

Incidental Ingestion Intake [(mg/kg-day)(kg/mg)] = (IR x EF x ED x CF x FI x RBA))/(BW x AT) [CS and RBA are factored into the risk calculation in Table 7s]

Dermal Intake [(mg/kg-day)(kg/mg)] = (SA x AF x EV x EF x ED x CF)/(BW x AT) [CS and ABS are factored into the risk calculation in Table 7s]

Inhalation Intake [(ug/m^3)(kg/mg)] = (ET x EF x ED x CF2)/([PEF + VF] x AT x CF1) [CS and VF are factored into the risk calculation in Table 7s]

Commercial/Industrial Worker:	Cancer Ingestion Intake =	3.06E-07	Cancer Dermal Intake =	1.29E-06	Cancer Inhalation Intake =	6.00E-08
Commercial/Industrial Worker:	Noncancer Ingestion Intake =	8.56E-07	Noncancer Dermal Intake =	3.62E-06	Noncancer Inhalation Intake =	1.68E-07
Resident - Adult:	Cancer Ingestion Intake =	3.42E-07	Cancer Dermal Intake =	1.45E-06	Cancer Inhalation Intake =	2.01E-07
Resident - Adult:	Noncancer Ingestion Intake =	1.20E-06	Noncancer Dermal Intake =	5.06E-06	Noncancer Inhalation Intake =	7.05E-07
Resident - Child:	Cancer Ingestion Intake =	1.10E-06	Cancer Dermal Intake =	2.60E-06	Cancer Inhalation Intake =	6.04E-08
Resident - Child:	Noncancer Ingestion Intake =	1.28E-05	Noncancer Dermal Intake =	3.03E-05	Noncancer Inhalation Intake =	7.05E-07
Recreator - Adult:	Cancer Ingestion Intake =	7.63E-08	Cancer Dermal Intake =	3.22E-07	Cancer Inhalation Intake =	3.74E-09
Recreator - Adult:	Noncancer Ingestion Intake =	2.67E-07	Noncancer Dermal Intake =	1.13E-06	Noncancer Inhalation Intake =	1.31E-08
Recreator - Child:	Cancer Ingestion Intake =	2.44E-07	Cancer Dermal Intake =	5.80E-07	Cancer Inhalation Intake =	1.12E-09
Recreator - Child:	Noncancer Ingestion Intake =	2.85E-06	Noncancer Dermal Intake =	6.76E-06	Noncancer Inhalation Intake =	1.31E-08
Trespasser:	Cancer Ingestion Intake =	4.16E-08	Cancer Dermal Intake =	1.06E-07	Cancer Inhalation Intake =	1.12E-09
Trespasser:	Noncancer Ingestion Intake =	3.24E-07	Noncancer Dermal Intake =	8.21E-07	Noncancer Inhalation Intake =	8.73E-09
Construction Worker:	Cancer Ingestion Intake =	2.02E-08	Cancer Dermal Intake =	6.47E-08	Cancer Inhalation Intake =	1.20E-09
Construction Worker:	Noncancer Ingestion Intake =	1.41E-06	Noncancer Dermal Intake =	4.53E-06	Noncancer Inhalation Intake =	8.39E-08

Cancer risk from ingestion = Soil concentration x Cancer Ingestion Intake x Oral Cancer Slope Factor

Cancer risk from dermal contact = Soil concentration x Cancer Dermal Intake x Absorption Factor x Dermal Cancer Slope Factor

Cancer risk from inhalation = Soil concentration x Cancer Inhalation Intake x Inhalation Unit Risk

Hazard Index from ingestion = Soil concentration x Noncancer Ingestion Intake / Oral Reference Dose

Hazard Index from dermal contact = Soil concentration x Noncancer Dermal Intake x Absorption Factor / Dermal Reference Dose

Hazard Index from inhalation = Soil concentration x Noncancer Inhalation Intake / Inhalation Reference Concentration

TABLE 4

VALUES USED FOR DAILY INTAKE CALCULATIONS - SHALLOW GROUNDWATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Groundwater
Exposure Medium:	Shallow Groundwater

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Incidental Ingestion	Construction Worker (Current/Future)	Adult	On-Site; Excavation Trench	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	0.005	liters/day	USEPA, 1989; USEPA, 2011 (1)	$\frac{CW \times IR \times EF \times ED \times CF}{BW \times AT}$
				EF	Exposure Frequency	62.5	days/yr	(2)	
				ED	Exposure Duration	1	years	USEPA, 2002	
				CF	Conversion Factor	0.001	mg/ug	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	365	days	USEPA, 1989	
Dermal	Construction Worker (Current/Future)	Adult	On-Site; Excavation Trench	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Organics:
				DA	Dose Absorbed per Unit Area per Event	Chemical Specific	mg/cm^2-event	--	Intake (mg/kg-day) =
				SA	Surface Area	3,527	cm^2	USEPA, 2014	
				PC	Permeability Constant	Chemical Specific	cm/hr	--	$\frac{DA \times SA \times EV \times EF \times ED \times CF1 \times CF2}{BW \times AT}$
				ET	Event Time	4	hour/event	(3)	
				EV	Event Frequency	1	event/day	USEPA, 2004	
				EF	Exposure Frequency	62.5	days/yr	(2)	
				ED	Exposure Duration	1	years	USEPA, 2002	Inorganics:
				BW	Body Weight	80	kg	USEPA, 2014	Intake (mg/kg-day) =
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	$\frac{CW \times SA \times PC \times ET \times EV \times EF \times ED \times CF1 \times CF2}{BW \times AT}$
				ATnc	Averaging Time - noncancer	365	days	USEPA, 1989	
				CF1	Conversion Factor 1	0.001	L/cm^3	--	
				CF2	Conversion Factor 2	0.001	mg/ug	--	
Inhalation of volatiles	Construction Worker (Current/Future)	Adult	On-Site; Excavation Trench	CA	Chemical Concentration in Trench Air	Chemical Specific	ug/m^3	--	Intake (ug/m^3) =
				ET	Exposure Time	4	hrs/day	(3)	$\frac{CA \times ET \times EF \times ED}{AT \times CF}$
				EF	Exposure Frequency	62.5	days/yr	(2)	
				ED	Exposure Duration	1	year	USEPA, 2002	
				CF	Conversion Factor	24	hrs/day	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	365	days	USEPA, 1989	

TABLE 4

VALUES USED FOR DAILY INTAKE CALCULATIONS - SHALLOW GROUNDWATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Groundwater
Exposure Medium:	Shallow Groundwater

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name

Notes:

RME = Reasonable Maximum Exposure.

USEPA = United States Environmental Protection Agency.

(1) Based on professional judgment and USEPA guidance (1989 and 2011). Value is one-tenth of that assumed to occur during a swimming event via incidental ingestion. Assumes groundwater is not used for drinking water.

(2) Based on professional judgment; Assumes a construction worker contacts groundwater within an excavation trench for one-half of their time on-Site (i.e., 62.5 days out of 125 days) (for RME scenario).

(3) Based on professional judgment; Assumes contact with groundwater in an excavation trench occurs for 4 hours of a typical workday.

Sources:

USEPA, 1989. Risk Assessment Guidance for Superfund. Vol 1: Human Health Evaluation Manual, Part A. EPA/540/1-86/060.

USEPA, 2002. Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites.

USEPA, 2004. Risk Assessment Guidance for Superfund. Part E, Supplemental Guidance for Dermal Risk Assessment. Final. EPA/540/R/99/005.

USEPA, 2011. Exposure Factors Handbook. September 2011.

USEPA, 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. February 6, 2014. Revised September 2015.

Unit Intake Calculations

Incidental Ingestion Intake [(mg/kg-day)(L/ug)]= (IR x EF x ED x CF)/(BW x AT) [CW is factored into the risk calculation in Table 7s]

Dermal Intake [(mg/kg-day)(L/ug)(event/cm)]= (SA x EV x EF x ED x CF1 x CF2)/(BW x AT) [DA (including CW, PC, and ET) is factored into the risk calculation in Table 7s]

Inhalation Intake (unitless) = (ET x EF x ED)/(AT x CF) [CA is factored into the risk calculation in Table 7s]

Construction Worker:	Cancer Ingestion Intake =	1.53E-10	Cancer Dermal Intake =	1.08E-07	Cancer Inhalation Intake =	4.08E-04
Construction Worker:	Noncancer Ingestion Intake =	1.07E-08	Noncancer Dermal Intake =	7.55E-06	Noncancer Inhalation Intake =	2.85E-02

Cancer risk from ingestion = Groundwater concentration x Cancer Ingestion Intake x Oral Cancer Slope Factor

Cancer risk from dermal contact = Groundwater concentration x Cancer Dermal Intake x Absorption Factor x Dermal Cancer Slope Factor

Cancer risk from inhalation = Air concentration x Cancer Inhalation Intake x Inhalation Unit Risk

Hazard Index from ingestion = Groundwater concentration x Noncancer Ingestion Intake / Oral Reference Dose

Hazard Index from dermal contact = Groundwater concentration x Noncancer Dermal Intake x Absorption Factor / Dermal Reference Dose

Hazard Index from inhalation = Air concentration x Noncancer Inhalation Intake / Inhalation Reference Concentration

TABLE 5

VALUES USED FOR DAILY INTAKE CALCULATIONS - GROUNDWATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Groundwater
Exposure Medium:	Groundwater

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Ingestion	On-Site Commercial/ Industrial Worker (Future)	Adult	On-Site	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	1.2	liters/day	USEPA, 2011 (1)	CW x IR x EF x ED x CF
				EF	Exposure Frequency	250	days/yr	USEPA, 2014	BW x AT
				ED	Exposure Duration	25	years	USEPA, 2014	
				CF	Conversion Factor	0.001	mg/ug	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	9,125	days	USEPA, 1989	
	Resident (Current/Future)	Adult	On-Site and Off-Site	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	2.5	liters/day	USEPA, 2014	CW x IR x EF x ED x CF
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	BW x AT
				ED	Exposure Duration	20	years	USEPA, 2014	
				CF	Conversion Factor	0.001	mg/ug	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site and Off-Site	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	0.78	liters/day	USEPA, 2014	CW x IR x EF x ED x CF
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	BW x AT
				ED	Exposure Duration	6	years	USEPA, 2014	
				CF	Conversion Factor	0.001	mg/ug	--	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	

TABLE 5

VALUES USED FOR DAILY INTAKE CALCULATIONS - GROUNDWATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Groundwater
Exposure Medium:	Groundwater

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Dermal	Resident	Adult	On-Site and Off-Site	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Organics:
(showering/bathing)	(Current/Future)			DA	Dose Absorbed per Unit Area per Event	Chemical Specific	mg/cm^2-event	--	Intake (mg/kg-day) =
				SA	Surface Area	19,652	cm^2	USEPA, 2014	
				PC	Permeability Constant	Chemical Specific	cm/hr	--	$\frac{DA \times SA \times EV \times EF \times ED \times CF1 \times CF2}{BW \times AT}$
				ET	Event Time	0.71	hour/event	USEPA, 2014	
				EV	Event Frequency	1	event/day	USEPA, 2004	
				EF	Exposure Frequency	350	days/year	USEPA, 2014	
				ED	Exposure Duration	20	years	USEPA, 2014	Inorganics:
				BW	Body Weight	80	kg	USEPA, 2014	Intake (mg/kg-day) =
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	$\frac{CW \times SA \times PC \times ET \times EV \times EF \times ED \times CF1 \times CF2}{BW \times AT}$
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
				CF1	Conversion Factor 1	0.001	L/cm^3	--	
				CF2	Conversion Factor 2	0.001	mg/ug	--	
		Child	On-Site and Off-Site	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Organics:
				DA	Dose Absorbed per Unit Area per Event	Chemical Specific	mg/cm^2-event	--	Intake (mg/kg-day) =
				SA	Surface Area	6,365	cm^2	USEPA, 2014	
				PC	Permeability Constant	Chemical Specific	cm/hr	--	$\frac{DA \times SA \times EV \times EF \times ED}{BW \times AT}$
				ET	Event Time	0.54	hour/event	USEPA, 2014	
				EV	Event Frequency	1	event/day	USEPA, 2004	
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	6	years	USEPA, 2014	Inorganics:
				BW	Body Weight	15	kg	USEPA, 2014	Intake (mg/kg-day) =
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	$\frac{CW \times SA \times PC \times ET \times EV \times EF \times ED \times CF1 \times CF2}{BW \times AT}$
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
				CF1	Conversion Factor 1	0.001	L/cm^3	--	
				CF2	Conversion Factor 2	0.001	mg/ug	--	
Inhalation	Resident	Adult	On-Site and Off-Site	CA	Chemical Concentration in Shower Air	Chemical Specific	ug/m^3	--	Intake (ug/m^3) =
(showering/bathing)	(Current/Future)			ET	Exposure Time	0.71	hrs/day	USEPA, 2014	$\frac{CA \times ET \times EF \times ED}{AT \times CF}$
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	20	years	USEPA, 2014	
				CF	Conversion Factor	24	hrs/day	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	On-Site and Off-Site	CS	Chemical Concentration in Shower Air	Chemical Specific	ug/m^3	--	Intake (ug/m^3) =
				ET	Exposure Time	0.54	hrs/day	USEPA, 2014	$\frac{CA \times ET \times EF \times ED}{AT \times CF}$
				EF	Exposure Frequency	350	days/yr	USEPA, 2014	
				ED	Exposure Duration	6	years	USEPA, 2014	
				CF	Conversion Factor	24	hrs/day	--	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	

TABLE 5

VALUES USED FOR DAILY INTAKE CALCULATIONS - GROUNDWATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Current/Future
Medium:	Groundwater
Exposure Medium:	Groundwater

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name

Notes:

RME = Reasonable Maximum Exposure.

USEPA = United States Environmental Protection Agency.

(1) Mean consumer-only drinking water ingestion rate for adults (> 21 years of age) used for commercial worker (USEPA, 2011; Table 3-33).

Sources:

USEPA, 1989. Risk Assessment Guidance for Superfund. Vol 1: Human Health Evaluation Manual, Part A. EPA/540/1-86/060.

USEPA, 2004. Risk Assessment Guidance for Superfund. Part E, Supplemental Guidance for Dermal Risk Assessment. Final. EPA/540/R/99/005.

USEPA, 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. February 6, 2014. Revised September 2015.

Unit Intake Calculations

Incidental Ingestion Intake [(mg/kg-day)(L/ug)]= (IR x EF x ED x CF)/(BW x AT) [CW is factored into the risk calculation in Table 7s]

Dermal Intake [(mg/kg-day)(L/ug)(event/cm)]= (SA x EV x EF x ED x CF1 x CF2)/(BW x AT) [DA (including CW, PC, and ET) is factored into the risk calculation in Table 7s]

Inhalation Intake (unitless) = (ET x EF x ED)/(AT x CF) [CA is factored into the risk calculation in Table 7s]

Commercial/Industrial Worker	Cancer Ingestion Intake =	3.67E-06	Cancer Dermal Intake =	Not Applicable	Cancer Inhalation Intake =	Not Applicable
Commercial/Industrial Worker	Noncancer Ingestion Intake =	1.03E-05	Noncancer Dermal Intake =	Not Applicable	Noncancer Inhalation Intake =	Not Applicable
Resident - Adult:	Cancer Ingestion Intake =	8.56E-06	Cancer Dermal Intake =	6.73E-05	Cancer Inhalation Intake =	8.11E-03
Resident - Adult:	Noncancer Ingestion Intake =	3.00E-05	Noncancer Dermal Intake =	2.36E-04	Noncancer Inhalation Intake =	2.84E-02
Resident - Child:	Cancer Ingestion Intake =	4.27E-06	Cancer Dermal Intake =	3.49E-05	Cancer Inhalation Intake =	1.85E-03
Resident - Child:	Noncancer Ingestion Intake =	4.99E-05	Noncancer Dermal Intake =	4.07E-04	Noncancer Inhalation Intake =	2.16E-02

Cancer risk from ingestion = Groundwater concentration x Cancer Ingestion Intake x Oral Cancer Slope Factor

Cancer risk from dermal contact = Groundwater concentration x Cancer Dermal Intake x Absorption Factor x Dermal Cancer Slope Factor

Cancer risk from inhalation = Air concentration x Cancer Inhalation Intake x Inhalation Unit Risk

Hazard Index from ingestion = Groundwater concentration x Noncancer Ingestion Intake / Oral Reference Dose

Hazard Index from dermal contact = Groundwater concentration x Noncancer Dermal Intake x Absorption Factor / Dermal Reference Dose

Hazard Index from inhalation = Air concentration x Noncancer Inhalation Intake / Inhalation Reference Concentration

TABLE 6

VALUES USED FOR DAILY INTAKE CALCULATIONS - SEDIMENT

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe: Future
Medium: Sediment
Exposure Medium: Sediment

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Incidental Ingestion	Recreational User (Future)	Adult	Off-Site Streams/Wetlands	CS	Chemical Concentration in Sediment	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	100	mg/day	USEPA, 2014 (1)	CS x IR x EF x ED x CF x FI x RBA
				EF	Exposure Frequency	78	days/yr	(2)	BW x AT
				ED	Exposure Duration	20	years	USEPA, 2014	
				FI	Fraction Ingested from Site	1	unitless	(3)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	Off-Site Streams/Wetlands	CS	Chemical Concentration in Sediment	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				IR	Ingestion Rate	200	mg/day	USEPA, 2014 (4)	CS x IR x EF x ED x CF x FI x RBA
				EF	Exposure Frequency	78	days/yr	(2)	BW x AT
				ED	Exposure Duration	6	years	USEPA, 2014	
				FI	Fraction Ingested from Site	1	unitless	(3)	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				RBA	Relative Bioavailability Factor	0.6 (Arsenic only) / 1	unitless	USEPA, 2012	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
Dermal	Recreational User (Future)	Adult	Off-Site Streams/Wetlands	CS	Chemical Concentration in Sediment	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	6,032	cm^2	USEPA, 2014 (7)	CS x SA x AF x ABS x EV x EF x ED x CF
				AF	Adherence Factor	0.3	mg/cm^2-event	USEPA, 2004 (5)	BW x AT
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(6)	
				EF	Exposure Frequency	78	days/yr	(2)	
				ED	Exposure Duration	20	years	USEPA, 2014	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	80	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
		Child	Off-Site Streams/Wetlands	CS	Chemical Concentration in Sediment	Chemical Specific	mg/kg	--	Intake (mg/kg-day) =
				SA	Surface Area	2,373	cm^2	USEPA, 2014 (8)	CS x SA x AF x ABS x EV x EF x ED x CF
				AF	Adherence Factor	0.2	mg/cm^2-event	USEPA, 2004 (9)	BW x AT
				ABS	Dermal absorption fraction	Chemical Specific	unitless	--	
				EV	Event Frequency	1	event/day	(6)	
				EF	Exposure Frequency	78	days/yr	(2)	
				ED	Exposure Duration	6	years	USEPA, 2014	
				CF	Conversion Factor	1.00E-06	kg/mg	--	
				BW	Body Weight	15	kg	USEPA, 2014	
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	

TABLE 6

VALUES USED FOR DAILY INTAKE CALCULATIONS - SEDIMENT

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Future
Medium:	Sediment
Exposure Medium:	Sediment

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name

Notes:

< = Less than.

RME = Reasonable Maximum Exposure.

USEPA = United States Environmental Protection Agency.

(1) USEPA default value for adult ingestion of soil conservatively assumed for sediment.

(2) Assumes access to sediment may occur 3 days per week for the warmest 6 months (i.e., 26 weeks) of the year (May to October).

(3) Professional judgment; conservatively assumes 100 percent of sediment ingested is from the Site.

(4) USEPA default value for child ingestion of soil conservatively assumed for sediment.

(5) Represents the geometric mean weighted value for reed gatherers (USEPA, 2004; Exhibit 3-3).

(6) Professional judgment; assumes one event per day.

(7) Represents the weighted mean surface area for male and female adults, including head, hands, forearms, and lower legs (USEPA, 2011; Table 7-2).

(8) Represents the weighted mean surface area for males and females ages 0 to <6 years old, including head, hands, forearms, lower legs, and feet (USEPA, 2011; Table 7-2).

(9) Represents the geometric mean weighted value for children playing in wet soil (USEPA, 2004; Exhibit 3-3).

Sources:

USEPA, 1989. Risk Assessment Guidance for Superfund. Vol 1: Human Health Evaluation Manual, Part A. EPA/540/1-86/060.

USEPA, 2004. Risk Assessment Guidance for Superfund. Part E, Supplemental Guidance for Dermal Risk Assessment. Final. EPA/540/R/99/005.

USEPA, 2011. Exposure Factors Handbook. September 2011.

USEPA, 2012. Recommendations for default value for relative bioavailability of arsenic in soil. OSWER Directive 9200.1-113. December 2012.

USEPA, 2014. Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. February 6, 2014. Revised September 2015.

Unit Intake Calculations

Incidental Ingestion Intake [(mg/kg-day)(kg/mg)] = (IR x EF x ED x CF x FI x RBA))/(BW x AT) [CS and RBA are factored into the risk calculation in Table 7s]

Dermal Intake [(mg/kg-day)(kg/mg)] = (SA x AF x EV x EF x ED x CF)/(BW x AT) [CS and ABS are factored into the risk calculation in Table 7s]

Recreational User - Adult:	Cancer Ingestion Intake =	7.63E-08	Cancer Dermal Intake =	1.38E-06
Recreational User - Adult:	Noncancer Ingestion Intake =	2.67E-07	Noncancer Dermal Intake =	4.83E-06
Recreational User - Child:	Cancer Ingestion Intake =	2.44E-07	Cancer Dermal Intake =	5.80E-07
Recreational User - Child:	Noncancer Ingestion Intake =	2.85E-06	Noncancer Dermal Intake =	6.76E-06

Cancer risk from ingestion = Sediment concentration x Cancer Ingestion Intake x Oral Cancer Slope Factor

Cancer risk from dermal contact = Sediment concentration x Cancer Dermal Intake x Absorption Factor x Dermal Cancer Slope Factor

Hazard Index from ingestion = Sediment concentration x Noncancer Ingestion Intake / Oral Reference Dose

Hazard Index from dermal contact = Sediment concentration x Noncancer Dermal Intake x Absorption Factor / Dermal Reference Dose

TABLE 7

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE WATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Future
Medium:	Surface Water
Exposure Medium:	Surface Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name
Dermal	Recreational User (Future)	Adult	Off-Site Streams/Wetlands	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Organics:
				DA	Dose Absorbed per Unit Area per Event	Chemical Specific	mg/cm^2-event	--	Intake (mg/kg-day) =
				SA	Surface Area	6,032	cm^2	USEPA, 2014 (4)	
				PC	Permeability Constant	Chemical Specific	cm/hr	--	DA x SA x EV x EF x ED x CF1 x CF2
				ET	Event Time	3	hour/event	(3)	BW x AT
				EV	Event Frequency	1	event/day	USEPA, 2004	
				EF	Exposure Frequency	78	days/yr	(2)	
				ED	Exposure Duration	20	years	USEPA, 2014	Inorganics:
				BW	Body Weight	80	kg	USEPA, 2014	CW x SA x PC x ET x EV x EF x ED x CF1 x CF2
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	BW x AT
				ATnc	Averaging Time - noncancer	7,300	days	USEPA, 1989	
				CF1	Conversion Factor 1	0.001	L/cm^3	--	
				CF2	Conversion Factor 2	0.001	mg/ug	--	
		Child	Off-Site Streams/Wetlands	CW	Chemical Concentration in Water	Chemical Specific	ug/L	--	Organics:
				DA	Dose Absorbed per Unit Area per Event	Chemical Specific	mg/cm^2-event	--	Intake (mg/kg-day) =
				SA	Surface Area	2,373	cm^2	USEPA, 2014 (5)	
				PC	Permeability Constant	Chemical Specific	cm/hr	--	DA x SA x EV x EF x ED x CF1 x CF2
				ET	Event Time	3	hour/event	(3)	BW x AT
				EV	Event Frequency	1	event/day	USEPA, 2004	
				EF	Exposure Frequency	78	days/yr	(2)	
				ED	Exposure Duration	6	years	USEPA, 2014	Inorganics:
				BW	Body Weight	15	kg	USEPA, 2014	CW x SA x PC x ET x EV x EF x ED x CF1 x CF2
				ATc	Averaging Time - cancer	25,550	days	USEPA, 1989	BW x AT
				ATnc	Averaging Time - noncancer	2,190	days	USEPA, 1989	
				CF1	Conversion Factor 1	0.001	L/cm^3	--	
				CF2	Conversion Factor 2	0.001	mg/ug	--	

TABLE 7

VALUES USED FOR DAILY INTAKE CALCULATIONS - SURFACE WATER

REASONABLE MAXIMUM EXPOSURE

HUMAN HEALTH RISK ASSESSMENT

FORMER NIKE PR-79 CONTROL AREA

FOSTER, RHODE ISLAND

Scenario Timeframe:	Future
Medium:	Surface Water
Exposure Medium:	Surface Water

Exposure Route	Receptor Population	Receptor Age	Exposure Point	Parameter Code	Parameter Definition	Value	Units	Rationale/ Reference	Intake Equation/ Model Name

Notes:

N/A = Not applicable.

RME = Reasonable Maximum Exposure.

USEPA = United States Environmental Protection Agency.

(1) Assumes incidental ingestion while wading is one tenth the ingestion rate while swimming.

(2) Assumes dermal contact may occur while wading 3 days per week for the warmest 6 months (i.e., 26 weeks) of the year (May to October).

(3) Assumes contact with surface water for 3 hours on each day exposure occurs.

(4) Represents the weighted mean surface area for male and female adults, including head, hands, forearms, and lower legs (USEPA, 2011; Table 7-2).

(5) Represents the weighted mean surface area for males and females ages 0 to <6 years old, including head, hands, forearms, lower legs, and feet (USEPA, 2011; Table 7-2).

Sources:

USEPA, 1989: Risk Assessment Guidance for Superfund. Vol 1: Human Health Evaluation Manual, Part A. EPA/540/1-86/060.

USEPA, 2004: Risk Assessment Guidance for Superfund (Part E, Supplemental Guidance for Dermal Risk Assessment) Final. EPA/540/R/99/005.

USEPA, 2014: Human Health Evaluation Manual, Supplemental Guidance: Update of Standard Default Exposure Factors. February 6, 2014. Corrected February 2015.

Unit Intake Calculations

Dermal Intake [(mg/kg-day)(L/ug)(event/cm)]= (SA x EV x EF x ED x CF1 x CF2)/(BW x AT) [DA (including CW, PC, and ET) is factored into the risk calculation in Table 7s]

Recreational User - Adult (Wading):	Cancer Ingestion Intake =	N/A	Cancer Dermal Intake =	4.60E-06
Recreational User - Adult (Wading):	Noncancer Ingestion Intake =	N/A	Noncancer Dermal Intake =	1.61E-05
Recreational User - Child (Wading):	Cancer Ingestion Intake =	N/A	Cancer Dermal Intake =	2.90E-06
Recreational User - Child (Wading):	Noncancer Ingestion Intake =	N/A	Noncancer Dermal Intake =	3.38E-05

Cancer risk from ingestion = Surface water concentration x Cancer Ingestion Intake x Oral Cancer Slope Factor

Cancer risk from dermal contact = Surface water concentration x Cancer Dermal Intake x Absorption Factor x Dermal Cancer Slope Factor

Hazard Index from ingestion = Surface water concentration x Noncancer Ingestion Intake / Oral Reference Dose

Hazard Index from dermal contact = Surface water concentration x Noncancer Dermal Intake x Absorption Factor / Dermal Reference Dose

TABLE 8
EXPOSURE PARAMETERS FOR WILDLIFE RECEPTORS
ECOLOGICAL RISK ASSESSMENT
FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Receptor Species	Body Weight (kg)	Food Ingestion Rate (kg_{dw}/day)	Dietary Assumptions (%; kg_{dw}/day)			Incidental Sediment Ingestion (%; kg_{dw}/day)	Water Ingestion Rate (kg/day)	Average Home Range (acres)
			Soil / Wetland Invertebrates	Wetland / Terrestrial Plants	Amphibians			
Birds								
American robin	0.0804 [a]	0.0119 [b]	100% [c]	--	--	6.4% [d]	0.0109 [e]	0.6095 [f]
(Turdus migratorius)			0.0119	--	--	0.0008	0.00013	
Upland Exposures								
Bobwhite quail	0.1751 [a]	0.0088 [b]	--	100% [c]	--	6.1% [d]	0.018 [e]	24.7 [f]
(Colinus virginianus)			--	0.0088	--	0.00054	0.00016	
Upland Exposures								
Marsh Wren	0.0106 [a]	0.0029 [b]	100%	--	--	0.9% [d]	0.0028 [e]	0.295 [f]
(Cistothorus palustris)			0.0029	--	--	0.000026	0.0000080	
Stream/Wetland Exposures								
Mammals								
Raccoon	5.78 [a]	0.1535 [b]	46% [c]	42% [c]	12% [c]	9.4% [d]	0.48 [e]	1558 [f]
(Procyon lotor)			0.0706	0.0645	0.0184	0.014	0.074	
Stream/Wetland & Upland Exposures								
Meadow vole	0.033 [a]	0.0077 [b]	--	100% [c]	--	1.2% [d]	0.0046 [e]	0.066 [f]
(Microtus pennsylvanicus)			--	0.0077	--	0.000093	0.000035	
Stream/Wetland & Upland Exposures								
Short-tailed shrew	0.0161 [a]	0.0021 [b]	100% [c]	--	--	0.9% [d]	0.0024 [e]	0.96 [f]
(Blarina brevicauda)			0.0021	--	--	0.000019	0.0000051	
Stream/Wetland & Upland Exposures								

General Notes:

BW - Body Weight
DW - Dry Weight
FIR - Food Ingestion Rate
g - grams
kg - kilograms
L - liters
WIR - Water Ingestion Rate

Notes continued on following page.

TABLE 8
EXPOSURE PARAMETERS FOR WILDLIFE RECEPTORS
ECOLOGICAL RISK ASSESSMENT
FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Notes for American robin

[a] Average of adult body weights listed by USEPA (1993).

[b] Food ingestion rate calculated using algorithm for insectivorous birds developed by Nagy, 2001 [FIR (gdw/day) = $0.540*BW^{0.705}$] using average body weight.

[c] Diet assumed to be exclusively soil invertebrates.

[d] In the absence of data for the robin, the incidental soil ingestion rate is based on 50th percentile value for woodcock (USEPA, 2007a).

[e] Water ingestion rate calculated using algorithm for all birds developed by Calder and Braun, 1983 [WIR (kg/day) = $0.059*BW^{0.67}$] using average body weight.

[f] Home range listed by USEPA (1993) ranges from 0.27 to 1.04 acres with an average of 0.6095 acres.

Notes for Bobwhite quail

[a] Average of adult body weights listed by USEPA (1993).

[b] Food ingestion rate calculated using algorithm for quail birds developed by Nagy, 2001 [FIR (gdw/day) = $0.088*BW^{0.891}$] using average body weight.

[c] Diet assumed to be exclusively plants.

[d] In the absence of data for the quail, the incidental soil ingestion rate is based on 50th percentile value for mourning dove (USEPA, 2007a).

[e] Water ingestion rate calculated using algorithm for all birds developed by Calder and Braun, 1983 [WIR (kg/day) = $0.059*BW^{0.67}$] using average body weight.

[f] Home range listed by USEPA (1993) ranges from 8.9 to 41.3 acres with an average of 24.7 acres.

Notes for Marsh Wren

[a] Average of adult body weights listed by USEPA (1993).

[b] Food ingestion rate calculated using algorithm for insectivorous birds developed by Nagy, 2001 [FIR (g_{dw}/day) = $0.540*BW^{0.705}$] using average body weight.

[c] Diet assumed to be exclusively benthic invertebrates.

[d] In the absence of data for the wren, the incidental soil ingestion rate is based on 50th percentile value for shrew (consuming 100% earthworms; USEPA, 2007)a.

[e] Water ingestion rate calculated using algorithm for all birds developed by Calder and Braun, 1983 [WIR (kg/day) = $0.059*BW^{0.67}$] using average body weight.

[f] Home range for freshwater wetlands listed by USEPA (1993) ranges from 0.17 to 0.42 acres with an average of 0.295 acres.

Notes for Raccoon

[a] Average of adult body weights listed by USEPA (1993).

[b] Food ingestion rate calculated using algorithm for omnivorous mammals developed by Nagy, 2001 [FIR (g_{dw}/day) = $0.432*BW^{0.678}$] using average body weight.

[c] Diet composition based on the average of seasonal diets for raccoon in a Maryland forested bottomland (USEPA, 1993).

[d] The incidental soil ingestion rate is based on the value identified by Beyer (1994) for raccoons.

[e] Water ingestion rate calculated using algorithm for all mammals developed by Calder and Braun, 1983 [WIR (kg/day) = $0.099*BW^{0.90}$]using average body weight.

[f] Home range listed by USEPA (1993) ranges from 96 to 6325 acres with an average of 1558 acres.

Meadow vole

[a] Average of adult body weights listed by USEPA (1993).

[b] Food ingestion rate calculated using algorithm for herbivorous mammals developed by Nagy, 2001 [FIR (kg_{dw}/day) = $0.859*BW^{0.628}$] using average body weight.

[c] Diet is expected to be composed entirely of plants.

[d] The incidental soil ingestion rate is based on 50th percentile value for vole (USEPA, 2007a).

[e] Water ingestion rate calculated using algorithm for all mammals developed by Calder and Braun, 1983 [WIR (kg/day) = $0.099*BW^{0.90}$] using average body weight.

[f] Home range listed by USEPA (1993) ranges from 0.000494 to 0.2051 acres with an average of 0.0659 acres.

TABLE 8
EXPOSURE PARAMETERS FOR WILDLIFE RECEPTORS
ECOLOGICAL RISK ASSESSMENT
FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Notes for Short-tailed Shrew

[a] Average of adult body weights listed by USEPA (1993).

[b] Food ingestion rate calculated using algorithm for insectivorous mammals developed by Nagy, 2001 [FIR (kg_{dw}/day) = $0.373*BW^{0.622}$] using average body weight.

[c] An exclusive soil invertebrate diet was selected.

[d] The incidental soil ingestion rate is based on 50^{th} percentile value for shrew (USEPA, 2007a).

[e] Water ingestion rate calculated using algorithm for all mammals developed by Calder and Braun, 1983 [WIR (kg/day) = $0.099*BW^{0.90}$] using average body weight.

[f] Average home range for short-tailed shrew in Michigan, Manitoba, and New York (USEPA, 1993).

Appendix H

Laboratory LOQ Verification Acceptance Limits

8260 Aqueous Acceptance Limits

Target List	LOQ Lower Limits	LOQ Upper Limits
1,1,1-Trichloroethane	60	146
1,1-Dichloroethane	58	148
1,1-Dichloroethene	75	140
1,2,3-Trichlorobenzene	53	139
1,2,4-Trichlorobenzene	60	142
1,2,4-Trimethylbenzene	71	130
1,2-Dichlorobenzene	78	121
1,3,5-Trichlorobenzene	63	134
1,3-Dichlorobenzene	78	118
2-Butanone (MEK)	51	152
4-methyl-2-pentanone(MiBK)	70	135
Acetone	26	209
Benzene	76	126
Bromodichloromethane	72	135
Bromomethane	31	161
Carbon Disulfide	52	149
Chloroethane	18	192
Chloroform	62	144
Chloromethane	37	145
cis-1,2-Dichloroethene	72	135
Dibromochloromethane	74	130
Ethylbenzene	80	121
Isopropylbenzene	83	149
m+p-Xylene	79	126
Methylene Chloride	54	147
n-Butylbenzene	63	136
n-Propylbenzene	71	134
o-Xylene	82	125
p-Isopropyltoluene	77	132
Sec-Butylbenzene	68	135
Tertiary-butyl alcohol	10	198
tert-Butylbenzene	71	133
Toluene	73	129
trans-1,2-Dichloroethene	62	141
Trichloroethene	65	135
Vinyl Chloride	42	153

8260 Solid Acceptance Limits

Target List	LOQ Lower Limits	LOQ Upper Limits
1,1,1-Trichloroethane	66	134
1,1-Dichloroethane	57	143
1,1-Dichloroethene	49	159
1,2,3-Trichlorobenzene	29	160
1,2,4-Trichlorobenzene	36	160
1,2,4-Trimethylbenzene	64	128
1,2-Dichlorobenzene	63	131
1,3,5-Trichlorobenzene	41	156
1,3-Dichlorobenzene	65	132
2-Butanone (MEK)	55	171
4-methyl-2-pentanone(MiBK)	55	157
Acetone	30	200
Benzene	71	123
Bromodichloromethane	69	130
Bromomethane	41	160
Carbon Disulfide	47	161
Chloroethane	53	146
Chloroform	71	129
Chloromethane	50	147
cis-1,2-Dichloroethene	68	137
Dibromochloromethane	67	133
Ethylbenzene	71	122
Isopropylbenzene	73	152
m+p-Xylene	68	127
Methylene Chloride	24	184
n-Butylbenzene	52	142
n-Propylbenzene	63	136
o-Xylene	71	126
p-Isopropyltoluene	66	138
Sec-Butylbenzene	60	137
Tertiary-butyl alcohol	33	177
tert-Butylbenzene	66	131
Toluene	69	124
trans-1,2-Dichloroethene	46	154
Trichloroethene	73	123
Vinyl Chloride	53	154

8260 SIM Aqueous Acceptance Limits

Target List	LOQ Lower Limits	LOQ Upper Limits
1,2,3-Trichlorobenzene	50	150
1,2,4-Trichlorobenzene	50	150
Benzene	50	150
Chloroform	50	150
Trichloroethene (TCE)	50	150
Vinyl chloride	50	150

8270 SIM Aqueous Acceptance Limits

Target List	LOQ Lower Limits	LOQ Upper Limits
1,4-Dioxane	10	190
2-Methylnaphthalene	30	135
Acenaphthene	41	102
Acenaphthylene	38	122
Anthracene	62	103
Benzo(a)anthracene	57	123
Benzo(a)pyrene	51	110
Benzo(b)fluoranthene	55	114
Benzo(g,h,i)perylene	47	121
Benzo(k)fluoranthene	56	115
Chysene	62	103
Dibenzo(a,h)anthracene	52	122
Fluoranthene	72	118
Fluorene	39	109
Indeno(1,2,3-cd)pyrene	44	129
Naphthalene	33	97
Pentachlorophenol	10	85
Phenanthrene	64	109
Pyrene	60	115

8270 SIM Solid Acceptance Limits

Target List	LOQ Lower Limits	LOQ Upper Limits
1,4-Dioxane	10	190
2-Methylnaphthalene	10	199
Acenaphthene	11	120
Acenaphthylene	10	117
Anthracene	13	117
Benzo(a)anthracene	31	117
Benzo(a)pyrene	48	114
Benzo(b)fluoranthene	37	116
Benzo(g,h,i)perylene	36	120
Benzo(k)fluoranthene	33	112
Chysene	28	119
Dibenzo(a,h)anthracene	38	122
Fluoranthene	12	142
Fluorene	23	109
Indeno(1,2,3-cd)pyrene	32	123
Naphthalene	10	169
Pentachlorophenol	10	122
Phenanthrene	29	113
Pyrene	10	136

Metals Aqueous Acceptance Limits

Metals -ICP	Method	LOQ Lower Limits	LOQ Upper Limits
Aluminum	6010	67	133
Antimony	6010	67	133
Arsenic	6010	67	133
Barium	6010	67	133
Cadmium	6010	67	133
Calcium	6010	67	133
Chromium	6010	67	133
Cobalt	6010	67	133
Copper	6010	67	133
Iron	6010	67	133
Lead	6010	67	133
Magnesium	6010	67	133
Manganese	6010	67	133
Nickel	6010	67	133
Potassium	6010	67	133
Selenium	6010	67	133
Silver	6010	67	133
Sodium	6010	67	133
Thallium	6010	67	133
Vanadium	6010	67	133
Zinc	6010	67	133
Mercury			
Mercury	7471/7470	67	133
Metals -ICPMS			
Antimony	6020	67	133
Arsenic	6020	67	133
Cadmium	6020	67	133
Cobalt	6020	67	133
Lead	6020	67	133
Nickel	6020	67	133
Selenium	6020	67	133
Silver	6020	67	133
Thallium	6020	67	133

Metals Solids Acceptance Limits

Metals -ICP	Method	LOQ Lower Limits	LOQ Upper Limits
Aluminum	6010	67	133
Barium	6010	67	133
Calcium	6010	67	133
Chromium	6010	67	133
Copper	6010	67	133
Iron	6010	67	133
Lead	6010	67	133
Magnesium	6010	67	133
Manganese	6010	67	133
Nickel	6010	67	133
Potassium	6010	67	133
Silver	6010	67	133
Sodium	6010	67	133
Vanadium	6010	67	133
Zinc	6010	67	133
Mercury			
Mercury	7471/7470	67	133
Metals -ICPMS			
Antimony	6020	67	133
Arsenic	6020	67	133
Cadmium	6020	67	133
Cobalt	6020	67	133
Selenium	6020	67	133
Thallium	6020	67	133

Wet Chemistry Aqueous Acceptance Limits

	Method	LOQ Lower Limits	LOQ Upper Limits
Alkalinity	SM 2320 B	67	133
Sulfide	SM 4500 S2 F	67	133
TOC	9056A	67	133
Chloride	9056A	67	133
Sulfate	9056A	67	133
Nitrate	6010	67	133
Nitrite	6010	67	133
MEE			
Methane	RSK-175	50	150
Ethane	RSK-175	50	150
Ethene	RSK-175	50	150

Wet Chemistry Solid Acceptance Limits

	Method	LOQ Lower Limits	LOQ Upper Limits
TOC	Lloyd Kahn	67	133

AVS/SEM Acceptance Limits

	Method	LOQ Lower Limits	LOQ Upper Limits
AVS SEM			
Sulfide	EPA-821-R-51-100	67	133
Cadmium	6010	67	133
Copper	6010	67	133
Lead	6010	67	133
Nickel	6010	67	133
Silver	6010	67	133
Zinc	6010	67	133