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INTERIM OFFSHORE MONITORING PLAN FOR OPERABLE UNIT 4 (OU 4) REVISION 1 NSY PORTSMOUTH ME

11/1/2010

TETRA TECH NUS

Interim Offshore Monitoring Plan for Operable Unit 4 Revision 1

Portsmouth Naval Shipyard

Kittery, Maine

Naval Facilities Engineering Command Mid-Atlantic

Contract Number N62470-08-D-1001
Contract Task Order WE29

November 2010

SAP Worksheet #1A – Tetra Tech Title and Approval Page
(UFP-QAPP Manual Section 2.1)

INTERIM OFFSHORE MONITORING PLAN FOR OPERABLE UNIT 4, REVISION 1

PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE

Prepared for:
Naval Facilities Engineering Command Mid-Atlantic
9742 Maryland Avenue
Norfolk, Virginia 23511-3095

Prepared by:
Tetra Tech, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, Pennsylvania 19406

Prepared under:
Contract Number N62470-08-D-1001
Contract Task Order WE29

NOVEMBER 2010

Tetra Tech Review Signatures:

Daniel Witt, P.E.
Project Manager
Tetra Tech, Inc.

Tom Johnston, Ph.D.
Quality Assurance Manager
Tetra Tech, Inc.

SAP Worksheet #1B – Regulatory Title and Approval Page
(UFP-QAPP Manual Section 2.1)

INTERIM OFFSHORE MONITORING PLAN FOR
OPERABLE UNIT 4,
REVISION 1
PORTSMOUTH NAVAL SHIPYARD
KITTERY, MAINE

Prepared for:
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Contract Task Order WE29

NOVEMBER 2010

Approval Signatures :

Matthew Audet
Regulatory Remedial Project Manager
U.S. EPA Region 1

Iver McLeod
Regulatory Remedial Project Manager
MEDEP

SAP Worksheet #1C – Draft Title and Approval Page
(UFP-QAPP Manual Section 2.1)

Draft

INTERIM OFFSHORE MONITORING PLAN FOR OPERABLE UNIT 4

PORTSMOUTH NAVAL SHIPYARD
KITTERY, MAINE

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JUNE 2010

Approval Signatures :

Cole 6/9/10

Linda Cole, P.E.
Remedial Project Manager
NAVFAC MIDLANT
Northeast IPT
Code OPTE3-2

Jonathan Tucker 6/9/10

Jonathan Tucker
NAVFAC QA Officer
NAVFAC LANT

EXECUTIVE SUMMARY

This Operable Unit (OU) 4 Interim Offshore Monitoring Plan (IOMP), Revision 1 provides the basis and method for conducting interim monitoring for OU4 at Portsmouth Naval Shipyard (PNS), Kittery, Maine, subsequent to Round 10 until a final remedy is implemented for OU4. This plan incorporates modifications to the program as recommended in the Rounds 1 through 10 Interim Offshore Monitoring Program Report (Tt, February 2010). Tetra Tech (Tt) prepared this OU4 IOMP for the United States Department of Navy, Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62470-08-D-1001, Contract Task Order (CTO) WE29. This OU4 IOMP follows the format of the Uniform Federal Policy (UFP) for Quality Assurance Project Plans (QAPPs) (U.S. EPA, March 2005), and will be used during interim monitoring activities at OU4. OU4 is currently in the Feasibility Study (FS) stage of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process, and a final remedy has yet to be implemented. The remedy is scheduled to be in place by 2014.

OU4 includes areas offshore of PNS that potentially were impacted by PNS onshore Installation Restoration (IR) program sites. OU4 consists of Site 5, Former Industrial Waste Outfalls, and six offshore areas referred to as Areas of Concern (AOCs). Monitoring Stations (MS) were selected within OU4 to provide spatial coverage of the offshore AOCs.

The Navy, as lead agency, in consultation with U.S. EPA as a support agency, determined that interim monitoring was warranted for OU4. Per the Interim Record of Decision (ROD) (Navy, May 1999), the Navy is conducting monitoring in the offshore area of PNS until the FS is prepared and the final remedy has been implemented. Environmental media is being monitored to determine whether over the course of interim monitoring current and future concentrations of chemicals of concern in the offshore areas identified herein are at acceptable levels. In addition, interim monitoring is being used to meet the following objectives: (1) provide information on the current condition of the offshore areas; (2) provide information to support the identification and selection of any removal action, any additional interim action, or a final remedy; (3) be consistent with any final remedial action; and (4) provide a basis for any monitoring that may be incorporated as part of the final remedy.

Revision 0 of the IOMP (Tt, October 1999) was prepared as required by the Interim ROD to present the monitoring program for the interim action. Ten rounds of sampling have been conducted at OU4 from September 1999 to April 2010, as per the Revision 0 of the IOMP with modifications based on evaluation of the results of the monitoring. Modifications since initiation of the interim monitoring, including elimination of biota sampling, reduction of analytes, and reduction and changes in sampling locations, are summarized in Appendix D.

Modifications outlined in Revision 1 of the IOMP, to be implemented in Round 11, were made based on the evaluation of results from the 10 rounds of sampling and the recommendations in the Rounds 1 through 10 Interim Offshore Monitoring Program Report (Tt, February 2010). The recommendations in the Rounds 1 through 10 Report are also summarized in Appendix D.

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Appendices

Appendix A Field Documentation Forms
Appendix B Field Standard Operating Procedures
Appendix C Laboratory Standard Operating Procedures and Certifications
Appendix D Excerpts from the Rounds 1 thorugh 10 IOMP Report

ACRONYMS AND ABBREVIATIONS

AES	Atomic Emissions Spectrometer
AOC	Area of Concern
ARF	Administrative Record File
CA	Corrective Action
CAS	Chemical Abstract Service
CCC	calibration check compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLLE	Continuous Liquid Liquid Extractor
COC	chemicals of concern
COD	coefficient of determination
CSM	conceptual site model
CTO	Contract Task Order
% D	percent difference
DFTPP	decafluorotriphenyl phosphine
DoD	Department of Defense
DoN	Department of Navy
DQI	Data Quality Indicator
DQO	data quality objective
DQR	data quality review
DRMO	Defense Reutilization and Marketing Office
DVM	Data Validation Manager
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
ER-L	Effect Range-Low
ER-M	Effect Range-Median
FCS	Final Confirmation Study
FFA	Federal Facility Agreement
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
GC/MS	Gas Chromatograph/Mass Spectrometer

GIS	Geographic Information System
GPS	global positioning system
HASP	Health and Safety Plan
HMW	High Molecular Weight
HRGC/HRMS	High Resolution Gas Chromatography/High Resolution Mass Spectrometer
HSM	Health and Safety Manager
IAS	Initial Assessment Study
ICP	Inductively Coupled Plasma
ICB	Initial Calibration Blank
ICV	Independent Calibration Verification
ICS	Interference Check Standard
IDW	Investigation-derived waste
IRG	Interim Remediation Goal
IR	Installation Restoration
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
MDL	Method Detection Limit
MEDEP	Maine Department of Environmental Protection
MPC	Measurement Performance Criterion
MS	Monitoring Station
MS/MSD	matrix spike/matrix spike duplicate
NAD	North American Datum
NAVFAC	Naval Facilities Engineering Command
NFESC	Naval Facilities Engineering Service Center
NIRIS	Naval Installation Restoration Information Solution
NOAA	National Oceanic and Atmospheric Administration
NCCOSC	Naval Command, Control, and Ocean Surveillance Center
ORP	ongoing precision standard
ORPD	ongoing precision standard duplicate
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PAH	polycyclic aromatic hydrocarbon
PARCCS	Precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	polychlorinated biphenyl

PCDD	polychlorinated dibenzodioxin
PCDF	polychlorinated dibenzofuran
PF	project file
PFK	perfluorokerosene
PM	Project Manager
PNS	Portsmouth Naval Shipyard
POC	Point of contact
PPE	Personal protective equipment
PSL	Project Screening Level
PT	Proficiency Testing
PWD	Public Works Department
QA	quality assurance
QAM	Quality Assurance Manager
QAMS	Quality Assurance Management Staff
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quatitation Limit
QSM	Quality Systems Manual
%R	percent recovery
RAB	Restoration Advisory Board
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFI	RCRA Facility Investigation
RI	Remedial Investigation
ROD	Record of Decision
RPD	relative percent difference
RSD	relative standard deviation
RSL	Regional screening level
RT	Retention Time
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SHSC	Site Health and Safety Coordinator
SIM	Selective Ion Monitoring
SMP	Site Management Plan
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound

SSO	Site Safety Officer
SQL	Structured Query Language
TAL	Target Analyte List
TBD	To be determined
TCDD	2,3,7,8-tetrachlorodibenzo-p-dioxin
TCL	Target Compound List
TEF	Toxicity Equivalency Factor
TEQ	Toxicity Equivalency Quotient
Tt	Tetra Tech, Inc.
UFP	Uniform Federal Policy
U.S. EPA	United States Environmental Protection Agency
VOA	volatile organic analyte
WHO	World Health Organization

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: **OU4**
OU: **4**
Contractor Name: **Tt**
Contract Number: **N62470-08-D-1001**
Contract Title: **CLEAN**
Work Assignment Number: **CTO WE29**

1. This OU4 SAP was prepared per the guidelines of the United States Environmental Protection Agency's (U.S. EPA) UFP for QAPPs (March 2005) and Guidance for Quality Assurance Project Plans, EPA QA/G-5, Quality Assurance Management Staff (QAMS) (U.S. EPA, December 2002).

2. Identify regulatory program: CERCLA

3. This document is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Internal Meeting	November 17, 2009
Meeting with Regulators (conference call)	March 23, 2010

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date	ARF/PF NUMBER
Interim Record of Decision for Operable Unit 4	May 1999	N00102.AR.000676
Interim Offshore Monitoring Plan for Operable Unit 4	October 1999	N00102.AR.000750
Preliminary Remediation Goals for Operable Unit 4	November 2001	
Baseline Interim Offshore Monitoring Report for Operable Unit 4	July 2002	N00102.PF.00150
Rounds 1 through 7 Report Interim Offshore Monitoring Program	November 2004	N00102.AR.001416 and N00102.AR.001417
Additional Scrutiny Quality Assurance Project Plan for Operable Unit 4	August 2005	N00102.PF.001484 and N00102.PF.001467
Additional Scrutiny Report for Operable Unit 4	August 2007	N00102.PF.001612
Technical Memorandum-Recommendation for Modifications to the Interim Offshore Monitoring Program for Operable Unit 4	September 2008	N00102.PF.001682
Rounds 1 through 10 Interim Offshore Monitoring Program Report for Operable Unit 4	February 2010	N00102.AR.001716

6. List organizational partners (stakeholders):

NAVFAC Mid-Atlantic
U.S. EPA Region 1
Maine Department of Environmental Protection (MEDEP)

Natural Resource Trustees
PNS Restoration Advisory Board (RAB)

Lead organization: Navy – NAVFAC Mid-Atlantic

If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

Not applicable

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	Not Applicable
2	Table of Contents SAP Identifying Information	Not Applicable
3	Distribution List	Not Applicable
4	Project Personnel Sign-Off Sheet	Not Applicable
Project Organization		
5	Project Organizational Chart	Not Applicable
6	Communication Pathways	Not Applicable
7	Personnel Responsibilities and Qualifications Table	Not Applicable
8	Special Personnel Training Requirements Table	Not Applicable
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs Tables) Project Scoping Session Participants Sheet	Not Applicable
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Not Applicable
11	Site-Specific Project Quality Objectives	Not Applicable
12	Measurement Performance Criteria Table	Not Applicable
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Not Applicable
14	Summary of Project Tasks	Not Applicable
15	Reference Limits and Evaluation Table	Not Applicable
16	Project Schedule/Timeline Table	Not Applicable
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Not Applicable
18	Sampling Locations and Methods/SOP Requirements Table Sample Location Map(s)	Not Applicable
19	Analytical Methods/SOP Requirements Table	Not Applicable
20	Field Quality Control Sample Summary Table	Not Applicable

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
21	Project Sampling SOP References Table Sampling SOPs	Not Applicable
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Not Applicable
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	Not Applicable
24	Analytical Instrument Calibration Table	Not Applicable
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Not Applicable
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Not Applicable
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	Not Applicable
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	Not Applicable
Data Management Tasks		
29	Project Documents and Records Table	Not Applicable
30	Analytical Services Table Analytical and Data Management SOPs	Not Applicable
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Not Applicable
32	Assessment Findings and Corrective Action Responses Table	Not Applicable
33	QA Management Reports Table	Not Applicable
D. Data Review		
34	Verification (Step I) Process Table	Not Applicable
35	Validation (Steps IIa and IIb) Process Table	Not Applicable
36	Validation (Steps IIa and IIb) Summary Table	Not Applicable
37	Usability Assessment	Not Applicable

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipient	Title/Role	Organization (1)(2)	Telephone Number (Optional)	E-Mail Address
Linda Cole	Navy Remedial Project Manager (RPM)/Manages Project	NAVFAC Mid-Atlantic	(757) 341-2011	linda.cole@navy.mil
Matthew Audet	U.S. EPA RPM/Manages Project	U.S. EPA Region 1	(617) 918-1449	audet.matthew@epa.gov
Iver McLeod	MEDEP RPM/Manages Project	MEDEP	(207) 287-8010	iver.j.mcleod@maine.gov
Matt Thyng	Public Works Department (PWD) Maine Installation Restoration (IR) Program Manager / Shipyard Point of Contact (POC)	NAVFAC Mid-Atlantic	(207) 438-6618	frederick.thyng@navy.mil
Deborah Cohen	Facility Coordinator/Manages Tt work pertaining to the shipyard	Tt	(412) 921-7118	deborah.cohen@tetratech.com
Daniel Witt	Project Manager (PM)/Manages project on a daily basis	Tt	(412)921-8259	daniel.witt@tetratech.com
Kelly Perkins	PM/Manages project	Katahdin Analytical Services, Inc.	(207) 874-2400	kperkins@katahdinlab.com

Name of SAP Recipient	Title/Role	Organization (1)(2)	Telephone Number (Optional)	E-Mail Address
Tom Dolce	PM/Manages project	Aqua Survey, Inc.	(908) 788-8700	dolce@aquasurvey.com

1. SAP recipients from each organization will be responsible for distributing the SAP within their organization.
2. Tetra Tech and Tetra Tech subcontractors must be contacted through the Navy RPM.

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

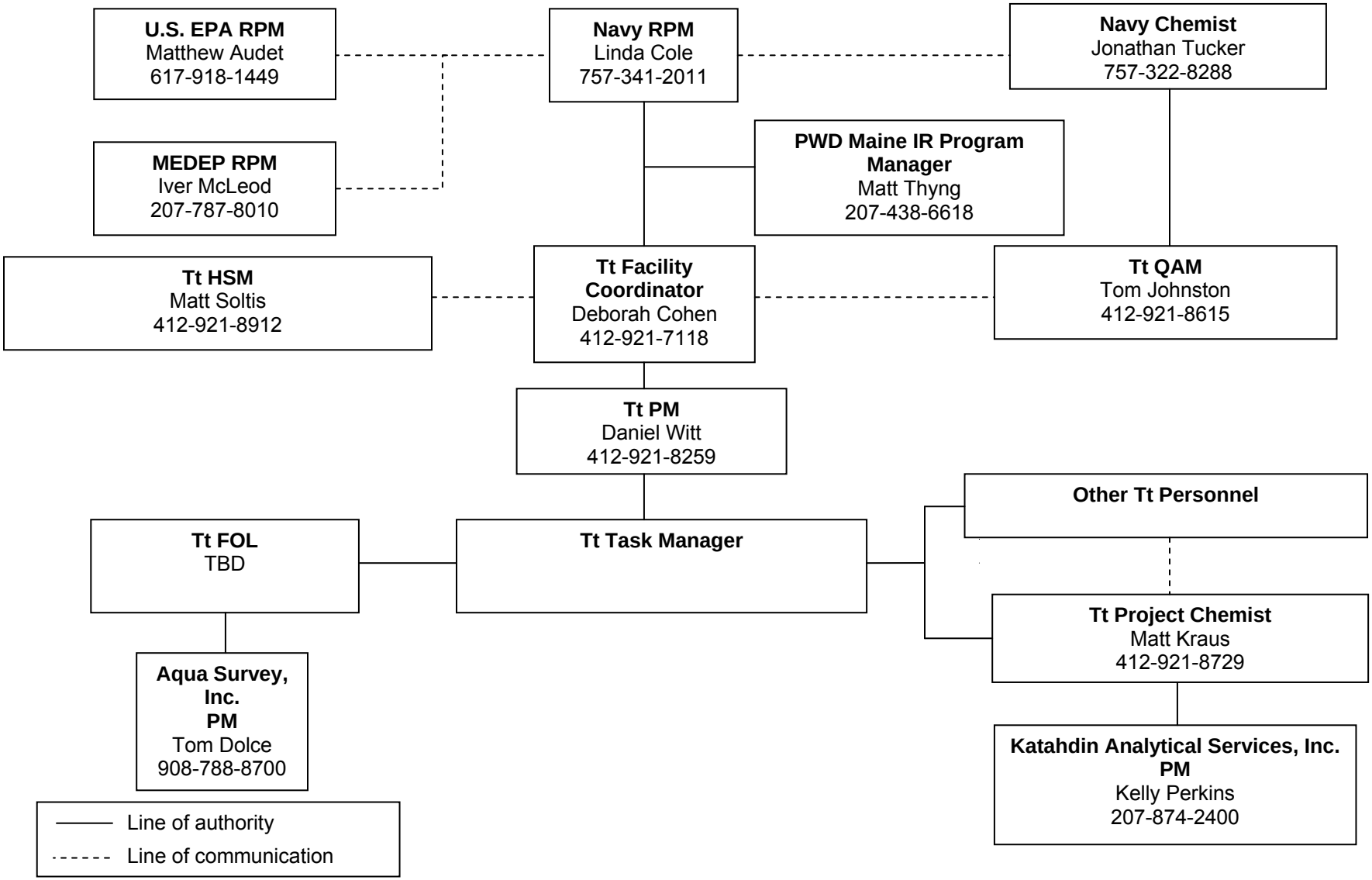
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Deborah Cohen	Tt/Facility Coordinator/Manages Tt work pertaining to PNS		All	
Daniel Witt	PM/Manages project	See Worksheet #1A Tetra Tech Title and Approval Page	All	
Tom Johnston	Tt/ Quality Assurance Manager (QAM)/Reviews and oversees project QA	See Worksheet #1A Tetra Tech Title and Approval Page	All	
Matt Soltis	Tt/ Health and Safety Manager (HSM)/Reviews and approves site Health and Safety Plan (HASP)			HASP
Joseph Samchuck	Tt/Data Validation Manager (DVM)/Oversees and approves data validation		Worksheets #12, 14, 15, 19, 20, 23-28, 30, 34-37	
Aaron Bernhardt	Tt//Ecological Risk Assessor/Performs ecological risk assessment evaluations		Worksheets #10,11, 15,17, and Appendix A	
TBD	Tt/Field Operations Leader (FOL)/Ensures that field operations are carried out in accordance with this SAP		All	

Name	Organization/Title/Role	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Matthew Kraus	Tt/Project Chemist/Coordinates with the laboratory and provides project chemistry support		All	
Kelly Perkins	Katahdin Analytical Services, Inc./PM/Manages project		Worksheets #12, 14, 15, 19, 20, 23-28, 30, 34-37	
Tom Dolce	Aqua Survey, Inc./PM/Manages project		Worksheets #10,11, 14,17,18,26,27, Appendix B, Appendix C	

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)



* All contact with Tt personnel and subcontractors by non-field personnel should be through Linda Cole.

SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Driver	Responsible Affiliation	Name	Phone Number	Procedure
SAP amendments	Tt PM NAVFAC RPM	Daniel Witt Linda Cole	412-921-8259 757-341-2011	The Tt PM will send the NAVFAC RPM a concurrence letter within 7 days of realizing an amendment is needed. The NAVFAC RPM will sign the letter within 5 days of receipt.
Schedule changes	Tt PM	Daniel Witt	412-921-8259	The Tt PM will send NAVFAC a schedule concurrence letter within 7 days of realizing a schedule impact or prior to the first affected deliverable date.
Field issues that require changes in scope or implementation of field work	Tt FOL Tt PM NAVFAC RPM	TBD Daniel Witt Linda Cole	TBD 412-921-8259 757-444-0806	The Tt FOL will inform the Tt PM verbally the day the issue is realized. Tt PM informs the NAVFAC RPM of the issue verbally within 1 day of the FOL's notification. Tt PM also sends a concurrence letter to the NAVFAC RPM within 7 days if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Document changes on a Field Task Modification Request (FTMR) form. Place the form in the project file, with signatures as determined by the Tt PM.

Communication Driver	Responsible Affiliation	Name	Phone Number	Procedure
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work	Tt FOL/Site Safety Officer (SSO) Tt PM Tt QAM	TBD Daniel Witt Tom Johnston	TBD 412-921-8259 412-921-8615	On the day the problem is identified, the Tt FOL, Tt SSO, or Tt QAM will inform Tt PM verbally and documents problem on an FTMR. On the same day of the notification from the Tt FOL, the Tt PM will inform the NAVFAC and Installation RPMs via e-mail.
Field or laboratory data issues	Laboratory PM Tt Project Chemist	Kelly Perkins Matt Kraus	207-874-2400 412-921-8729	When issue is related to chemical data, the Laboratory PM will notify the Tt Project Chemist verbally within 2 days of identification of problem. The Project Chemist will notify the Tt PM verbally within 1 day.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Linda Cole	RPM/Manages project	NAVFAC Mid-Atlantic	Oversees project implementation, including scoping, data review, and evaluation.
Iver McLeod	RPM/Manages Project	MEDEP	Participates in scoping, data review and evaluation, and approves the SAP preparation.
Matthew Thyng	PWD Maine IR Program Manager/Manages daily site activities related to this project	PNS	Oversees site activities, participates in scoping, data review, and evaluation, and reviews the SAP.
Matthew Audet	RPM/Manages project	U.S. EPA Region 1	Participates in scoping, data review and evaluation, and SAP preparation.
Deborah Cohen	Facility Coordinator/Manages Tt work pertaining to PNS	Tt	Oversees and manages Tt responsibilities associated with the shipyard.
Daniel Witt	PM/Manages project on a daily basis	Tt	Oversees project, financial, schedule, and technical day-to-day management of the project.
TBD	FOL/Manages field operations	Tt	Supervises, coordinates, and performs field sampling activities.
TBD	SSO/Oversees site activities to ensure that safety requirements are met	Tt	Responsible for on-site project specific health and safety training and monitoring site conditions. Details of these responsibilities are presented in the HASP.
Tom Johnston	QAM/Oversees program and project QA activities	Tt	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Joseph Samchuck	DVM/Oversees data validation activities	Tt	Manages data validation activities within Tt, including: • Ensuring QA of data validation deliverables. • Providing technical advice on data usability. • Coordinating and maintaining data validation review schedule.
Matt Soltis	HSM/Oversees health and safety activities	Tt	Oversees Tt CLEAN Health and Safety Program.
Matthew Kraus	Project Chemist/Oversees review of data quality and coordinates with the laboratory	Tt	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.

Name	Title/Role	Organizational Affiliation	Responsibilities
Kelly Perkins	Laboratory PM/Manages project	Katahdin Analytical Inc.	Coordinates analyses with laboratory chemists, ensures that the scope of work is followed, provides QA of data packages, and communicates with Tt project staff.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker performing sampling of hazardous materials will be required to have completed a 40-hour course (and annual 8-hour refresher) in health and safety training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements are addressed in greater detail in the Tt site-specific HASP.

Katahdin Analytical Services, Inc., the selected analytical laboratory, and SGS North America Inc., a laboratory subcontracted by Katahdin, are both accredited under the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP). DoD ELAP Certifications for Katahdin and SGS are included in Appendix C.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: PNS OU4 Interim Offshore Monitoring **PM:** Daniel Witt	**Site Name:** OU4 **Site Location:** PNS, Kittery, Maine

Date of Session: November 17, 2009
Scoping Session Purpose: Tt internal discussion to determine draft data quality objectives (DQOs).

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Aaron Bernhardt	Ecological Risk Assessor	Tt	(412) 921-8433	aaron.bernhardt@tetratech.com	Ecological risk assessment
Debbie Cohen, P.E.	Process Engineer	Tt	(412) 921-8729	debbie.cohen@tetratech.com	PNS Facility Coordinator
Martha Gray	Environmental Engineer	Tt	(412) 921-8194	martha.gray@tetratech.com	Task Manager
Ronald Kotun, Ph.D.	Senior Toxicologist	Tt	(412) 921-8291	ronald.kotun@tetratech.com	DQO Facilitator and Risk Assessor
Daniel Witt, P.E.	PM/Civil Engineer	Tt	(412) 921-8259	daniel.witt@tetratech.com	PM/Engineer

Comments/Decisions:

Draft SAP Worksheet #s 10 and 11 and SAP Figures 1 through 4 were reviewed. It was decided that the sampling plan would follow the recommendations for future monitoring in the Rounds 1 through 10 Interim Offshore Monitoring Program Report (Tt, February 2010). This report was draft at the time of this meeting; however the project team (including the Navy, MEDEP, and U.S. EPA) were in agreement with the recommendations of the draft report. Draft language for the problem statements and decision rules were discussed. It was discussed that the purpose of collecting additional data would be to provide an uninterrupted concentration record at the various monitoring stations until a remedy is chosen for OU4.

The pictorial conceptual site model (CSM) was discussed, and it was agreed that revisions would be made to the CSM figure. It was decided that Worksheet #'s 10 and 11 would be completed in addition to a Power Point presentation of the DQOs to facilitate discussion of the DQOs. It was decided that Worksheet #s 10 and 11 and the Power Point presentation would be sent to the Navy for review (on December 8, 2009) prior to presenting the DQOs to the regulators.

Action Items:

See Comments/Decisions above.

Consensus Decisions:

See Comments/Decisions above.

Project Name: OU4 Interim Offshore Monitoring Plan Update Projected Date(s) of Sampling: June 2011 **Project Manager:** Daniel Witt	**Site Name:** Operable Unit 4 **Site Location:** Sediment Monitoring Stations surrounding PNS

Date of Session: Conference call on March 23, 2010
Scoping Session Purpose: Discuss draft DQOs

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Linda Cole	Navy RPM	NAVFAC Mid-Atlantic	757-218-8747	linda.cole@navy.mil	RPM
Matt Audet	U.S. EPA RPM	U.S. EPA	617-918-1449	audet.matthew@epa.gov	RPM
Iver McLeod	MEDEP RPM	MEDEP	207-287-8010	Iver.j.mcleod@maine.gov	RPM
Debbie Cohen	Tt Facility Coordinator	Tt	412-921-7118	deborah.cohen@tetratech.com	Facility Coordinator
Dan Witt	Tt Project Manager	Tt	412-921-8259	daniel.witt@tetratech.com	PM

Comments/Decisions:

The project team reviewed the DQOs and information provided in draft versions of Worksheet #s 10 and 11 prepared to support the DQO discussion. There were no major concerns with the DQOs because the sampling is a continuation of routine monitoring that has been ongoing for 10 rounds over 11 years. The proposed sampling is in accordance with the recommendations of the Final Rounds 1 through 10 Interim Offshore Monitoring Program Report of Operable Unit 4 (Tt, February 2010).

Monitoring offshore of OU2 during the remediation of OU2 was discussed. It was decided that the Interim Offshore Monitoring Plan Update was not the appropriate document for any monitoring that might be required during the remediation of OU2.

The Rounds 1 through 10 Interim Offshore Monitoring Program Report recommended discontinuing sampling at five monitoring stations. It was discussed what vehicle would be used to document this decision. The project team agreed to discuss this further as a later date. This decision will not affect the Interim Offshore Monitoring Plan Update.

Synchronization of the five-year review sampling among various OUs at PNS was also discussed. It was agreed that the next sampling event for OU4 would be in spring 2011 and would occur at the same time as the five-year review sampling for OU3.

Consensus Decisions:

The DQOs are acceptable.

The next sampling event for OU4 would support the PNS five-year review and would occur in spring 2011.

SAP Worksheet #10 – Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)

This worksheet provides site information and a CSM for the OU4 Interim Offshore Monitoring Program. Although there is currently an existing Interim Offshore Monitoring Plan for OU4 (Tt, October 1999), it has been modified throughout the last 10 rounds of sampling.

10.1 SITE DESCRIPTION AND HISTORY

PNS is a military facility with restricted access. PNS spans all of Seavey Island and is situated in a tidal estuary of the Piscataqua River, which forms a border between Maine and New Hampshire (see Figure 1). PNS is located in the State of Maine about 1 mile north of Portsmouth, New Hampshire. The location of PNS places it at the mouth of the Great Bay Estuary (commonly referred to as Portsmouth Harbor). PNS is engaged in the conversion, overhaul, and repair of submarines for the Navy. PNS was established as a government facility in 1800, and it served as a repair and building facility for ships during the Civil War. PNS continues to service submarines as its primary military focus.

OU4 comprises areas offshore of PNS that were potentially affected by contamination from PNS onshore IR program sites. IR program sites include areas that are or have been scheduled for environmental investigations. OU4 consists of Site 5 (which is spread out over multiple locations) and six offshore areas referred to as Area of Concern (AOCs), as shown on Figure 2. In May 1999, an Interim Record of Decision (ROD) for OU4 (Navy, May 1999) was signed requiring the Navy to conduct monitoring in the offshore area of PNS in the interim period before completion of the offshore Feasibility Study (FS) (used to evaluate potential remedies for environmental problems) and implementation of the final remedy for OU4. Fourteen Monitoring Stations (MSs) were selected to provide coverage of the offshore AOCs for interim monitoring purposes, as shown on Figure 3. Four reference stations (not shown) located upstream and downstream of PNS in the Piscataqua River were sampled to provide information about non-PNS impacted areas.

Two IR program sites, Site 5 and Site 26, were identified as posing potential offshore impacts but no onshore impacts; therefore, these two sites were included as part of OU4. Site 5 consists of discharge points (outfalls) along the Piscataqua River at the western end of PNS in the dry dock area. The outfalls are believed to have been used from 1945 to 1975 to discharge liquid industrial wastes before the current Industrial Waste Treatment Plant was constructed. The wastewaters may have contained heavy metals (mercury, lead, cadmium, chromium, copper, and zinc), oils [potentially containing polychlorinated biphenyls (PCBs)] and grease. In 1978, dredging was conducted offshore in the vicinity of the outfalls (in the berth areas by the dry docks), and maintenance dredging is conducted periodically in the berth areas. Site 26 consists of portable oil/water tanks used along the berths and dry docks of PNS. Before 1980, acidic and alkaline cleaning solutions may have been stored in the tanks. Before 1991, wastes from

numerous spills during filling of the tanks reportedly discharged into the adjacent Piscataqua River near the dry docks. In August 2001, the Decision Document for Site 26 was signed indicating that no further action under CERCLA is necessary for Site 26; therefore, Site 26 is no longer included in OU4 (Navy, August 2001). However, previous releases from Site 26 to the offshore area are being included as part of the OU4 interim monitoring investigation.

The offshore areas at PNS include habitats categorized as pelagic, channel bottom/subtidal, eelgrass, intertidal mudflat, rocky intertidal, and salt marsh. The pelagic habitat around PNS is the open water of the Piscataqua River including the back channel of the river, Jamaica Cove, and Clark Cove. The channel bottom/subtidal habitat is the bottom of the pelagic area and includes hard-bottom areas and fine-grained depositional areas. The hard-bottom areas occur where the river experiences tidal scouring and active erosion such as offshore of PNS in the main flow of the Piscataqua River. The fine-grained depositional areas occur outside the main flow of the Piscataqua River, along the Back Channel, and in Jamaica Cove and Clark Cove. Eelgrass habitats, characterized by the presence of the rooted marine angiosperm *Zostera marina*, occur in subtidal areas by Jamaica Cove, Clark Cove, Sullivan Point, and the Dry Docks and in the Back Channel. Intertidal mudflats are generally muddy-sand or sandy-mud areas fringing the shoreline along the Back Channel, off Jamaica Island (in Jamaica Cove), and around Clark's Island. The rocky intertidal habitat occurs in many locations along Seavey and Jamaica Islands where the shoreline is exposed to river currents and where there are no appreciable fine-grained sediment accumulations [such as offshore of the Defense Reutilization and Marketing Office (DRMO), Site 6)]. Salt marsh habitats have been identified in Clark Cove, by Clark's Island, and in the Back Channel (including Jamaica Cove).

10.2 HISTORY OF ENVIRONMENTAL INVESTIGATIONS AND ACTIONS

OU4 is currently being investigated using the CERCLA process. Various investigations have been conducted at PNS including an Initial Assessment Study (IAS) (Weston, June 1983), Final Confirmation Study (FCS), (LEA, June 1986), Draft Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI Report (McLaren/Hart, July 1992), Human Health Risk Assessment (McLaren/Hart, May 1994), Public Health and Environmental Risk Evaluation: Part A Human Health Risk Assessment (PHERE) (McLaren/Hart, March 1994), RFI Data Gap Report (Halliburton NUS, November 1995), and an Estuarine Ecological Risk Assessment (NCCOSC, May 2000). The 1999 Interim ROD for OU4 (Navy, May 1999) required the Navy to conduct monitoring in the subtidal area of PNS during the interim period until final remedies were selected. The Interim Offshore Monitoring Plan for Operable Unit 4 (Tt, October 1999) was also completed in 1999 which is required by the 1999 Interim ROD.

Data from the first four rounds of sampling were evaluated and documented in the Baseline Interim Offshore Monitoring Report for OU4 (Tt, July 2002). The objective of the baseline report was to

recommend the appropriate season for future sampling. Subsequently, the data from the first seven rounds were evaluated and documented in the Rounds 1 through 7 Interim Offshore Monitoring Program Report (Tt, November 2004). The objective of this report was to determine the appropriate frequency of monitoring for each monitoring station for the next 5 years and to determine whether additional scrutiny was needed at any monitoring station. The Rounds 1 through 10 Report compiled in February 2010 summarized the results of the offshore investigations conducted at each monitoring and reference stations to date and presented recommendations and associated rationales for modifications to the monitoring program after Round 10 (Tt, February 2010). The findings of the previous investigations have been assimilated into a CSM (presented in Section 10.3) that explains current conditions and describes how those conditions might change over time.

10.3 CONCEPTUAL SITE MODEL

The CSM for the Interim Offshore Monitoring Program focuses on potential sources to, contaminants in, and exposure routes for organisms (i.e., receptors) potentially exposed to sediment in the offshore area. The CSM for OU4 is summarized on Figure 4, and the text below describes the CSM. Sediment is emphasized because contaminants that migrate via various mechanisms described below generally then accumulate in sediment either through direct deposition as particulates or through preferential partitioning from surface water to sediment.

10.3.1 Potential Sources of Contamination

The key environmental release, transport, and dispersion mechanisms that have resulted in contamination at OU4 are as follows:

- Historically, contaminants such as heavy metals, oils (potentially containing PCBs) and grease, were released into the Piscataqua River through outfalls. Specifically, Site 5, the Former Industrial Waste Outfalls, released contaminants to surface water at the Dry Docks AOC. With the activation of an Industrial Waste Treatment Plant at PNS, no hazardous wastes have been discharged through these outfalls since about 1975.

- Physical movement of contaminated soil (e.g., through snow plowing) at IR program sites prior to the paving over or vegetation of such soil was a secondary release mechanism that could have resulted in contamination of the offshore area. Snow was historically plowed over the shoreline into the Piscataqua River. Snow plowing activities have been recorded at OU2, DRMO Impact Area, which is adjacent to the offshore area MS-11. Currently, contaminated soil at PNS is either paved over or covered by vegetation; therefore, physical movement of contaminated soil is not a current release mechanism.

- Offshore sediment dredging activities that took place at PNS, without the use of turbidity curtains, could have resulted in the migration of contaminated sediment to offshore areas that were previously uncontaminated. Dredging activities have occurred at MS-12 and in the main channel of the Piscataqua River. Runoff from contaminated dredged materials placed onshore for dewatering, without monitoring or treatment of the runoff water, could also have resulted in contamination of offshore areas.

- Contaminated groundwater migration to sediment could have occurred from onshore at OU3 and OU7 to the offshore areas.

- Tidal erosion and storm water runoff from any IR program site at PNS could have resulted in contamination of offshore sediment. Suspended and dissolved contaminants carried by erosion or storm water could have been deposited in offshore areas. Erosion controls have been installed at MS-03, MS-04, MS-05, MS-06, MS-08, MS-09, and MS-11.

- Tidal erosion and storm water runoff from non-IR program sites at PNS could have resulted in contamination of offshore sediment. Contaminants carried by erosion or storm water could include contaminants from historical shipbuilding operations, materials used to construct buildings (e.g., roof shingles and tar paper), ambient background levels, or motor oils and gasoline that leak from vehicles.

- Non-PNS related activities, such as boating and fishing activities, could result in contamination of offshore sediment, including in reference areas. Contaminants could be released from spills during fueling activities, illegal dumping of waste, and possibly other mechanisms. Non-PNS contamination affecting reference areas currently and in the future would not have an impact on site environmental decisions; reference stations are no longer sampled because it was determined that chemicals of concern (COC) reference concentrations are less than COC concentrations in areas affected by onshore activities (Tt, February 2010). However, future non-PNS contamination (e.g. oil tanker spill) could affect site decisions in those areas already affected by onshore activities.

10.3.2 Potential Exposure Routes and Receptors

The primary ecological risk to benthic invertebrates is from exposure to bioavailable/bioaccessible COCs in sediment. Exposure routes of contaminants in sediment to benthic invertebrates include directed contact, ingestion, and ingestion of prey. The biologically active zone in sediment varies depending on season, grain size, and currents. Sediment from 0 to 10 cm includes the biologically active zone (benthic organisms are living and mixing sediment within this depth) and was the general depth of surficial sediment investigation during previous sediment investigations around PNS.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

11.1 PROBLEM DEFINITION

OU4 is currently in the FS stage of the CERCLA process, and a final remedy for OU4 has yet to be implemented. The remedy, which will be selected after completion of the FS, is scheduled to be in place by 2014. Per the Interim ROD (Navy, May 1999), interim offshore monitoring will continue until a final remedy is selected for OU4. Environmental media will be monitored to determine whether over the course of interim monitoring current and future concentrations of chemicals of concern in the offshore areas indentified herein are at acceptable levels [the project screening levels (PSLs)]. In addition, interim monitoring will be used to meet the following objectives: (1) provide information on the current condition of the offshore areas; (2) provide information to support the identification and selection of any removal action, any additional interim action, or a final remedy; (3) be consistent with any final remedial action; and (4) provide a basis for any monitoring that may be incorporated as part of the final remedy.

11.2 IDENTIFY INFORMATION INPUTS

Data and information that will be required to respond to the decision statement includes the following:

- **Analytical Data**

  Requirements for sediment sampling and analysis methods are described below. Additional details (e.g., the exact methods and SOPs to be used) are provided in Worksheet #s 15, 19, and 21.

  As presented in Table 6-1 of the Rounds 1 through 10 Interim Offshore Monitoring Report (Tt, February 2010) samples will be collected from select monitoring stations on a biennial basis, and at all monitoring stations every 5 years until a final remedy for OU4 is implemented in accordance with the Interim ROD for OU4 (Navy, May 1999). The biennial samples will be analyzed for the parameters that were determined to be of greatest potential concern at those stations and have IRGs or PRGs (except for lead which is a chemical of concern but does not have an IRG or PRG) to determine if concentrations of those chemicals are decreasing over time. However, the 5-year samples will be analyzed for a larger list of parameters (presented in Tt, February 2010) to ensure that these additional parameters are not becoming a problem at those locations. Worksheet #17 provides the list of stations for biennial and five year sampling.

- **Required Analyses**

  Required analyses are provided in Worksheet #18.

- **Analytical Methods**

Analytical methods were selected based on recommendations from the Rounds 1 through 10 Interim Offshore Monitoring Report (Tt, February 2010). Future sediment samples must be analyzed for metals using standard U.S. EPA analytical methods (U.S. EPA Method 6010B/6020 for all metals except mercury and U.S. EPA Method 7471A for mercury). Regression equations presented in Appendix E of the Rounds 1 through 10 Interim Offshore Monitoring Report should be used to convert the Interim Remediation Goals (IRGs) for copper and nickel developed using the National Oceanic and Atmospheric Administration (NOAA) method to IRGs based on results from the U.S. EPA analytical method. For lead, the regression equation should be used to convert lead concentrations when using standard U.S. EPA analytical methods for comparing future results to previous results analyzed using NOAA methods. Analytical methods for all other contaminants must remain consistent with previous rounds of monitoring.

- **Project Screening Levels**

  Detection limits were set below IRGs that were developed for OU4 (Tt, November 2001). If IRGs were not available for analytes to be analyzed, then conservative ecological screening criteria were used to set detection limits to ensure that those analytes could be detected at levels protective of offshore ecological receptors. Target analytes, Project Screening Levels (PSLs), and detection limits are presented in Worksheet #15.

- **Decision Documents**

  Decision documents (such as RODs) related to OU4 will be reviewed before a sampling round is conducted to determine if sampling is still required.

- **Sampling Methods**

  The sampling methods were selected to be the same as those used previously or at least consistent with the representativeness and comparability of sediment samples collected previously. Sediment sampling must be completed using a Smith-MacIntyre grab sampler or equivalent sediment sample collection device at all monitoring stations where sediment is available. Worksheet #18 lists sampling methods.

11.3 DELINEATION OF STUDY BOUNDARY

The geographical/physical boundary of OU4 is illustrated on Figure 2 as the offshore area of PNS potentially impacted by onshore IR program sites. Figure 3 presents the boundaries of the 14 MSs for OU4. Future monitoring must occur at nine MSs based on the recommendations in the Rounds 1 through 10 Interim Offshore Monitoring Report. Future sampling must be conducted at the locations sampled during the last round.

The vertical boundary for sampling is 10 cm below the sediment surface, which is consistent with previous sediment studies at PNS and will allow direct comparison of data collected under this plan to previously collected data.

These lateral and vertical boundaries represent sediment populations potentially affected by contaminants from onshore activities and sediments that have been monitored in the past under the Interim Offshore Monitoring Program. Continued investigation of these populations is critical to achieving a temporally continuous data set. If future site conditions require relocation of sampling points, a preference for and emphasis on collecting data from these populations is paramount. If site conditions change such that sediment becomes unavailable at a particular location, another location representative of the targeted sediment population in that area must be selected, if available.

11.4 DECISION RULE

Determine if COC concentrations are within the range of corresponding COC concentrations by comparing the most recently collected COC concentration data to historical COC data collected from the same area. If the most recently collected COC concentrations are within the concentration range of corresponding historical COC concentrations in the same area, then continuing monitoring until a final remedy is selected as specified in the Interim ROD (Navy, May 1999). If the most recently collected COC concentrations are not within the concentration range of corresponding historical COC concentrations in the same area, then consult the project team to determine future actions.

11.5 PERFORMANCE CRITERIA

The project team will use future interim monitoring results to determine whether the amount, type, and quality of data collected are sufficient to support the Interim Offshore Monitoring Program. The amount (i.e., number of samples) and type (i.e., analyses and sample locations) must be checked against the amounts and types of data specified in this SAP to ensure that the correct amounts and types of data were collected. The quality of the data will be evaluated via data verification, data validation, and a data quality review as specified in Worksheet #s 34, 35, 36, and 37. If the data do not appear to be compromised by quality deficiencies, the data set must be considered sufficient. Spatial contamination patterns, contamination levels, the ability to collect data from intended populations, and analytical sensitivity, among other factors, will be evaluated. Spatial contamination patterns must be evaluated by comparing concentrations of a given analyte across MSs. The ability to collect data from intended populations must be evaluated by reviewing sampling logs sheets and determining via information on that sheet (i.e., sample coordinates and descriptions) that the intended sediment populations were sampled. Contamination levels and analytical sensitivity must be evaluated by comparing analyte concentrations and detections limits to the corresponding analyte PALs and detection limits in Worksheet #15.

The project team selected locations and numbers of samples that based on previous sampling events and corresponding data evaluations will support the attainment of the stated project objectives. If all intended data have been collected and the data do not appear to be compromised by quality deficiencies, the data set will be considered complete and sufficient to attain project objectives. If data quality deficiencies are detected, the project team will evaluate them on a case-by-case basis and take corrective action if necessary.

Results of data validation (i.e., data qualifications) will be considered in these evaluations with an increased tendency to recommend additional investigation with decreased data quality. The data usability evaluation process is described in more detail in Worksheet #37.

11.6 PLAN FOR OBTAINING DATA

The plan for obtaining the data for the OU4 Interim Offshore Monitoring Plan is described in detail in Worksheet #17 along with the sampling designs and rationales.

SAP Worksheet #12 -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field Quality Control Samples – Sediment

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicates	Metals	One per 10 samples per matrix	Precision/ Comparability	Values ≥ 5x Quantitation Limit (QL): relative percent difference (RPD) ≤ 50 percent Values < 5x QL: absolute difference ≤ 4x QL	S & A
	4,4'-DDT, Dioxins/Furans, Polycylic Aromatic Hydrocarbons (PAHs), PCB Congeners	One per 10 samples per sediment matrix	Precision/ Comparability	RPD ≤ 50 percent; If samples results are < 2x QL, professional judgment is used	S & A
Equipment Blanks	Metals, 4,4'-DDT, Dioxins/Furans, PAH, PCB Congeners	One per sampling event	Bias/Contamination	No target analytes > QL for organic compounds or MDLs for inorganic analytes	S
Cooler Temperature Indicator	Metals, 4,4'-DDT, Dioxins/Furans, PAH, PCB Congeners	One per cooler	Accuracy/ Representativeness	Between 2 and 6 degrees C.	S
All Samples	Metals, 4,4'-DDT, Dioxins/Furans, PAH, PCB Congeners	All samples	Sensitivity	QL < PSLs listed in Worksheet #15	A
			Data Completeness	95 percent overall	S & A

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title, and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Draft Final Rounds 1 through 10 Interim Offshore Monitoring Program Report for OU4	Tt, February 2010	Tt/sediment/analytical/September 1999 to December 2008	Historical contaminant concentration ranges will be compared to concentrations collected for corresponding contaminants collected from similar areas.	None

SAP Worksheet #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

Summary of Project Tasks

Sampling Tasks:

- Mobilization/demobilization
- Site-specific health and safety training
- Obtain sampling coordinates. A global positioning system (GPS) unit capable of sub meter accuracy will be used to record sampling locations and obtain sampling coordinates.
- Sediment sampling at MS-01, MS-03, MS-04, MS-05, MS-07, MS-08, MS-09, MS-11, and MS-12 (see Figures 5 through 10)
- Equipment decontamination
- Investigation-derived waste (IDW) will be drummed by Tt. PNS will provide the appropriate IDW containers, and PNS is responsible for sampling, analyzing, and disposing of IDW in accordance with state and federal guidelines.
- The Tt FOL will contact the PWD IR POC prior to any sampling event to arrange for the proper disposal of IDW.
- Field quality assurance/quality control (QA/QC) management tasks
- Field documentation QA/QC and review tasks
- Sample custody and shipment task

Analysis Tasks:

- The fixed-base laboratories will analyze sediment samples for PAHs, metals, PCB congeners, pesticides, and dioxins/furans as outlined in Worksheet #s11 and 15.

Quality Control Tasks:

- Implement standard operating procedures (SOPs) for sampling activities, sample preservation/shipping methods, and sample preparation/analysis methods in accordance with the SAP.
- Collect and analyze QA/QC field samples including duplicates and temperature blanks (for coolers).
- Field duplicate samples will be collected following the same sampling procedure as environmental samples.

- Laboratory analysis of matrix spike/matrix spike duplicate (MS/MSD) samples.
- Calibration and other QC checks as specified in Worksheet #s 24 and 28.

Secondary Data:

- See Worksheet #13.

Data Management Tasks:

- Data receipt and Electronic Data Deliverable (EDD) upload (Sample Management Coordinator)
- Data verification and validation (Data Validation Chemist or Environmental Scientist and DVM)
- Upload of validated data to the project database (Project Database Manager)
- Upload of validated data to Naval Installation Restoration Information Solution (NIRIS).

Documentation and Records:

- Documentation of sample location coordinates, chain-of-custody forms, samples logs, and shipping documents for all samples.
- The EDDs and corresponding databases will be formatted in both Navy and MEDEP formats.
- Preparation of electronic and hard copies of the finalized IOMP for OU4.

Data Packages:

- Receipt of analytical data packages from the fixed-base laboratory.
- Generation of Tt data validation reports.
- Generation of Rounds 11, 12, and 13 OU4 Interim Offshore Monitoring Data Packages.

Data Review Tasks:

- The fixed-base laboratory will verify that all samples listed on the chain-of-custody form are analyzed in accordance with methods specified on the laboratory scope of work, and in this SAP. Data verification and validation will be performed by Tt as provided in Worksheet #s 35 and 36. A data validation report will be produced for each Sample Delivery Group (SDG).

- All field data records and validated data will be reviewed by Tt to determine the usability of the data. The outcome of this assessment will be conveyed to the project team for agreement before the project report is finalized. Data limitations pertaining to attainment of Project Quality Objectives and Action Limits, if identified, will be conveyed to the project team, and corrective actions will be taken as necessary.

SAP Worksheet #15 -- Reference Limits for Sediment

(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment
Analytical Groups: Metals, PAHs, 4,4'-DDT, PCB Congeners, and Dioxins/Furans

Analyte	CAS Number	Screening Level	Screening Level Reference[1]	Project QL Goal	Laboratory Detection Limits[2]	
					QL	MDL
POLYCYLIC AROMATIC HYDROCARBONS (µg/kg)						
1,1-BIPHENYL	**92-52-4**	**17**	**SQuiRTs MS T20**	**5.7**	**20**	**1.1**
ACENAPHTHENE	**83-32-9**	**16**	**SQuiRTs MS ERL**	**5.3**	**20**	**1.5**
ACENAPHTHYLENE	208-96-8	210	OU4 IRG	70	20	1.2
ANTHRACENE	120-12-7	1236	OU4 IRG	412	20	1.2
BENZO(A)ANTHRACENE	56-55-3	261	SQuiRTs MS ERL	87	20	1.9
BENZO(A)PYRENE	50-32-8	430	SQuiRTs MS ERL	143	20	3.3
BENZO(B)FLUORANTHENE	205-99-2	130	SQuiRTs MS T20	43	20	2.4
BENZO(E)PYRENE	192-97-2	-	-	-	20	3.3
BENZO(G,H,I)PERYLENE	191-24-2	67	SQuiRTs MS T20	22	20	2.0
BENZO(K)FLUORANTHENE	207-08-9	70	SQuiRTs MS T20	23	20	3.1
CHRYSENE	218-01-9	384	SQuiRTs MS ERL	128	20	1.7
DIBENZO(A,H)ANTHRACENE	53-70-3	63.4	SQuiRTs MS ERL	21	20	1.8
DIBENZOTHIOPHENE	132-65-0	-	-	-	20	0.47
FLUORANTHENE	206-44-0	600	SQuiRTs MS ERL	200	20	1.8
FLUORENE	86-73-7	500	OU4 IRG	167	20	3.2
INDENO(1,2,3-CD)PYRENE	193-39-5	-	-	-	20	1.9
NAPHTHALENE	91-20-3	160	SQuiRTs MS ERL	53	20	2.6
PERYLENE	198-55-0	74	SQuiRTs MS T20	25	20	0.82
PHENANTHRENE	85-01-8	240	SQuiRTs MS ERL	80	20	1.8
PYRENE	129-00-0	665	SQuiRTs MS ERL	222	20	2.1
METALS (mg/kg)						
ALUMINUM	**7429-90-5**	**18**	**SQuiRTs MS AET**	**6.0**	**30**	**2.9**
ARSENIC	7440-38-2	8.2	SQuiRTs MS ERL	2.7	0.8	0.09
CADMIUM	7440-43-9	1.2	SQuiRTs MS ERL	0.40	1	0.007

Analyte	CAS Number	Screening Level	Screening Level Reference[1]	Project QL Goal	Laboratory Detection Limits[2]	
					QL	MDL
METALS (mg/kg)						
CHROMIUM	7440-47-3	81	SQuiRTs MS ERL	27	1.5	0.1
COPPER	7440-47-3	486	OU4 IRG	162	2.5	0.095
IRON	7439-89-6	22	SQuiRTs MS AET	7.3	10	1.3
LEAD	7439-92-1	426	2x ER-M	142	0.5	0.14
MANGANESE	7439-95-4	260	SQuiRTs MS AET	87	0.5	0.079
MERCURY	7439-97-6	0.15	SQuiRTs MS ERL	0.05	0.04	0.002
NICKEL	7440-02-0	124	OU4 IRG	41	4	0.033
SILVER	**7440-22-4**	**1**	**SQuiRTs MS ERL**	**0.33**	**1.5**	**0.065**
ZINC	7440-66-6	150	SQuiRTs MS ERL	50	2.5	0.12
4,4'-DDT (µg/kg)						
4,4'-DDT	50-29-3	66.4	OU4 PRG	22	3	3
PCB CONGENERS (µg/kg)						
PCB-101	37680-73-2	1.26	SQuiRTs MS [3]	0.42	1.0	0.22
PCB-105	3259-81-44	3	Bird TEQ [4]	1	1.0	0.52
PCB-114	74472-37-0	3	Bird TEQ [4]	1	1.0	0.09
PCB-118	31508-00-6	3	Bird TEQ [4]	1	1.0	0.23
PCB-123	65510-44-3	3	Bird TEQ [4]	1	1.0	0.16
PCB-126	**5746-52-88**	**0.75**	**Bird TEQ [4][5]**	**0.25**	**1.0**	**0.16**
PCB-128	38380-07-3	1.26	SQuiRTs MS [3]	0.42	1.0	0.2
PCB-138	35065-28-2	1.26	SQuiRTs MS [3]	0.42	1.0	0.2
PCB-153	35065-27-1	1.26	SQuiRTs MS [3]	0.42	1.0	0.2
PCB-156	38380-08-4	3	Bird TEQ [4]	1	1.0	0.11
PCB-167	52663-72-6	3	Bird TEQ [4]	1	1.0	0.058
PCB-169	32774-16-6	3	Bird TEQ [4]	1	1.0	0.13
PCB-170	35065-30-6	1.26	SQuiRTs MS [3]	0.42	1.0	0.2
PCB-18	37680-65-2	1.26	SQuiRTs MS [3]	0.42	1.0	0.28
PCB-180	35065-29-3	1.26	SQuiRTs MS [3]	0.42	1.0	0.19
PCB-187	52663-68-0	1.26	SQuiRTs MS [3]	0.42	1.0	0.55
PCB-189	39635-31-9	3	Bird TEQ [4]	1	1.0	0.069

Analyte	CAS Number	Screening Level	Screening Level Reference[1]	Project QL Goal	Laboratory Detection Limits[2]	
					QL	MDL
PCB-195	52663-78-2	1.26	SQuiRTs MS [3]	0.42	1.0	0.19
PCB-157	69782-90-7	3	Bird TEQ [4]	1	1.0	0.08
PCB-206	40186-72-9	1.26	SQuiRTs MS [3]	0.42	1.0	0.17
PCB-209	2051-24-3	1.26	SQuiRTs MS [3]	0.42	1.0	0.23
PCB-28	7012-37-5	1.26	SQuiRTs MS [3]	0.42	1.0	0.66
PCB-44	41464-39-5	1.26	SQuiRTs MS [3]	0.42	1.0	0.21
PCB-52	35693-99-3	1.26	SQuiRTs MS [3]	0.42	1.0	0.23
PCB-66	32598-10-0	1.26	SQuiRTs MS [3]	0.42	1.0	0.23
PCB-77	32598-13-3	1.0	Bird TEQ [4]	0.33	1.0	0.20
PCB-8	34883-43-7	1.26	SQuiRTs MS [3]	0.42	1.0	0.26
PCB-81	**70362-50-4**	**0.75**	**Bird TEQ [4][5]**	**0.25**	**1.0**	**0.25**
TRANS-NONACHLOR	39765-80-5	3.99	OU4 PRG	1.33	1.7	0.08
Dioxins/Furans (ng/kg)						
2,3,7,8-TCDD	1746-01-6	1	Bird TEQ [4]	0.33	0.5	0.105
2,3,7,8-TCDF	51207-31-9	1	Bird TEQ [4]	4	0.5	0.112
1,2,3,7,8-PECDD	**40321-76-4**	**1**	**Bird TEQ [4]**	**0.33**	**2.5**	**0.436**
1,2,3,7,8-PECDF	57117-41-6	12	Bird TEQ [4]	14	2.5	0.152
2,3,4,7,8-PECDF	**57117-31-4**	**1**	**Bird TEQ [4]**	**1.3**	**2.5**	**0.207**
1,2,3,4,7,8-HXCDD	39227-28-6	25	Bird TEQ [4]	4	2.5	0.204
1,2,3,6,7,8-HXCDD	57653-85-7	120	Bird TEQ [4]	4	2.5	0.357
1,2,3,7,8,9-HXCDD	19408-74-3	12	Bird TEQ [4]	4	2.5	0.146
1,2,3,4,7,8-HXCDF	70648-26-9	12	Bird TEQ [4]	4	2.5	0.167
1,2,3,6,7,8-HXCDF	57117-44-9	12	Bird TEQ [4]	4	2.5	0.192
1,2,3,7,8,9-HXCDF	72918-21-9	12	Bird TEQ [4]	4	2.5	0.242
2,3,4,6,7,8-HXCDF	60851-34-5	12	Bird TEQ [4]	4	2.5	0.326
1,2,3,4,6,7,8-HPCDD	35822-46-9	1200	Bird TEQ [4]	40	2.5	0.270
1,2,3,4,6,7,8-HPCDF	67562-39-4	120	Bird TEQ [4]	40	2.5	0.326
1,2,3,4,7,8,9-HPCDF	55673-89-7	120	Bird TEQ [4]	40	2.5	0.139

Analyte	CAS Number	Screening Level	Screening Level Reference[1]	Project QL Goal	Laboratory Detection Limits[2]	
					QL	MDL
Dioxins/Furans (ng/kg)						
OCDD	3268-87-9	12000	Bird TEQ [4]	1400	5	0.382
OCDF	39001-02-0	12000	Bird TEQ [4]	1400	5	0.814

1 Screening Level References

Bird TEQ: Bird Toxicity Equivalent Quotient screening level of 0.21 µg/kg presented in Table 5-2 of the Rounds 1 Through 10 Interim Offshore Monitoring Program Report for OU4 (Tt, February 2010).
2x ER-M: The lead Effect Range–Median screening value multiplied by 2.
OU4 IRG: Operable Unit 4 Interim Remediation Goal value.
PRG: Preliminary Remediation Goal (Tt, November 2001).
SQuiRTs MS: National Oceanic and Atmospheric Administration Screening Quick References Tables Marine Sediment values. AET = Apparent Effects Threshold ; ER-L = Effect Range-Low; T20 = 20-percent effect level. http://response.restoration.noaa.gov/book_shelf/122_NEW-SQuiRTs.pdf.

2 Laboratory detection limits were provided by Katahdin Analytical Services, Inc., in January 2010 for the Round 11 sampling event and will be updated based on the Katahdin Analytical Services quarterly method detection limit (MDL) study that is current at the time of the Round 11 sampling event. Laboratory detection limits must be updated with each subsequent round of sampling that occurs during the OU4 Interim Offshore Monitoring Program to reflect the laboratory's most recent detection limits.

3 Calculated based on a total PCB congeners screening level (ER-L) of 22.7 µg/kg. Total PCB congener values were calculated by summing the following 18 PCB congeners, substituting one-half of the detection limits for non-detects: PCB-101/90, -105, -118, -128, -138/160, -153/132, -170/190, -18/17, -180, -187, -195/208, -206, -209, -28, -44, -52, -66, and -8/5, and multiplying the value by 2.0 because previous studies indicate that the 18 NOAA PCBs typically account for about one-half of the PCB concentration in a sample (NOAA, 1989).

4 Calculated based on the bird TEQ screening level of 0.21 µg/kg presented in Table 5-2 of the Rounds 1 Through 10 Interim Offshore Monitoring Program Report for OU4 (Tt, February 2010). Dioxins and furans were evaluated using Toxicity Equivalent Factors (TEFs) that relate the toxicity of various congeners to the toxicity of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), as specified in U.S. EPA's Interim Procedures for Estimating Risks Associated with Exposure to Mixtures of Chlorinated Dibenzo-p-Dioxins and Dibenzofurans (CDD and CDFs) 1989 Update. The World Health Organization (WHO) TEF values published in TEFs for PCBs, PCDDs, PCDFs for Humans and Wildlife (Van den Berg, et al., 2006) were used to calculate TEQ values for each congener. The concentration of each dioxin/furan was multiplied by the associated TEF to produce a TEQ value for each dioxin/furan congener. The individual TEQ values for all dioxins/furan were summed for each sample for an overall TEQ value for that sample. If any congeners were reported as non-detected in a sample, two total TEQ values were developed for that sample. In one case, the TEQ value was the sum of the toxic equivalent values of dioxin/furan congeners using zero for non-detects [i.e., TEQ BIRD (using bird TEFs)]. In the other case, the TEQ value was the sum of the toxic equivalent values using one-half the detection limit for non-detects [i.e.,TEQ BIRD HALFND (using bird TEFs)].

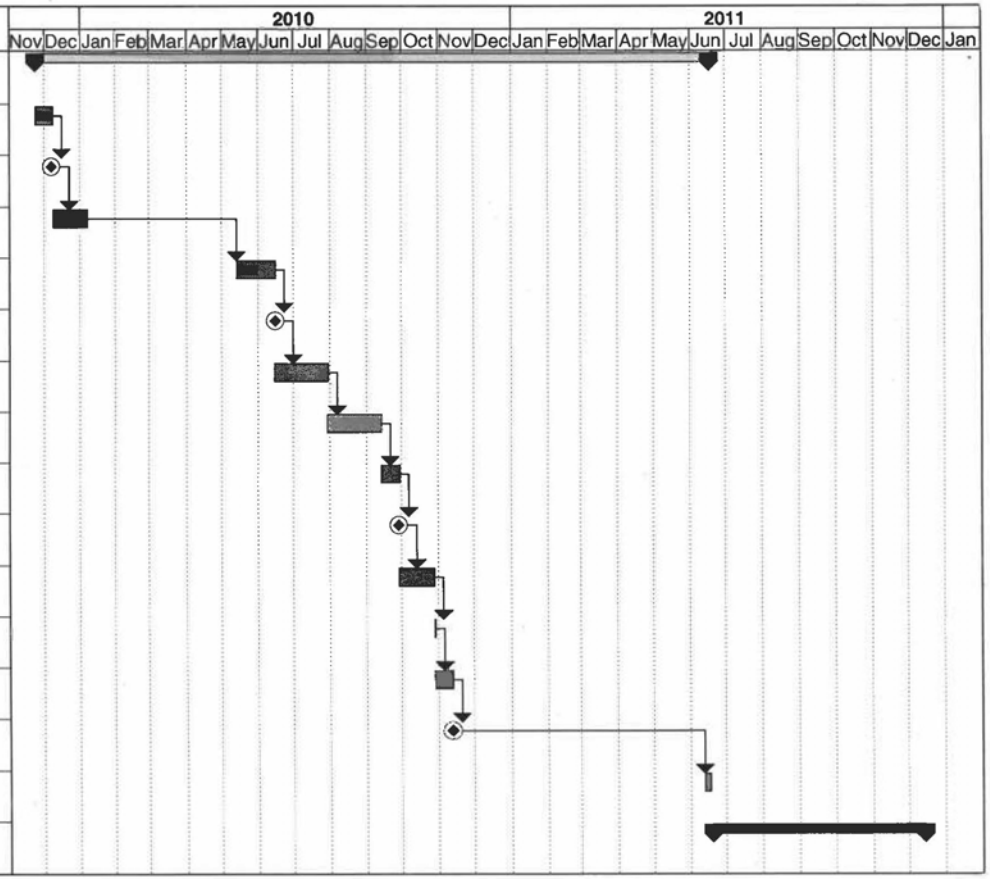
5 The project team is aware that the calculated PCB screening level is greater than the laboratory's lowest listed detection limit; however, this PCB compound will be used in combination with other PCB compounds to calculate a total PCB concentration that will be compared to a total PCB screening value. Individual PCB congener screening values were calculated and presented as listed in this table so that the total PCB concentration calculated will not exceed the total PCB screening value if all of the PCB congeners contributing to the total PCB calculation are reported as non-detected.

Bold rows indicate that the screening level is less than the corresponding QL but greater than the corresponding MDL.
Bold and shaded rows indicate that the screening level is less than the corresponding QL and the MDL.

SAP Worksheet #16 -- Project Schedule/Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Task	Duration
Interim Offshore Monitoring Plan for Operable Unit 4	409 d
Prepare DQOs	11 d
Submit DQOs	***0 d***
Navy Review	22 d
Prepare Draft UFP-SAP	23 d
Submit Draft UFP-SAP	***0 d***
Regulatory Review	34 d
Receive Comments/Comment Resolution	32 d
Prepare Draft Final UFP-SAP	12 d
Submit Draft Final UFP-SAP	***0 d***
Regulatory Review	22 d
Receive Comments/Comment Resolution	1 d
Prepare Final UFP-SAP	11 d
Submit Final UFP-SAP	***0 d***
Round 11 Sampling Event	5 d
Submit Final Round 11 Data Package	128 d



Project Manager:
Date: Fri 6/11/10

Task		External Tasks		External Milestone	
Progress		Project Summary		Deadline	
Milestone		Split			
Summary		Rolled Up Split			

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

The OU4 sampling strategy for Rounds 11 and subsequent rounds is the sampling design that was established in Revision 0 of the Interim Offshore Monitoring Plan for OU4 (Tt, October 1999) that incorporates modifications recommended in the Rounds 1 through 10 Interim Offshore Monitoring Program Report (Tt, February 2010). A summary of the modifications to the sampling program and recommended modifications based on the Rounds 1 through 10 Report are provided in Appendix D. The sampling and analysis program will monitor environmental media to determine whether over the course of interim monitoring current and future concentrations of chemicals in the offshore areas indentified herein are at acceptable levels. This updated monitoring program focuses on contaminant concentration monitoring in sediment because water-borne contaminants from PNS dilute rapidly once mixed with estuarine water, and particulate-bound contaminants tend to accumulate in nearby sediment depositional areas (Tt, October 1999). Sediment depositional areas around PNS were identified as areas most likely to be impacted by PNS IR program, sites; therefore, sediment is assumed to be the primary reservoir of shipyard-related contamination that could adversely affect offshore ecological receptors (Tt, October 1999).

Originally, it was determined that three sediment samples per monitoring location were required to account for the expected concentration variability at a given station and to provide estimates of chemical concentration trends over time (Tt, October 1999). At least three sediment samples will be collected from each MS sampled except for MS-11, where a lack of available sediment limits the collection of sediment to a single location. The sediment sampling locations within MSs were selected based on ensuring that habitats of concern were sampled utilizing the habitat prioritization outlined in the Interim Offshore Monitoring Plan for OU4 (Tt, October 1999). Habitat prioritization was listed as follows:

1. Mussel bed
2. Eelgrass bed
3. Seep location
4. Salt marsh
5. Subtidal habitat

Because sediment from 0 to 10 cm (0 to 4 inches) includes the biologically active zone (benthic organisms are living and mixing sediment within this depth) and was the general depth of surficial sediment investigation during previous sediment investigation around PNS, sediment contaminant monitoring will focus on sediment from 0 to 10 cm (Tt, October 1999).

Sediment Sample Collection

Sediment sampling locations have changed over the first 10 rounds of monitoring because of a lack of sediment at given locations, to obtain better special coverage of contaminant concentrations in sediment, or because an onshore remedy has made the sampling location inaccessible. During future monitoring for this project, samples will be collected at the MS locations where sediment samples were last collected. These locations are shown on Figures 5 to Figure 10. Some of the MSs at OU4 have both intertidal and subtidal areas. Sampling in the subtidal area will be conducted by boat. Sampling in the intertidal and shallow subtidal area will be conducted by walking to the sampling locations at low tide. Worksheet #18 lists MS sample locations and corresponding area types (i.e., subtidal or intertidal). The following tble lists frequency and parameters to be analyzed at each monitoring station.

AOC	Monitoring Station	Sampling Frequency	Parameters
Back Channel	MS-01	5-years	PAHs.
	MW-03 and MS-04	5-year	PAHs and copper.
Jamaica Cove	MS-05	2-years	Copper, lead, and nickel.
		5-years	PAHs and metals.
Clark Cove	MS-07	5-years	PAHs, 4,4-'DDT, dioxins/furans, PCBs, and metals.
	MS-08 and MS-09	2-years	PAHs, 4,4-'DDT, copper, lead, nickel, and dioxins/furans.
		5-years	PAHs, 4,4'-DDT, metals, PCBs, and dioxins/furans.
DRMO Storage Yard	MS-11	5-years	Copper, lead, and nickel.
Dry Docks	MS-12	2-years	Analyze additional sediment samples near AS12-SD12 for lead.
		5-years	PAHs and lead. Analyze additional sediment sample near AS12-SD12 for lead.

Sediment samples will be collected from the surface (0 to 10 cm below the sediment surface) of the sea floor using a grab sampler or equivalent sediment sampling device capable of providing adequate quantities of sediment that can be opened from the top. The sampling depth for surface sediment is consistent with previous sampling as part of the Interim Offshore Monitoring Program for OU4 as described in Section 11.3. A minimum of three grabs will be collected from each sample location and composited into one sample to be consistent with previous sampling methodology to provide a sample representative of sediment at the sampling location.

As each grab sample is retrieved, the water from the sampler will be slowly decanted (or suctioned off) in accordance with the SOP for Sediment Sampling presented in Appendix B. The decanted sediment

sample will be placed in a chemically cleaned stainless steel mixing bowl and mixed by hand to form a single composite sample from each sampling location. For sample locations that can be accessed by foot, the sample will be collected using a stainless steel spoon or similar device and treated as described above.

Surveying

Each sample location will be identified and mapped using a global positioning system (GPS) accurate to within 1 meter. The coordinate system used will be North American Datum (NAD) 1983 SPCS Maine West (feet). All locations/coordinates will be recorded in the field notebook. In addition, a known survey benchmark will be surveyed using the GPS equipment as a control point. Base datum will be identified and recorded. Location information will be converted to the State of Maine plane system for consistency in future mapping efforts. Permanent markers were placed at some of the intertidal locations. If these markers are present during subsequent sampling events, samples should be collected within 1 foot of the marker.

SAP Worksheet #18A -- Sampling Locations and Methods/SOP Requirements Table for the Round 11 Sampling Event
(UFP-QAPP Manual Section 3.1.1)

Sampling Location	Sampling Area Type	ID Number	Analytical Group	Matrix	Depth	Number of Samples	Sampling SOP Reference[1]
MS-01 Loc. 1	Subtidal	OU4-SD-M01-111B	PAHs	Surface Sediment	0 to 0.33 feet below sediment surface (4 inches)	26 sediment samples plus three field duplicates	CT-04, SA-6.1, SA-6.3, SA-7.1
MS-01 Loc. 2	Intertidal	OU4-SD-M01-211B					
MS-01 Loc. 3	Subtidal	OU4-SD-M01-311B					
MS-03 Loc. 1	Intertidal	OU4-SD-M03-111B	PAHs Copper				
MS-03 Loc. 2	Intertidial	OU4-SD-M03-211B					
MS-03 Loc. 3	Subtidal	OU4-SD-M03-311B					
MS-04 Loc. 1	Intertidal	OU4-SD-M04-111B					
MS-04 Loc. 2	Subtidal	OU4-SD-M04-211B					
MS-04 Loc. 3	Intertidal	OU4-SD-M04-310B					
MS-05 Loc. 1A	Intertidal	OU4-SD-M05-111B	PAHs Metals				
MS-05 Loc. 2A	Intertidal	OU4-SD-M05-211B					
MS-05 Loc. 3	Intertidal	OU4-SD-M05-311B					
MS-05 Loc. 4	Intertidal	OU4-SD-M05-411B					

Sampling Location	Sampling Area Type	ID Number	Analytical Group	Matrix	Depth	Number of Samples	Sampling SOP Reference[1]
MS-07 Loc. 1	Intertidal	OU4-SD-M07-111B	PAHs 4,4'-DDT, Metals Dioxins/furans PCB congeners	Surface Sediment	0 to 0.33 feet below sediment surface (4 inches)	See previous page	CT-04, SA-6.1, SA-6.3, SA-7.1
MS-07 Loc. 2	Subtidal	OU4-SD-M07-211B					
MS-07 Loc. 3	Subtidal	OU4-SD-M07-311B					
MS-08 Loc. 1	Intertidal	OU4-SD-M08-111B					
MS-08 Loc. 2	Subtidal	OU4-SD-M08-211B					
MS-08 Loc. 3	Intertidal	OU4-SD-M08-311B					
MS-09 Loc. 1A	Subtidal	OU4-SD-M09-111B					
MS-09 Loc. 2B	Subtidal	OU4-SD-M09-211B					
MS-09 Loc. 3	Subtidal	OU4-SD-M09-311B					
MS-11 Loc. 3	Intertidal	OU4-SD-M11-311B	Copper Lead Nickel				
MS-12 Loc. 1	Intertidal	OU4-SD-M12-111B	PAHs Lead				
MS-12 Loc. 2	Subtidal	OU4-SD-M12-211B					
MS-12 Loc. 3	Subtidal	OU4-SD-M12-311B					
MS-12 Loc. 4	Subtidal	OU4-SD-M12-411B	Lead				
MS-12 Loc. 5	Subtidal	OU4-SD-M12-511B					
MS-12 Loc. 6	Subtidal	OU4-SD-M12-611B					

1 SOP that describes the sample collection procedures.

SAP Worksheet #18B -- Sampling Locations and Methods/SOP Requirements Table for the Round 12 Sampling Event
(UFP-QAPP Manual Section 3.1.1)

Sampling Location	Sampling Area Type	ID Number	Analytical Group	Matrix	Depth	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
MS-05 Loc. 1A	Intertidal	OU4-SD-M05-113A	Copper Lead Nickel	Surface Sediment	0 to 0.33 feet below sediment surface (4 inches)	13 plus two duplicates	CT-04, SA-6.1, SA-6.3, SA-7.1
MS-05 Loc. 2A	Intertidal	OU4-SD-M05-213A					
MS-05 Loc. 3	Intertidal	OU4-SD-M05-313A					
MS-05 Loc. 4	Intertidal	OU4-SD-M05-413A					
MS-08 Loc. 1	Intertidal	OU4-SD-M08-113A	PAHs 4,4'-DDT Copper Lead Nickel Dioxins/Furans				
MS-08 Loc. 2	Subtidal	OU4-SD-M08-213A					
MS-08 Loc. 3	Intertidal	OU4-SD-M08-313A					
MS-09 Loc. 1A	Subtidal	OU4-SD-M09-113A					
MS-09 Loc. 2B	Subtidal	OU4-SD-M09-213A					
MS-09 Loc. 3	Subtidal	OU4-SD-M09-313A					
MS-12 Loc. 4	Subtidal	OU4-SD-M12-413A	Lead				
MS-12 Loc. 5	Subtidal	OU4-SD-M12-513A					
MS-12 Loc. 6	Subtidal	OU4-SD-M12-613A					

1 SOP that describes the sample collection procedures.

SAP Worksheet #18C -- Sampling Locations and Methods/SOP Requirements Table for the Round 13 Sampling Event
(UFP-QAPP Manual Section 3.1.1)

Sampling Location	Sampling Area Type	ID Number	Analytical Group	Matrix	Depth	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
MS-05 Loc. 1A	Intertidal	OU4-SD-M05-115A	Copper Lead Nickel	Surface Sediment	0 to 0.33 feet below sediment surface (4 inches)	13 plus two duplicates	CT-04, SA-6.1, SA-6.3, SA-7.1
MS-05 Loc. 2A	Intertidal	OU4-SD-M05-215A					
MS-05 Loc. 3	Intertidal	OU4-SD-M05-315A					
MS-05 Loc. 4	Intertidal	OU4-SD-M05-415A					
MS-08 Loc. 1	Intertidal	OU4-SD-M08-115A	PAHs 4,4'-DDT Copper Lead Nickel Dioxins/Furans				
MS-08 Loc. 2	Subtidal	OU4-SD-M08-215A					
MS-08 Loc. 3	Intertidal	OU4-SD-M08-315A					
MS-09 Loc. 1A	Subtidal	OU4-SD-M09-115A					
MS-09 Loc. 2B	Subtidal	OU4-SD-M09-215A					
MS-09 Loc. 3	Subtidal	OU4-SD-M09-315A					
MS-12 Loc. 4	Subtidal	OU4-SD-M12-415A	Lead				
MS-12 Loc. 5	Subtidal	OU4-SD-M12-515A					
MS-12 Loc. 6	Subtidal	OU4-SD-M12-615A					

1 SOP that describes the sample collection procedures.

SAP Worksheet No. 19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time [1] (preparation/ analysis)
Aqueous Field QC Samples	PAHs	SW-846 3510C/3520/8270C Selective Ion Monitoring (SIM), Katahdin CA-502/CA-213	Two 1-liter amber glass bottles	1,000 milliliters	Cool to 4 (±2) °C	7 days until extraction/40 days to analysis
	Pesticides	SW-846 3510C/3520/8081, Katahdin CA-515/ CA-302	Two 1-liter amber glass bottles	1,000 milliliters	Cool to 4 (±2) °C	7 days until extraction/40 days to analysis
	PCB Congeners	SW-846 3510C/3520/8082, Katahdin CA-515/ CA-334	Two 1-liter amber glass bottles	1,000 milliliters	Cool to 4 (±2) °C	7 days until extraction/40 days to analysis
	Metals	SW-846 3010A/6010C/6020A, Katahdin CA-604/ CA-608/CA-608 (metals), SW-846 7470B Katahdin CA-615 (mercury)	500-milliliter plastic bottle	50 milliliters/30 milliliters mercury	Nitric acid to a pH<2; Cool to 4 (±2) °C	180 days to analysis (28 days for mercury)
	Dioxins/Furans	SW-846 1613B and SW-846 3520/ SGS DC37.050809.28, DC38.042109.13, DC185.042109.11	Two 1-liter amber glass bottles	1,000 milliliters	Cool to 4 (±2) °C; If residual chlorine is present in aqueous samples, add 80 milligrams sodium thiosulfate per liter of water	1 year to extraction/45 days from extract to analysis

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time [1] (preparation/ analysis)
Sediment	PAHs	SW-846 3540C/8270C SIM, Katahdin CA-526/CA-213	4-ounce glass jars	30 grams	Cool to 4 (±2) °C	14 days to extraction/40 days to analysis
	Pesticides	SW-846 3540/8081A, Katahdin CA-524/ CA-302	4-ounce glass jars	30 grams	Cool to 4 (±2) °C	14 days until extraction/40 days to analysis
	PCB Congeners	SW-846 3540/8082, Katahdin CA-524/ CA-334	4-ounce glass jars	30 grams	Cool to 4 (±2)°C	14 days until extraction/40 days to analysis
	Metals	SW-846 3050B/6010B Katahdin CA-605/ CA-608 (metals), SW-846 7471B Katahdin Ca-611 (mercury)	4-ounce glass jars	1 to 2 grams / 0.3 gram mercury	Cool to 4 (±2) °C	180 days to analysis (28 days for mercury)
	Dioxins/Furans	SW-846 1613B and SW-846 3520/ SGS DC37.050809.28, DC38.042109.13, DC185.042109.11	Two 1-liter glass bottles	1,000 milliliters	<-10°C; If residual chlorine is present in aqueous samples, add 80 milligrams sodium thiosulfate per liter of water	One year to extraction/45 days from extract to analysis

1 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted

SAP Worksheet #20A -- Field Quality Control Sample Summary Table for the Round 11 Sampling Event

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs (1)	No. of Source Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Sediment	Select Metals	10	1	1	0	0	0	0	11
	Metals (Full scan)	13	2	1	0	0	0	0	15
	PAHs (Full scan)	25	3	2	0	0	0	0	28
	Dioxins/ Furans (Full scan)	9	1	1	0	0	0	0	10
	4,4'-DDT	9	1	1	0	0	0	0	10
	PCBs (Full scan)	9	1	1	0	0	0	0	10

1 Although MS/MSD samples are not typically considered field QC samples, they are included here because location determination will be established in the field by the FOL. The total number of samples to the lab column does not count MS/MSDs because MS/MSDs are additional volumes of samples planned to be collected and not separate stand-alone samples.

SAP Worksheet #20B -- Field Quality Control Sample Summary Table for the Round 12 Sampling Event

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs (1)	No. of Source Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Sediment	Select Metals	13	2	1	0	0	0	0	15
	PAHs (Full scan)	6	1	1	0	0	0	0	7
	Dioxins/ Furans (Full scan)	6	1	1	0	0	0	0	7
	4,4'-DDT	6	1	1	0	0	0	0	7

1 Although MS/MSD samples are not typically considered field QC samples, they are included here because location determination will be established in the field by the FOL. The total number of samples to the lab column does not count MS/MSDs because MS/MSDs are additional volumes of samples planned to be collected and not separate stand-alone samples.

SAP Worksheet #20C -- Field Quality Control Sample Summary Table for the Round 13 Sampling Event

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs (1)	No. of Source Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Sediment	Select Metals	13	2	1	0	0	0	0	15
	PAHs (Full scan)	6	1	1	0	0	0	0	7
	Dioxins/ Furans (Full scan)	6	1	1	0	0	0	0	7
	4,4'-DDT	6	1	1	0	0	0	0	7

1 Although MS/MSD samples are not typically considered field QC samples, they are included here because location determination will be established in the field by the FOL. The total number of samples to the lab column does not count MS/MSDs because MS/MSDs are additional volumes of samples planned to be collected and not separate stand-alone samples.

SAP Worksheet #21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature	Tt	NA	Y	Refer to Appendix C for field SOPs
CT-05	Database Records and Quality Assurance	Tt	NA	N	
SA-6.1	Non-Radiological Sample Handling	Tt	Sample bottleware, packaging materials, shipping materials	Y	
SA-6.3	Field Documentation	Tt	Field logbook, field sample forms, boring logs, soil sample log sheets	N	
SA-7.1	Decontamination of Field Equipment	Tt	Decontamination equipment, scrub brushes, phosphate-free detergent, deionized water	Y	
TBD	Standard Operating Procedure for Sediment Sampling	TBD	Boat, GPS, sediment sampling device, etc. (see SOP for complete equipment list)	TBD	

NA = Not applicable

TBD = To be determined

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

None

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1) (Y/N)
Katahdin CA-101	Equipment Maintenance, 08/09, Revision 8.	Definitve	Various	Various	Katahdin	N
Katahdin CA-213	Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified For Selected Ion Monitoring (SIM), 08/09, Revision 7.	Definitive	Aqueous field QC samples and Sediment/ PAHs	Gas Chromatography/ Mass Spectrometery (GC/MS)	Katahdin	N
Katahdin CA-302	Analysis of Pesticides By Gas Chromatography/Electron Capture Detector : SW-846 Method 8081, 08/09, revision 10.	Definitive	Aqueous, Sediment, Pesticides	GC ECD	Katahdin	N
Katahdin CA-334	Analysis of PCBs as Congeners by Gas Chromatography / Electron Capture Detector (: SW-846 Method 8082, revision 4, 08/09.	Definitive	Aqueous field QC samples and Sediment/ PCBs	GC/ECD	Katahdin	N
Katahdin CA-502	Preparation of Aqueous Samples For Extractable Semivolatile Analysis, 09/08, Revision 5.	Definitive	Aqueous field QC samples/ PAH Extraction	NA/Extraction	Katahdin	N
Katahdin CA-515	Preparation of Aqueous Samples for Pesticides/PCBs Analysis, 09/08, Revision 5.	Definitive	Aqueous field QC sample/Pesticide/ PCBs Extraction	NA/Extraction	Katahdin	N
Katahdin CA-524	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Pesticide/PCB Analysis, 08/09, Revision 5.	Definitive	Sediment Pesticide/PCBs Extraction	NA/Extraction	Katahdin	N
Katahdin CA-526	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis, 08/09, Revision 6.	Definitive	Sediment PAH/ Extraction	NA/Extraction	Katahdin	N
Katahdin CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP Analysis of Total or Dissolved Metals, 05/09, Revision 4.	Definitive	Aqueous field QC sample/Metals Digestion	NA /sample preparation	Katahdin	N
Katahdin CA-605	Acid Digestion of Solid Samples by U.S. EPA Method 3050 for Metals by ICP-AES and GFAA, 08/09, Revision 4.	Definitive	Sediment/ Metals Digestion	NA/sample preparation	Katahdin	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1) (Y/N)
Katahdin CA-608	Trace Metals Analysis by ICP-AES Using EPA Method 6010, 08/09, Revision 9.	Definitive	Sediment and Aqueous field QC sample/ Metals	Inductively coupled plasma - atomic emission spectroscopy (ICP-AES)	Katahdin	N
Katahdin CA-611	Digestion And Analysis Of Solid Samples For Mercury by U.S. EPA Method 7471, 08/09, Revision 9.	Definitive	Sediment/ Mercury	Mercury Analyzer	Katahdin	N
Katahdin CA-615	Digestion And Analysis Of Aqueous Samples For Mercury By U.S. EPA Method 7470,08/09, Revision 4.	Definitive	Aqueous field QC sample/ Mercury	Mercury Analyzer	Katahdin	N
Katahdin SD-902	Sample Receipt and Internal Control, 08/09, Revision 8.	Definitive	Various	NA	Katahdin	N
Katahdin SD-903	Sample Disposal, 05/09, Revision 4.	Definitive	Various	NA	Katahdin	N
SGS DC185.042 109.11	Method Manual of Standard Operating Procedures For the Determination of Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS) April 21, 2009, Revision 11	Definitive	Sediment and Aqueous QC samples/Dioxins and Furans	High Resolution Autospec Mass spectrometer/ Agilent HP6890	SGS North America Inc.	N
SGS DC37.0508 09.29	Sample Processing May 8, 2009, Revision 29	Definitive	Sediment and Aqueous QC samples/Dioxins and Furans	Continuous Liquid Liquid Extractors (CLLE)	SGS North America Inc.	N
SGS DC38.0421 09.13	Standard Operating Procedures for Analysis and Reporting (Method 1613) Issue date: 04/21/09 Revision: 13	Definitive	Sediment and Aqueous QC samples/Dioxins and Furans	High Resolution Autospec Mass Spectrometer/ Agilent HP6890	SGS North America Inc.	N

NA Not Applicable

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS PAHs SIM	Initial Calibration - A minimum six-point calibration performed for all analytes.	Instrument receipt, instrument change (new column, source cleaning, etc.), when continuing calibration verification (CCV) is out of criteria.	Average response factors (RFs) for System Performance Check Compounds (SPCCs) must be ≥0.010; Percent relative standard deviations (%RSDs) must be ≤30 for the calibration check compounds (CCCs); %RSD must be < 15% for all other compounds. If not met: Option 1) Linear least squares regression: r must be ≥ 0.995 Option 2) Non-linear regression: coefficient of determination (COD) r^2 must be ≥ 0.99 (six points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Katahdin SOP CA-213
	Initial Calibration Verification (ICV)	Once after each initial calibration.	Percent Recoveries (%Rs) of individual compounds must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenyl phosphine (DFTPP) tune.	Average RFs for SPCCs must be ≥0.010. Percent differences (%Ds) for all target compounds and surrogates must be ≤ 30%.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS PAHs SIM	Tune verification DFTPP	Every 12 hours.	Criteria listed in Section 7.4 current revision of SOP CA-213.	Retune and/or clean source.	Analyst, Department Manager	
GC/ECD Pesticides	Initial calibration - A six point calibration of individual pesticides with a mid-point calibration of Oxychlordane.	Upon instrument receipt, major instrument change, or when the Continuing Calibration does not meet criteria.	Calibration coefficient of determination must be ≥ 0.990.	Repeat Initial calibration. If single-point cal Oxychlordane is identified in analysis of sample, six point calibration run of identified compound with reanalysis of sample.	Analyst, Department Manager	Katahdin SOP CA-302
	ICV	Once after each initial calibration.	%Rs of individual compounds must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Once after each initial calibration and at the beginning and end of each run sequence and every 10 samples.	%Rs of individual compounds must be within 85-115%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/ECD PCBs	Initial calibration - A minimum six point calibration of all congeners.	Instrument receipt, major instrument change, when CCV does not meet criteria.	Calibration coefficient of determination must be ≥ 0.990.	Repeat initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	Katahdin SOP CA-334
	ICV	Once after each initial calibration.	%Rs of individual compounds must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
GC/ECD PCBs	CCV	Once after each initial calibration and at the beginning and end of each run sequence and every 10 samples.	%Rs of individual compounds must be within 85-115%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	Katahdin SOP CA-334

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP-AES Metals	Initial Calibration – one-point calibration.	At the beginning of each day or if QC is out of criteria.	One-point calibration per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	Katahdin SOP CA-608
	ICV (Second Source)	Before beginning a sample run.	%Rs of true values must be within 90-110%.	Do not use results for failing elements unless the ICV > 110% and the sample < the LOQ. Investigate and correct problem.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample sequence.	No analytes detected > Limit of Detection (LOD).	Correct the problem, then reprepare and reanalyze.	Analyst, Department Manager	
	CCV	At the beginning and end of each run sequence and every 10 samples.	%Rs of true values must be within 90-110%.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Department Manager	
	Low-Level Calibration Check Standard (if using one-point initial calibration)	At beginning and end of run.	%Rs must be within 80%-120% of the true value.	Do not use results for failing elements, unless Limit of Quantitation (LOQ) recovery > upper limit and sample result < LOQ.	Analyst, Department Manager	
	Interference Check Standards (ICS - ICSA and ICSB)	At the beginning of an analytical run.	ICSA recoveries must be less than the absolute value of the LOD, and ICSB recoveries must be within 80-120 %R of the true value.	Correct the problem, then reprepare and reanalyze all affected samples.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Mercury Analyzer	Initial Calibration	At instrument receipt, major instrument change, and at the start of each day.	Correlation coefficient must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	Katahdin SOPs CA-611 and CA-615
	ICV (Second Source)	Before beginning a sample run.	%Rs of true values must be within 90-110%.	Correct problem and repeat calibration.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample run.	No analytes detected > LOD.	Correct problem and repeat calibration.	Analyst, Department Manager	
	CCV	CCV-at beginning and end of each run sequence and every 10 samples.	%Rs of true values must be within 80-120%.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Department Manager	
Waters Autospec/HP 6890 HRGC/HRMS	Initial calibration - A minimum five-point calibration is required.	As needed to maintain acceptable CCV, after major maintenance, or at a minimum once per year.	20% RPD for native and 35% RPD for labeled species.	An acceptable initial calibration must be established and verified with an independent source (CCV) before sample reporting may begin.	Analyst, Department Manager	SGS DC185.042109.11
	CCV	At the beginning of sample analysis every 12 hours.	As listed, per analyte on Table 7 of the SOP referenced.	Acceptable CCV must be established before sample analysis may begin.	Analyst, Department Manager	
	Descriptor Defining Isomers	At the beginning of each 12-hour analytical sequence.	First and last eluters must be present within the switching times.	Perform a survey scan to identify the correct switching times.	Analyst, Department Manager	
	Mass Resolution	Before and after each 12-hour analytical sequence.	Mass resolution must be at 10,000 as estimated from a printout of a perfluorokerosene (PFK) peak.	Mass spec maintenance.	Analyst, Department Manager	

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person(1)	SOP Reference(2)
GC/MS	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.	PAHs	Ion source, injector liner, column, column flow	Prior to initial calibration and/or as necessary	Acceptable Calibration or Calibration Verification	Correct the problem and repeat Calibration or Calibration Verification	Analyst, Department Manager	Katahdin SOP CA-213
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Pesticides and PCBs	Injector liner, septa, column, column flow	Prior to initial calibration and/or as necessary	Acceptable Calibration or Calibration Verification	Correct the problem and repeat Calibration or Calibration Verification	Analyst, Department Manager	Katahdin SOPs CA-302 and CA-334

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person(1)	SOP Reference(2)
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.	Metals	Torch, nebulizer chamber, pump, pump tubing	Prior to initial calibration and as necessary	Acceptable Calibration or Calibration Verification	Correct the problem and repeat Calibration or Calibration Verification	Analyst, Department Manager	Katahdin SOP 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 initial calibration and as necessary	Acceptable Calibration or Calibration Verification	Correct the problem and repeat Calibration or Calibration Verification	Analyst, Department Manager	Katahdin SOPs CA-611 and CA-615

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person(1)	SOP Reference(2)
HR/GC	Check pressure and gas supply daily. Bake out column, change septa, liner, seal as needed, cut column as needed	Dioxins/ Furans	Liner, seal, septum, column	Prior to initial calibration or as necessary	See Section 7 of SOP.	Acceptable beginning CCV must be established before sample reporting may begin.	Analyst/ Supervisor	DC185.042109.11
HR/MS	Check for line pump fluid level and ballast. Change the PFK septum. Ensure 10,000 resolution. Clean source as necessary.	Dioxins/ Furans	Liner, seal, septum, column, foreline pump level and ballast, tune	Prior to initial calibration, and beginning of 12-hour analytical sequence	Static resolution must be 10,000 ppm at 5% peak height. See Section 7 of SOP.	Acceptable beginning CCV must be established before sample reporting may begin.	Analyst/ Supervisor	DC185.042109.11

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL or designee/Tt
Sample Packaging (Personnel/Organization): FOL or designee/Tt
Coordination of Shipment (Personnel/Organization): FOL or designee/Tt
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/Katahdin and SGS
Sample Custody and Storage (Personnel/Organization): Sample Custodians/Katahdin and SGS
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/Katahdin and SGS
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography Lab, Gas Chromatography/Mass Spectrometry Lab, Metals Lab/Katahdin and SGS
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/Katahdin and SGS
Number of Days from Analysis: 30 days from submittal of final report or 60 days from receipt, whichever is longer

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Tt CT-04 Sample Nomenclature, Tt SA-1.2 Surface Water and Sediment Sampling, Tt SA-1.3 Soil Sampling, Tt SA-6.1 Non-Radiological Sample Handling
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Katahdin SD-902-08 - Sample Receipt and Internal Control, Katahdin SD-903 – Sample Disposal, SGS MI3.122309.13 – SOP for the Login and Storage of Samples
Sample Identification Procedures: Katahdin SD-902-08 - Sample Receipt and Internal Control, SGS MI3.122309.13 – SOP for the Login and Storage of Samples
Chain-of-Custody Procedures: Katahdin SD-902-08 - Sample Receipt and Internal Control, SGS MI3.122309.13 – SOP for the Login and Storage of Samples

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Aqueous QC/ Sediment
Analytical Group	PAHs
Analytical Method/ SOP Reference	SW-846 8270C (SIM)/Katahdin SOP CA-213

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 is performed for each batch of up to 20 samples.	Contaminants in the method blank must be < ½ LOQ.	(1) Investigate source of contamination (2) Reprepare 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.
Surrogates	Three per sample: 2-Methylnaphthalene-d10 Fluorene-d10Pyrene-d10.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1)	(1) Check chromatogram for interference; if found, then flag data. (2) If not found, check instrument performance; if problem is found, then correct and reanalyze. (3) If still out, then re-extract and analyze sample. (4) If reanalysis is out, then flag data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1)	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS recoveries are high but the sample results are <LOQ, then narrate otherwise re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	PAHs
Analytical Method/ SOP Reference	SW-846 8270C (SIM)/Katahdin SOP CA-213

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)
Matrix Spike MS/MSD	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1) The relative percent difference (RPD) between MS and MSD should be ≤ 30%.	Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Five per sample – 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12	Retention times for ISs must be + 30 seconds and the responses within -50% to +100% of the midpoint initial calibration standard for each IS.	Reanalyze affected samples	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	Pesticides
Analytical Method/ SOP Reference	SW-846 8081A/ Katahdin SOP CA-302

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 is performed for each batch of up to 20 samples.	Contaminants in the method blank must be < ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC i.e., If the blank results are above the LOQ, then report the sample results tj=hat are <LOQ or > 10X the blank concentration. Otherwise, reprepare a blank and samples >LOQ and <10X LOQ.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	Pesticides
Analytical Method/ SOP Reference	SW-846 8081A/ Katahdin SOP CA-302

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	Two per sample. Tetrachloro-m-xylenes Decachlorobiphenyl	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1)	No corrective action will be taken when one surrogate is within criteria. If surrogates %Rs exceed upper limit and sample is <LOQ no corrective action is taken. If surrogates are below the lower limit, then the affected samples are re-extracted and reanalyzed.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1)	If an MS/MSD was performed and acceptable, then narrate. If an LCS/Laboratory Control Sample Duplicate (LCSD) was performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	Pesticides
Analytical Method/ SOP Reference	SW-846 8081A/ Katahdin SOP CA-302

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)
MS/MSD	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1) The RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC. If the LCS results are acceptable, then narrate If both the LCS and MS/MSD are unacceptable, then reprepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/Sediment
Analytical Group	PCB - Congeners
Analytical Method/ SOP Reference	SW-846 8082/Katahdin SOP CA-334

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 is performed for each batch of up to 20 samples.	Contaminants in the method blank must be < ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC i.e., If the blank results are above the LOQ, then report the sample results that are <LOQ or > 10X the blank concentration. Otherwise, reprepare a blank and samples >LOQ and <10X LOQ.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/Sediment
Analytical Group	PCB - Congeners
Analytical Method/ SOP Reference	SW-846 8082/Katahdin SOP CA-334

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	Three per sample - Tetrachloro-m-xylene, PCB-103 and PCB-192	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1)	No corrective action will be taken when one surrogate is within criteria. If surrogates %Rs exceed upper limit and sample is <LOQ no corrective action is taken. If surrogates below the lower limit, then the affected samples are re-extracted and reanalyzed.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1)	If an MS/MSD was performed and acceptable, then narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/Sediment
Analytical Group	PCB - Congeners
Analytical Method/ SOP Reference	SW-846 8082/Katahdin SOP CA-334

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)
MS/MSD	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory statistically derived control limits. Current limits are provided in the corresponding SOP in Appendix C). (1) The RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC. If the LCS results are acceptable, then narrate If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	Metals
Analytical Method/ SOP Reference	SW-846 3010A, 6010C/7470A/ Katahdin SOP CA-604, CA-608, CA615

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 is performed for each digestion batch of up to 20 samples.	Contaminants in the method blank must be < ½ LOQ.	If blank value > LOQ, then report sample results. If < LOQ or > 10 x the blank value, then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples.	Water: %R must be within 80 to 120% of the true value. Soil: %R must be within vendor-supplied limits.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/Duplicate	One per digestion batch of 20 or fewer samples.	Recovery should be within 75-125% of the true value if sample < 4x spike added. The RPD should be within ≤20%.	Flag results for affected analytes for all associated samples with "N."	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	One per preparation batch of 20 or fewer samples of similar matrix.	If original sample result is at least 50x the instrument detection limit, five-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E."	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	Dioxins and Furans
Analytical Method/ SOP Reference	EPA 1613B DC185.042109.11

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, run after calibration standards and before samples.	PCDDF< SOP reporting limit or <10% of level in sample.	Affected samples must be re-extracted.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as Method/SOP QC Acceptance Limits.
Ongoing precision recovery standard (OPR)	One per extraction batch	See Table 9 of SOP	Re-extract batch if low and detected. Evaluate data quality for high and non-detected	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
Ongoing precision recovery standard duplicate (OPRD)	One per extraction batch.	See Table 9, ≤ 20%RSD.	If needed and fails, re-extract batch.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy and Precision/outside method limits.	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous QC/ Sediment
Analytical Group	Dioxins and Furans
Analytical Method/ SOP Reference	EPA 1613B DC185.042109.11

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)
MS/MSD	One pair per method, per matrix, per extraction technique, per 30 days, per 20 samples.	%R should be within 75 to 125%, RPD must be less than 20%.	None.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy and Precision/Matrix affect.	Same as Method/SOP QC Acceptance Limits.
Lab Sample Duplicate	At client request	%D must be <20%.	Flag failures.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Matrix Affect	Same as Method/SOP QC Acceptance Limits.
Internal Standards	Every field sample and every standard.	%R for each internal standard in the original sample (prior to dilutions) must be within 40-135%.	Correct the problem, then reprepare and reanalyze the samples with failed internal standard.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Matrix Affect	Same as Method/SOP QC Acceptance Limits.

1 Limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document / Record	Where Maintained
Sample Collection Documents and Records Field logbook (and sampling notes) Field forms (e.g., boring logs, sample log sheets, drilling logs, etc.) Chain-of-custody records Equipment calibration logs Photographs Field Task ModificationRequest Forms Field Sampling SOPs	Tt Project File. Field forms, chain-of-custody records, field task modification request forms, and photographs will also be included in the field investigation data package.
Laboratory Documents and Records Sample receipt/login forms, custody, and tracking records Sample storage records Sample preparation logs Standard traceability logs Equipment calibration logs Sample analysis run logs Equipment maintenance, testing, and inspection logs Corrective action forms Reported field sample results Reported results for standards, quality control checks, and quality control samples Data completeness checklists Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data Electronic Data Deliverables	Tt Project File, long-term data package storage at third-party professional document storage firm. The EDDs and corresponding databases will be formatted in both Navy and MEDEP formats.
Data Assessment Documents and Records Field Sampling Audit Checklist (if an audit is conducted) Analytical Audit Checklist (if an audit is conducted) Data Validation Memoranda	Tt Project File. Data validation reports will also be included in the field investigation data package.

Document / Record	Where Maintained
Other Documents HASP All versions of SAP Field investigation data package All versions of project reports (e.g., RI, FS)	Tt Project File

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Sediment and aqueous field QC	PAHs	See Worksheet #18	SW-846 8270C SIM	21 calendar days	Kate Zaleski Project Manager Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 (207) 874-2400 Ext. 17 kzaleski@katahdinlab.com	Not applicable
	Pesticides	See Worksheet #18	SW-846 8081A	21 calendar days		Not applicable
	PCB Congeners	See Worksheet #18	SW-846 8082	21 calendar days		Not applicable
	Metals	See Worksheet #18	SW-846 6010C, 6020A, and 7470A, 7471A	21 calendar days		Not applicable
Sediment and aqueous field QC	Dioxin/Furans	See Worksheet #18	EPA 1613B	21 calendar days		Not applicable

SAP Worksheet #31 – Planned Projects Assessment Table
(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory System Audit(1)	Every 2 years	External	DoD ELAP	DoD ELAP accrediting body	Laboratory QAM or Laboratory Manager	Laboratory QAM or Laboratory Manager	Laboratory QAM or Laboratory Manager

1 Katahdin and SGS are DoD Environmental Laboratory Accreditation Program (DoD ELAP) accredited. The DoD ELAP accreditation letters are included in Appendix C.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Time Frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Time Frame for Response
Laboratory System Audit	Written audit report	Deb Nadeau Laboratory Director, Katahdin Leslie Dimond Laboratory QAM, Katahdin	Specified by DoD ELAP accrediting body	Letter	DoD ELAP Accrediting body	Specified by DoD ELAP accrediting body

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per SDG	Once validation is complete	Tetra Tech DVM or designee	Tetra Tech PM, project file
Major Analysis Problem Identification (internal memo)	When persistent analysis problems are detected	Immediately	Tetra Tech CLEAN QAM	Tetra Tech PM, CLEAN QAM, Program Manager, and project file
Project Monthly Progress Report	Monthly for duration of project	Monthly	Tetra Tech PM	Department of Navy (DON), project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately	Laboratory PM	Tetra Tech PM, project file

SAP Worksheet #34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler, and copies will be retained for the project file and Tetra Tech PM. See SOP SA-6.3.	Internal	Tt sampler and FOL
SAP Sample Tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	Tt FOL or designee
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	Tt FOL or designee
Sample Coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	Internal	Tt FOL or designee
Field SOPs/Field Logs/SampleCcollection	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	Tt FOL or designee
Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Katahdin QAM/SGS QAM
Documentation of Method QC Results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the laboratory will contact Tetra tech for guidance prior to report preparation.	Internal	Katahdin QAM/SGS QAM
Field QC Samples	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	Tt FOL or designee

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The laboratory sample custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech data validator will check that the Chain-of-Custody Form was signed/dated by the Tetra Tech FOL or designee relinquishing the samples and also by t Tetra Tech he laboratory sample custodian receiving the samples for analyses.	Internal/ External	Laboratory sample custodian/ Tt data validator
Analytical Data Package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QAM will sign the case narrative for each data package.	Internal	Katahdin QAM/SGS QAM
Analytical Data Package	The data package will be verified for completeness by Tetra Tech data validator. Missing information will be requested Tetra Tech from the laboratory.	External	Tt Data Validator
EDDs	The electronic data will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the method detection limit and the reporting limit will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Tt Data Validator

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table
(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples were maintained from collection to analysis and that custody records are complete and any deviations are recorded.	Tt Project Chemist or Data Validators
IIa	Holding Times	Review that the samples were shipped and stored at the required temperature and that sample pH for chemically preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Tt Project Chemist or Data Validators
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Tt Project Chemist or Data Validators
IIa/IIb	Laboratory Duplicate Analyses for Precision	Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSDs. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet #12.	Tt Project Chemist or Data Validators
IIa/IIb	Sample Results for Representativeness	Check that the laboratory recorded the temperature at sample receipt and the pH of chemically preserved samples to ensure sample integrity from sample collection to analysis.	Tt Project Chemist or Data Validators
IIa/IIb	PALs	Discuss the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminants, on the other target compounds reported as not detected. Document this usability issue and inform the PM.	Tt Project Chemist or Data Validators

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa/IIb	Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reasons for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Tt Project Chemist or Data Validators
IIa, IIb	SAP QC Sample Documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the laboratory shall have contacted the Tt PM.	Tt PM or designee
IIa, IIb	Documentation of Analytical Reports for Completeness	Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	Tt Project Chemist or Data Validators
IIa/IIb	PALs	Review and add PALs to the laboratory EDDs. Flag samples and notify Tt PM of samples that exceed PSLs as listed on Worksheet #15.	Tt PM or designee
IIb	QLs for Sensitivity	Ensure that the QLs listed in Worksheet #15 were achieved.	Tt Project Chemist or Data Validators
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results.	Tt Project Chemist or Data Validators

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Sediment and aqueous field QC	PAHs, Pesticides, and PCBs	SW-846 8270C SIM, 8081A, and 8082 method-specific criteria and those criteria listed in Worksheet #s 12, 15, 24, and 28 will be used. If not included in Worksheet #s12, 15, 24 or 28, default to U.S. EPA Region 1 Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996, and Part III, February 2004, then default to the logic outlined in U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008, October 1999 will be used to apply qualifiers to data.	Tt Data Validation Specialist

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Sediment and aqueous field QC	Metals	SW-846 6010C, 6020A, 7470A, 7471A, and the specific criteria and those listed in Worksheet #s 12, 15, 24, and #8 will be used. If not included in Worksheet #s 12, 15, 24, and 28, default to U.S. EPA Region 1 Data Validation Functional Guidelines for Evaluating Inorganic Analyses, Part VI, November 2008, then default to the logic outlined in U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review EPA 540-R-04-004, October 2004 will be used to apply qualifiers to data.	Tt Data Validation Specialist
IIa and IIb	Sediment and aqueous field QC	Dioxins and Furans	SW-846 8290 method specific criteria and those criteria listed in Worksheet #s 12, 15, 24, and 28 will be used. If not included in Worksheet #s 12, 15, 24 or 28, the logic outlined in U.S. EPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation, September 2005, will be used to apply qualifiers to data.	Tt Data Validation Specialist

SAP Worksheet #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

- If verification and validation requirements are not satisfied, the data will be qualified. Data will be classified as estimated (J, UJ) for minor QC deviations that not likely to compromise the data usability significantly, or the data will be classified as rejected (R, UR) for major QC deviations that are likely to have a significant effect on data usability. The impact of rejected data will be evaluated and the need for corrective actions such as resampling will be evaluated. This determination will be made by the Tt project team and will be based on an evaluation of whether project objectives can be achieved without resampling. The use of estimated data, if any, will be discussed in the project report and will be subject to project team review.
- All of the Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity (PARCCS) parameters will be evaluated during a Data Quality Review (DQR) to determine the overall usability of the data compared to project criteria outlined in this SAP. Precision, accuracy, sensitivity, and completeness will be evaluated based on numerical data; evaluations of representativeness and comparability will be qualitative.
- Deviations from the SAP will be reviewed to assess whether the deviations are significant enough to compromise the attainment of project objectives. If project objectives are compromised, as determined by the project team, additional planning meetings may be required to revise DQOs. This will be a team decision based on the numerous variables and interactions of variables that cannot be predicted before collecting the data.
- Analytical data will be checked to ensure that they are accurately transferred to the electronic project database. These inspections involve a series of cross-checks and inspections that are performed manually or with database software. The database is Microsoft SQL 2000 Version 8 and upgrades thereto.
- The data will be reviewed to identify potential outliers. This may involve statistical testing at the discretion of the Tt PM with input from the project team. If a review of the field and laboratory documentation indicates that the suspected outliers have an assignable cause for being out of line with other results, the outliers will be removed from the data sets, and any reprocessing required to compute aggregate statistics such as maximum and minimum values will be performed as necessary.
- Project required quantitation limits listed in Worksheet #15 will be evaluated.
- For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Laboratory duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and field duplicate will be used to represent the concentration at particular sample locations.
- Calculation of the mean and the lowest and highest detected concentrations for each parameter will be presented in summary tables in the OU4 Offshore Interim Monitoring Report(s). These calculations may be performed in Microsoft SQL 2000 Version 8 and subsequent upgrades, Microsoft Excel 2003 and subsequent upgrades, or Statsoft Statistica Version 6 or 7 and subsequent upgrades, depending on the nature of the computation. These are industry-standard software packages used without modification except to customize them by preparing “canned” data management routines that have been used successfully numerous times on various data sets and do not require any additional validation.
- Discussions will be held with the Navy RPM and regulatory RPMs on data issues that affect the attainment of project objectives to determine whether the data are usable for project decisions.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

- If a significant deviation occurs between laboratory and field precision, the cause will be investigated. The expectation is that laboratory precision values will be no greater than RPDs for field duplicates of the same matrix. If deviations occur, they will be described in the data package and interpreted for their impact on decision making in the OU4 Offshore Monitoring Report. Precision computations will be based on calculation of RPD. RPD = (Difference of two results)/(average of two results)*100 percent.
- If significant biases are detected (represented by low or high matrix spike, LCS, or surrogate recoveries), this will be noted and evaluated for impact on decision making. The tendency will be to emphasize review of low biases more than high biases unless biased results are near action levels. Low biases will be emphasized more because they are likely to represent an inability to detect analytes that are present at the site and, on a percentage basis, generally represent a greater proportion of the magnitude of individual values. Biases greater than ±30 percent from the ideal of 0 percent will generally be reviewed. Whether corrective action is necessary will be determined by the Tt Project Chemist. This judgment will be based on numerous variables such as the number of deviant bias indicators in a batch of samples and in related sample batches, the degree of bias, the number of precision indicators in a batch of samples and related samples, the type of bias indicator that is deviant (e.g., laboratory control samples or matrix spikes). Bias computations will be made by computing the difference between observed and expected results and dividing by the expected result then multiplying by 100 percent.

Identify the personnel responsible for performing the usability assessment:

- The Tt PM and Project Chemist or designee with contributions from other team members, as necessary, will perform these evaluations. Ultimately, NAVFAC will determine whether project objectives have been attained. Regulators will provide oversight of this process and will agree with the results if they find the results satisfactory.

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 project DQR report will identify and describe the data usability limitations and suggest resampling if necessary to fill any identified data gaps. A description of the PARCCS parameter evaluations will be included in the DQR report. This may include a short summary with supporting documentation or a more detailed summary, depending on the number and significance of deficiencies or SAP deviations.
- The DQR report and discussion of SAP deviations will be included in the investigation data package. Impacts to the project based on the DQR and SAP deviations will be evaluated as part of the OU4 Offshore Monitoring Report. The investigation data package and OU4 Offshore Monitoring Report will be provided to the Navy and regulators for review and comment.

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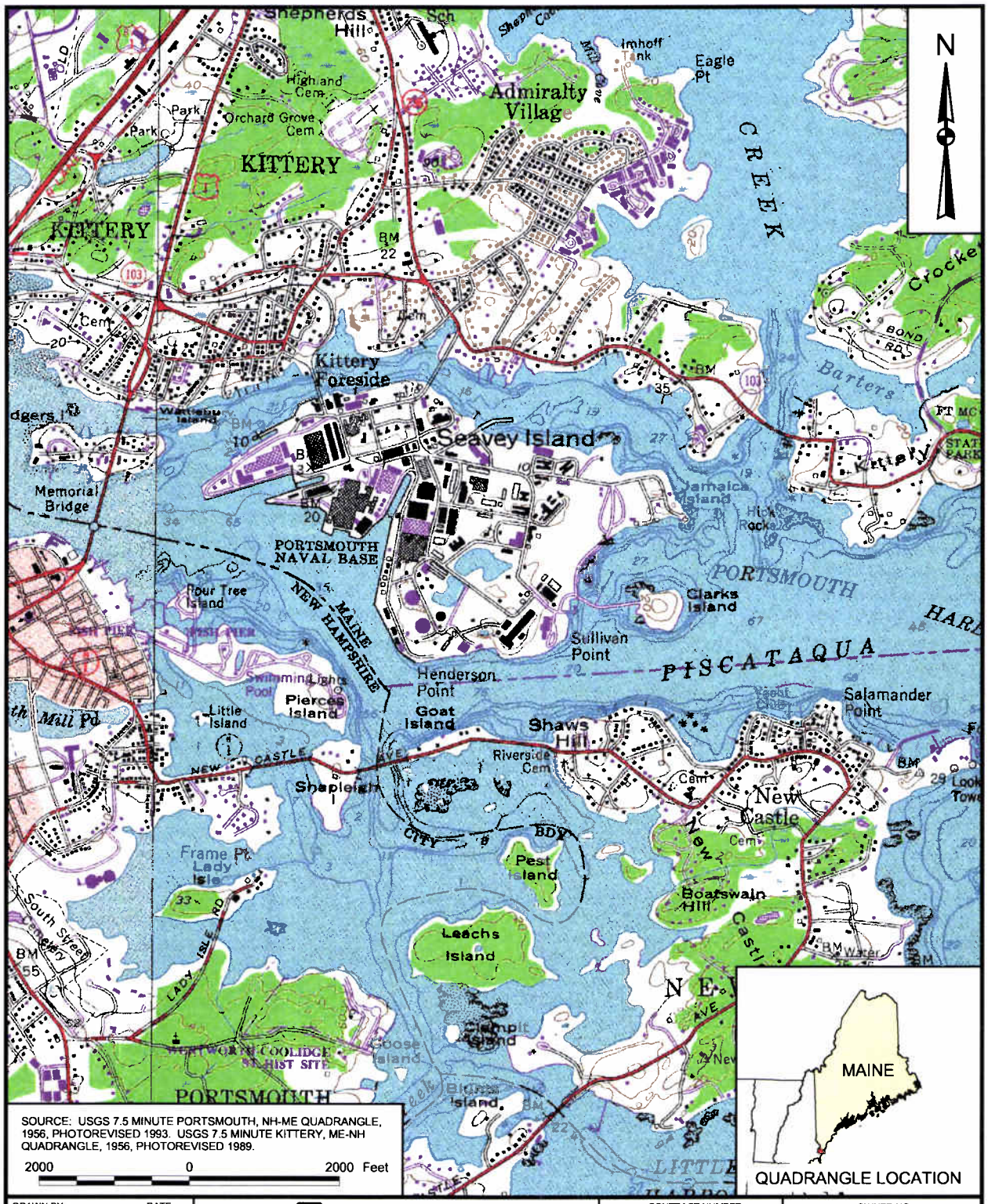
FIGURES



SOURCE: USGS 7.5 MINUTE PORTSMOUTH, NH-ME QUADRANGLE, 1956, PHOTOREVISED 1993. USGS 7.5 MINUTE KITTERY, ME-NH QUADRANGLE, 1956, PHOTOREVISED 1989.

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DRAWN BY J. LAMEY — DATE 12/29/03	Tetra Tech NUS, Inc.	CONTRACT NUMBER 0057	OWNER NO. CTO 123
CHECKED BY M. KRAUS — DATE 04/12/10	PORTSMOUTH NAVAL SHIPYARD VICINITY MAP PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY — DATE —	
COST/SCHEDULE-AREA		APPROVED BY — DATE —	
SCALE AS NOTED		DRAWING NO. FIGURE 1	REV 0

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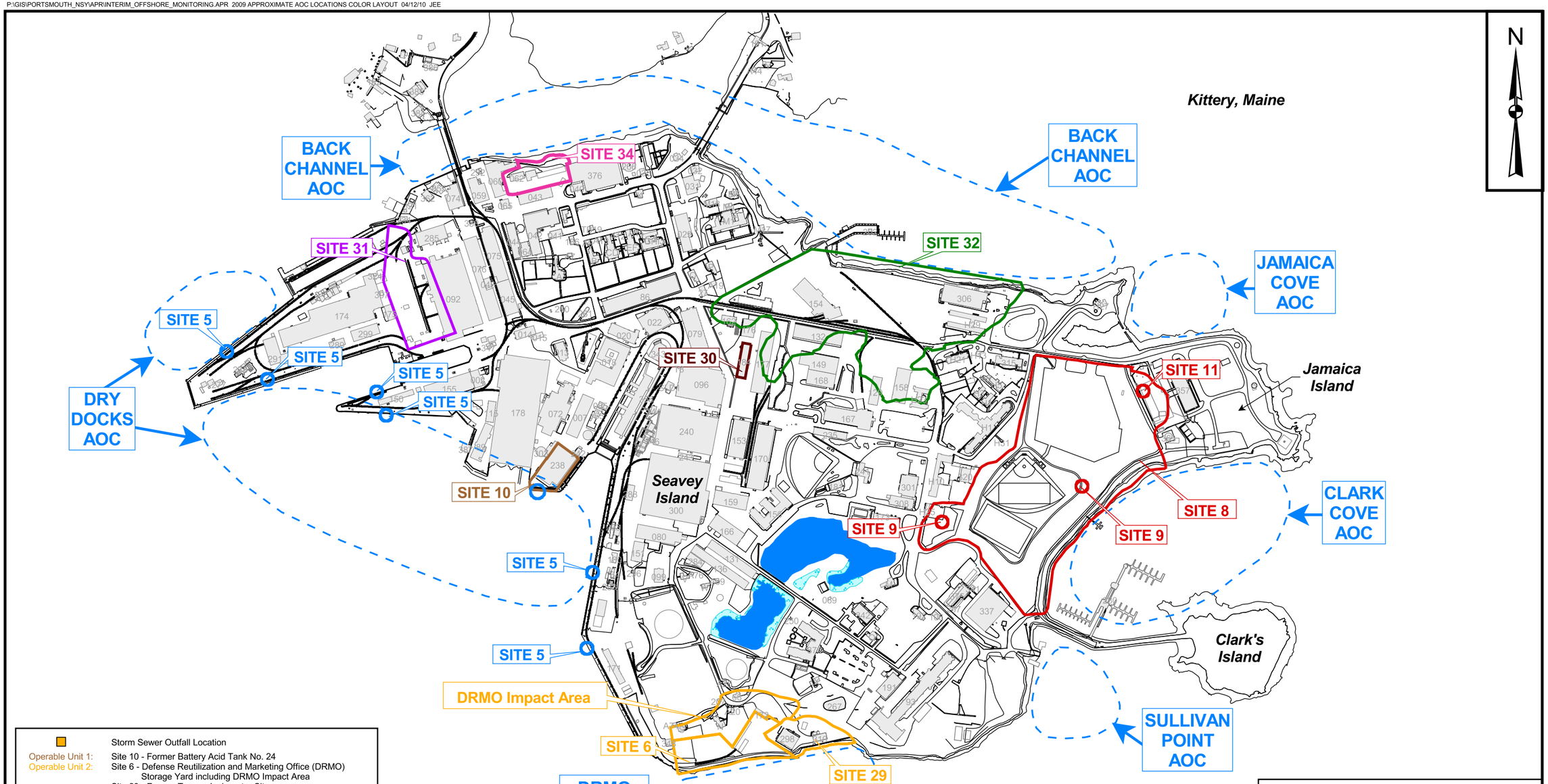
P:\GIS\PORTSMOUTH_NSY\APR\INTERIM_OFFSHORE_MONITORING.APR 2009 APPROXIMATE AOC LOCATIONS COLOR LAYOUT 04/12/10 JEE

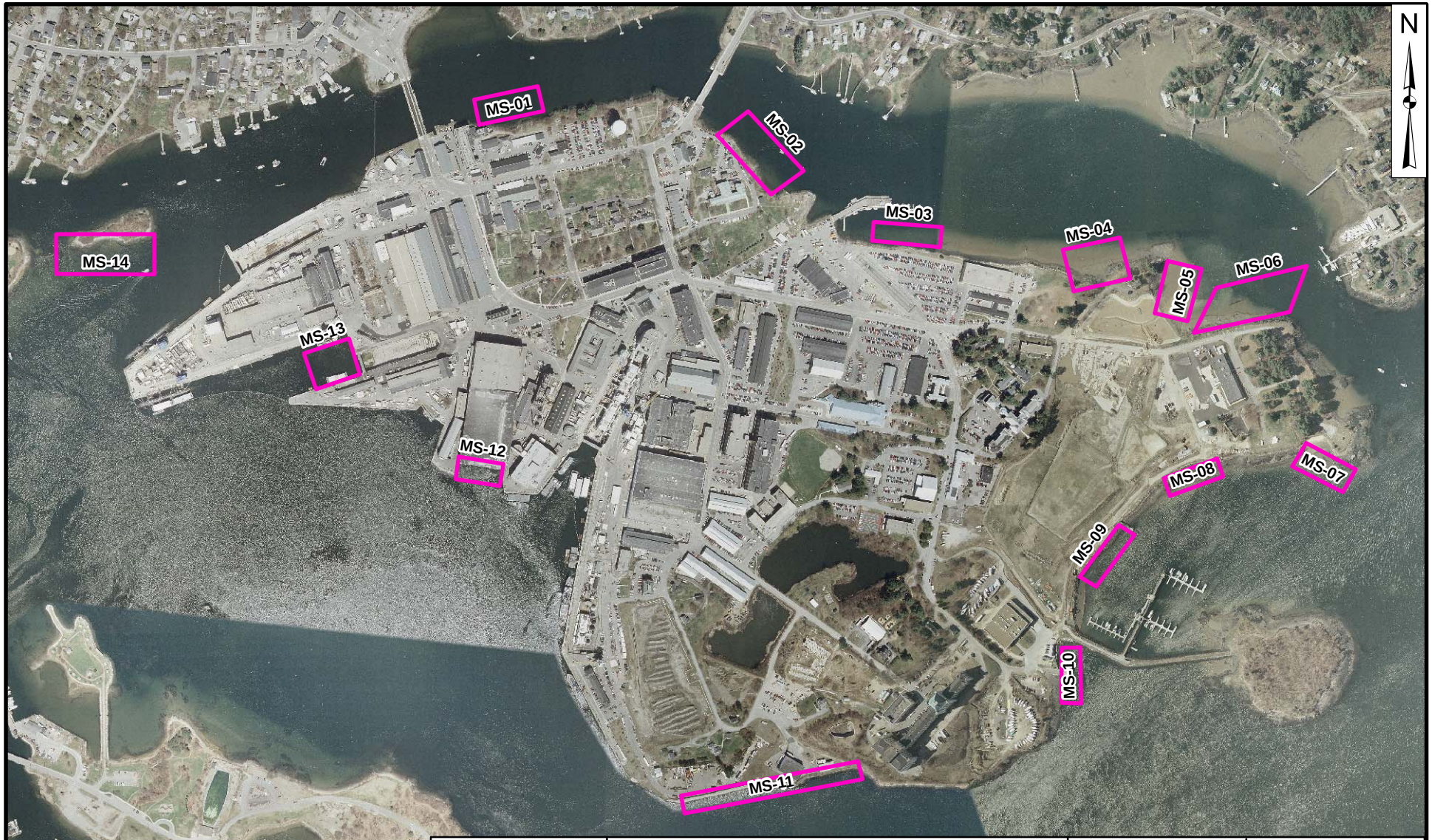


	Storm Sewer Outfall Location
Operable Unit 1:	Site 10 - Former Battery Acid Tank No. 24
Operable Unit 2:	Site 6 - Defense Reutilization and Marketing Office (DRMO) Storage Yard including DRMO Impact Area
	Site 29 - Former Teepee Incinerator Site
Operable Unit 3:	Site 8 - Jamaica Island Landfill (JILF)
	Site 9 - Former Mercury Burial Sites
	Site 11 - Former Waste Oil Tanks Nos. 6 & 7
Operable Unit 4:	Site 5 - Former Industrial Waste Outfalls Offshore Areas potentially impacted by onshore IRP sites (Six AOCs have been delineated)
Operable Unit 7:	Site 32 - Topeka Pier Site
Operable Unit 8:	Site 31 - West Timber Basin
Operable Unit 9:	Site 34 - Former Oil Gasification Plant, Building 62
Site Screening Area:	Site 30 - Galvanizing Plant, Building 184

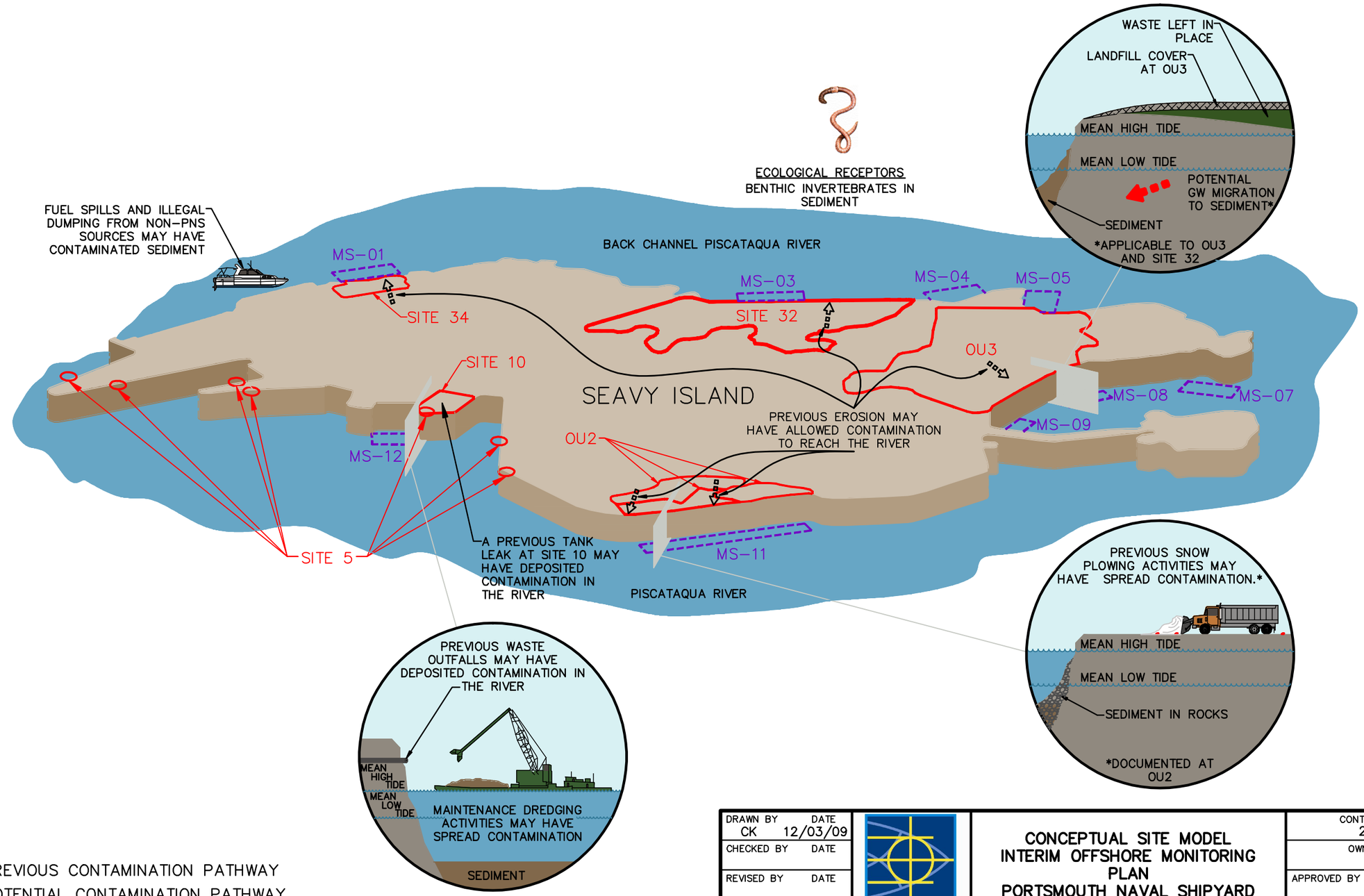
Note: 2008 PNS Base Map

DRAWN BY S. PAXTON	DATE 07/20/07	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO 123 / OWNER NUMBER —
CHECKED BY M. KRAUS	DATE 04/12/10	FACILITY SITE MAP PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY — / DATE —
COST/SCHEDULE-AREA			APPROVED BY — / DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 2 / REV 0

P:\GIS\PORTSMOUTH_NSY\MAPDOCS\MXD\MONITORING_SITE_MAPS\OVERVIEW_MONITORING_STATIONS.MXD 04/12/10 JEE



DRAWN BY T. WHEATON	DATE 09/15/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO 123	OWNER NUMBER ___
CHECKED BY M. KRAUS	DATE 04/12/10	LOCATIONS OF MONITORING STATIONS OPERABLE UNIT 4 FEASIBILITY STUDY REPORT PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___	DATE ___
COST/SCHEDULE-AREA			APPROVED BY ___	DATE ___
SCALE AS NOTED			DRAWING NO. FIGURE 3	REV 0



LEGEND

PREVIOUS CONTAMINATION PATHWAY

POTENTIAL CONTAMINATION PATHWAY

DRAWN BY CK DATE 12/03/09	NAVFAC	CONCEPTUAL SITE MODEL INTERIM OFFSHORE MONITORING PLAN PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	CONTRACT NO. 2225
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 4 / REV. 0

R:\2225\2225C002.dwg PIT CARLY.KRAMER 4/9/2010 1:27:28 PM

P:\GIS\PORTSMOUTH_NSY\MXD\MONITORING_SITE_MAPS\OU4_MS1_SD_SAMPLING.MXD 12/11/09 KM

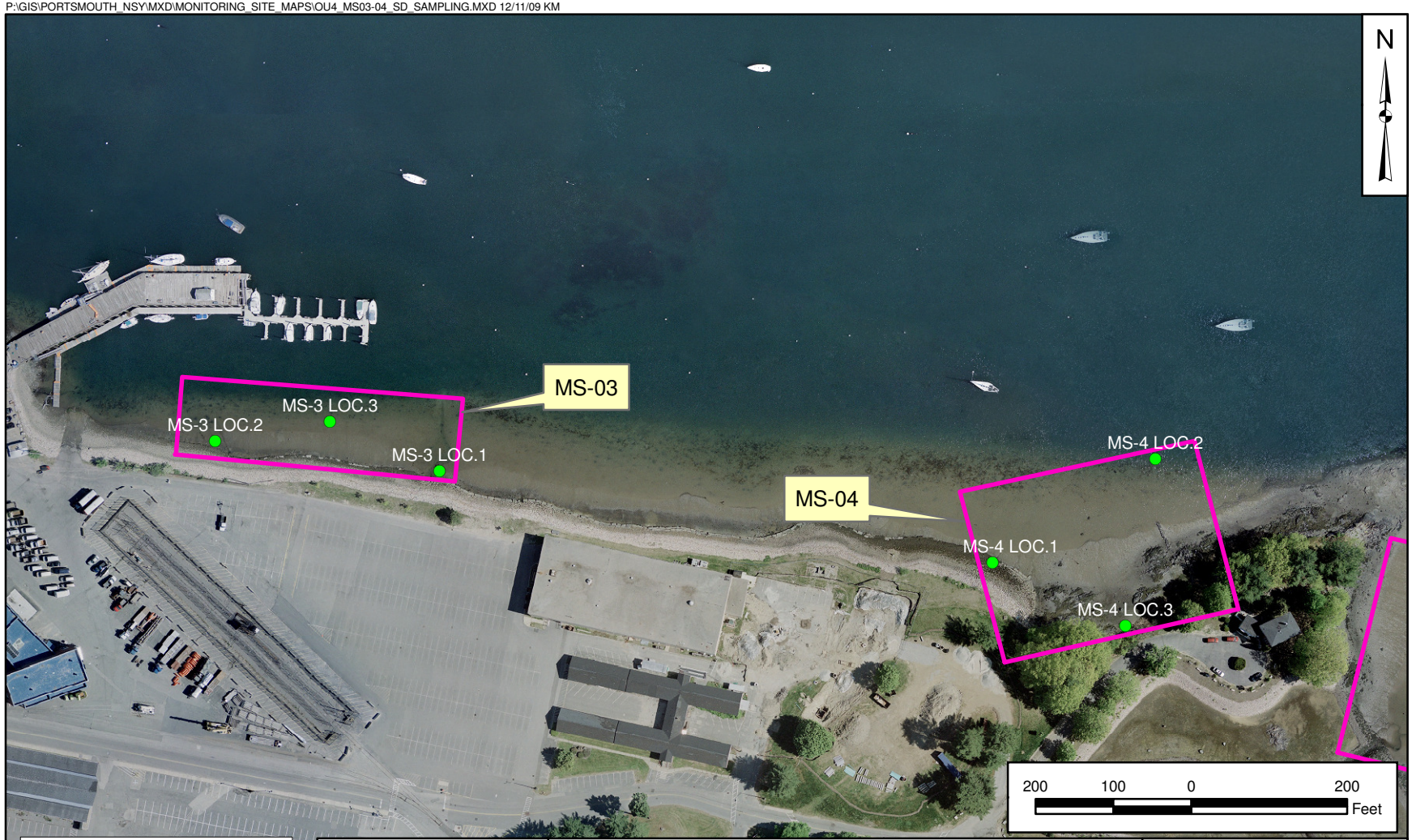
Legend

- Sediment Sampling Location
- Monitoring Station Boundary

NOTE:
2008 Aerial photograph recieved from Navy

DRAWN BY K. MOORE	DATE 12/11/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO WE29
CHECKED BY M. GRAY	DATE 12/11/09	SEDIMENT SAMPLING LOCATIONS AT MS-01 PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 5 / REV 0

P:\GIS\PORTSMOUTH_NSY\MXD\MONITORING_SITE_MAPS\OU4_MS03-04_SD_SAMPLING.MXD 12/11/09 KM



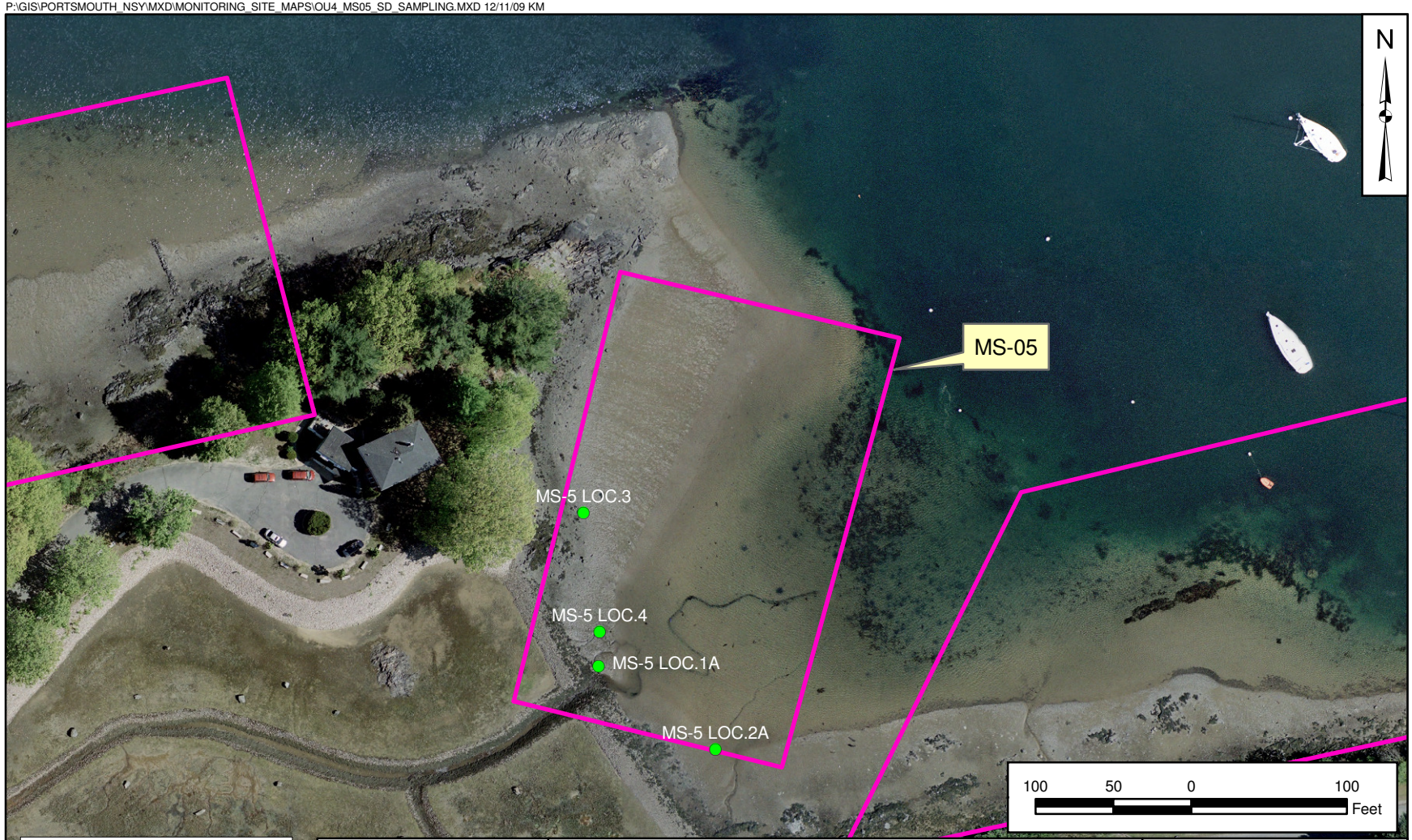
Legend

- Sediment Sampling Location
- Monitoring Station Boundary

NOTE:
2008 Aerial photograph recieved from Navy

DRAWN BY K. MOORE	DATE 12/11/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO WE29
CHECKED BY M. GRAY	DATE 12/11/09	SEDIMENT SAMPLING LOCATIONS AT MS-03 AND MS-04 PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 6 / REV 0

P:\GIS\PORTSMOUTH_NSY\MXD\MONITORING_SITE_MAPS\OU4_MS05_SD_SAMPLING.MXD 12/11/09 KM



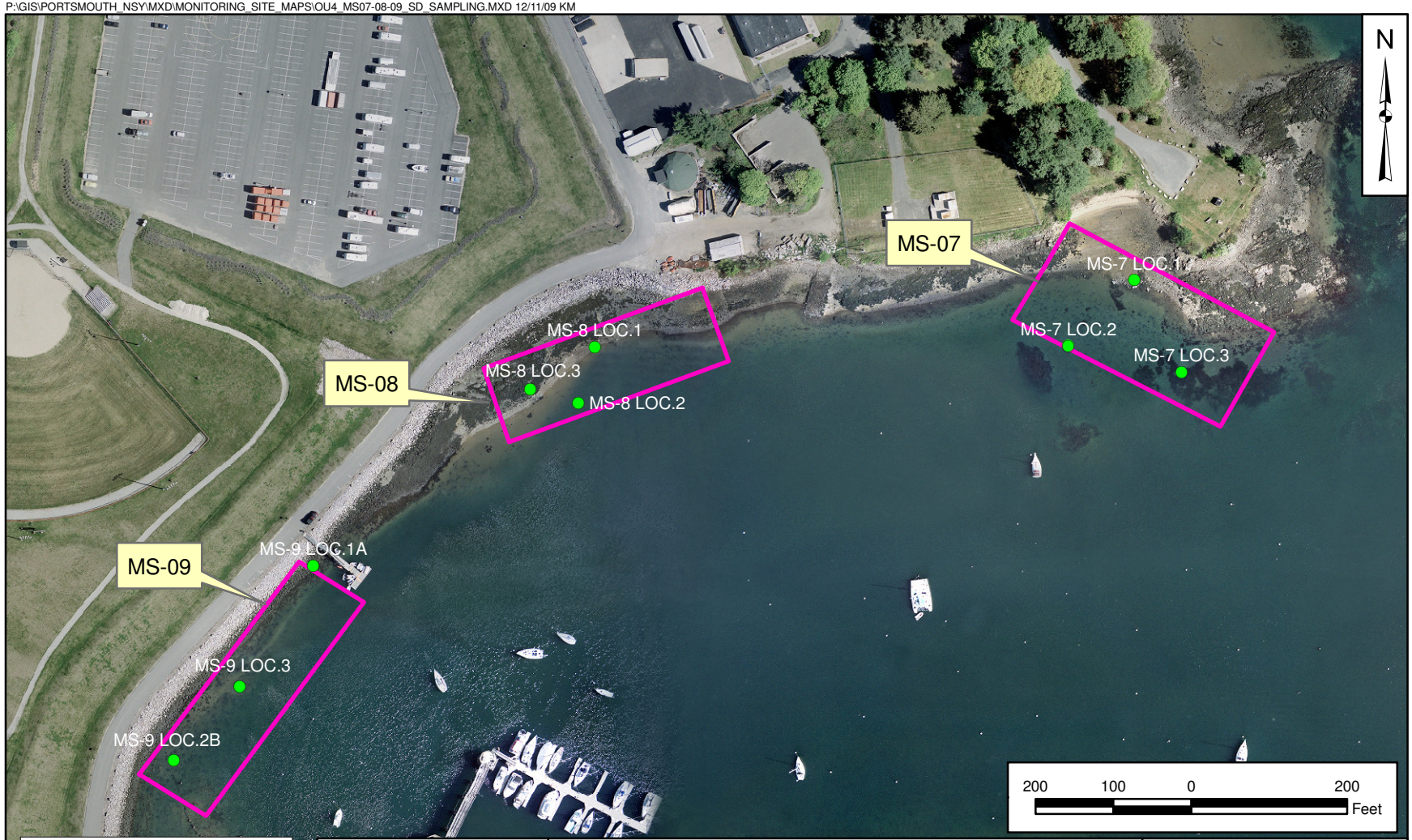
Legend

- Sediment Sampling Location
- Monitoring Station Boundary

NOTE:
2008 Aerial photograph recieved from Navy

DRAWN BY K. MOORE	DATE 12/11/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO WE29
CHECKED BY M. GRAY	DATE 12/11/09	SEDIMENT SAMPLING LOCATIONS AT MS-05 PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 7 / REV 0

P:\GIS\PORTSMOUTH_NSY\MXD\MONITORING_SITE_MAPS\OU4_MS07-08-09_SD_SAMPLING.MXD 12/11/09 KM



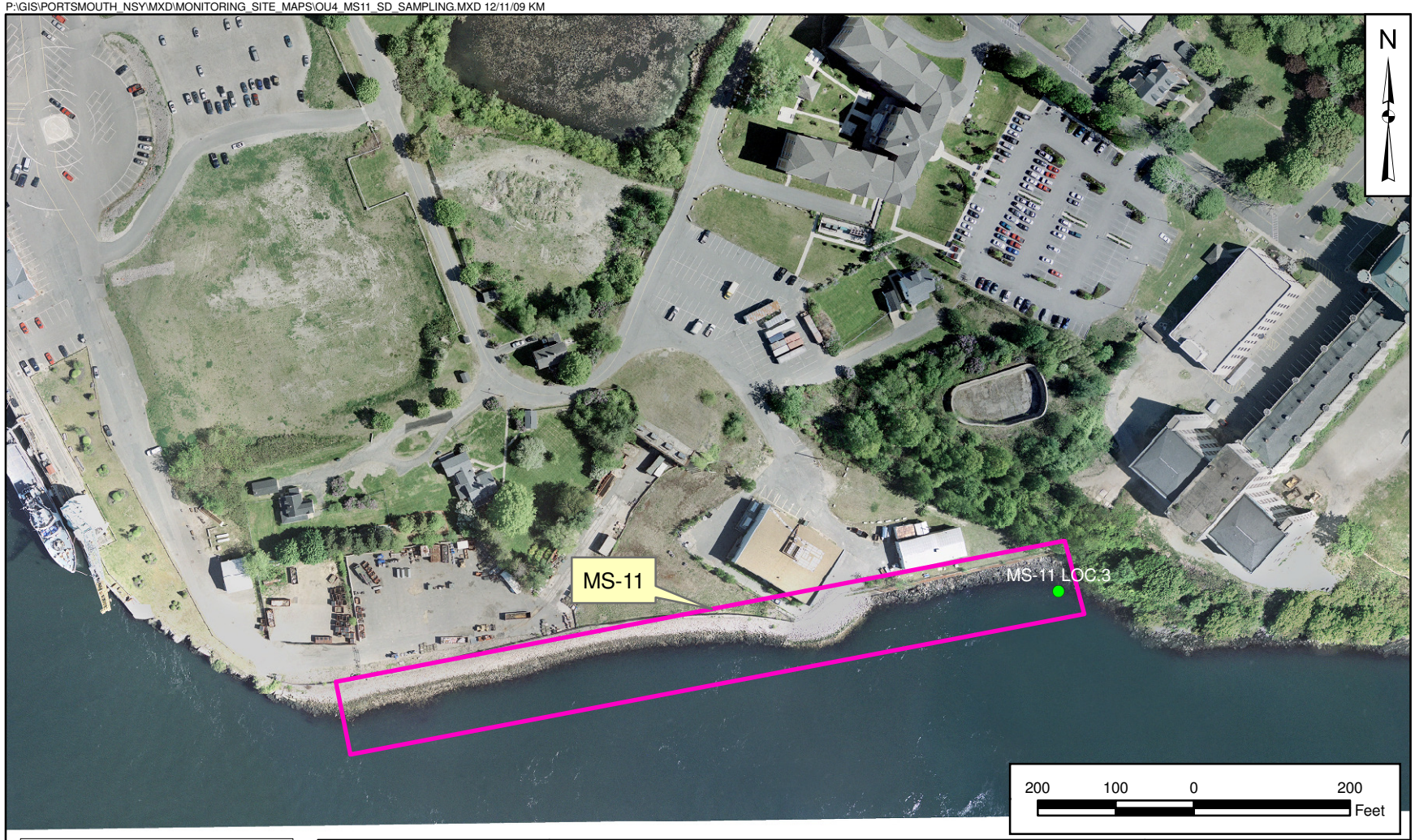
Legend

- Sediment Sampling Location
- Monitoring Station Boundary

NOTE:
2008 Aerial photograph recieved from Navy

DRAWN BY K. MOORE	DATE 12/11/09	**Tetra Tech NUS, Inc.**	CONTRACT NUMBER CTO WE29
CHECKED BY M. GRAY	DATE 12/11/09	SEDIMENT SAMPLING LOCATIONS AT MS-07, 08, AND 09 PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 8 / REV 0

P:\GIS\PORTSMOUTH_NSY\MXD\MONITORING_SITE_MAPS\OU4_MS11_SD_SAMPLING.MXD 12/11/09 KM



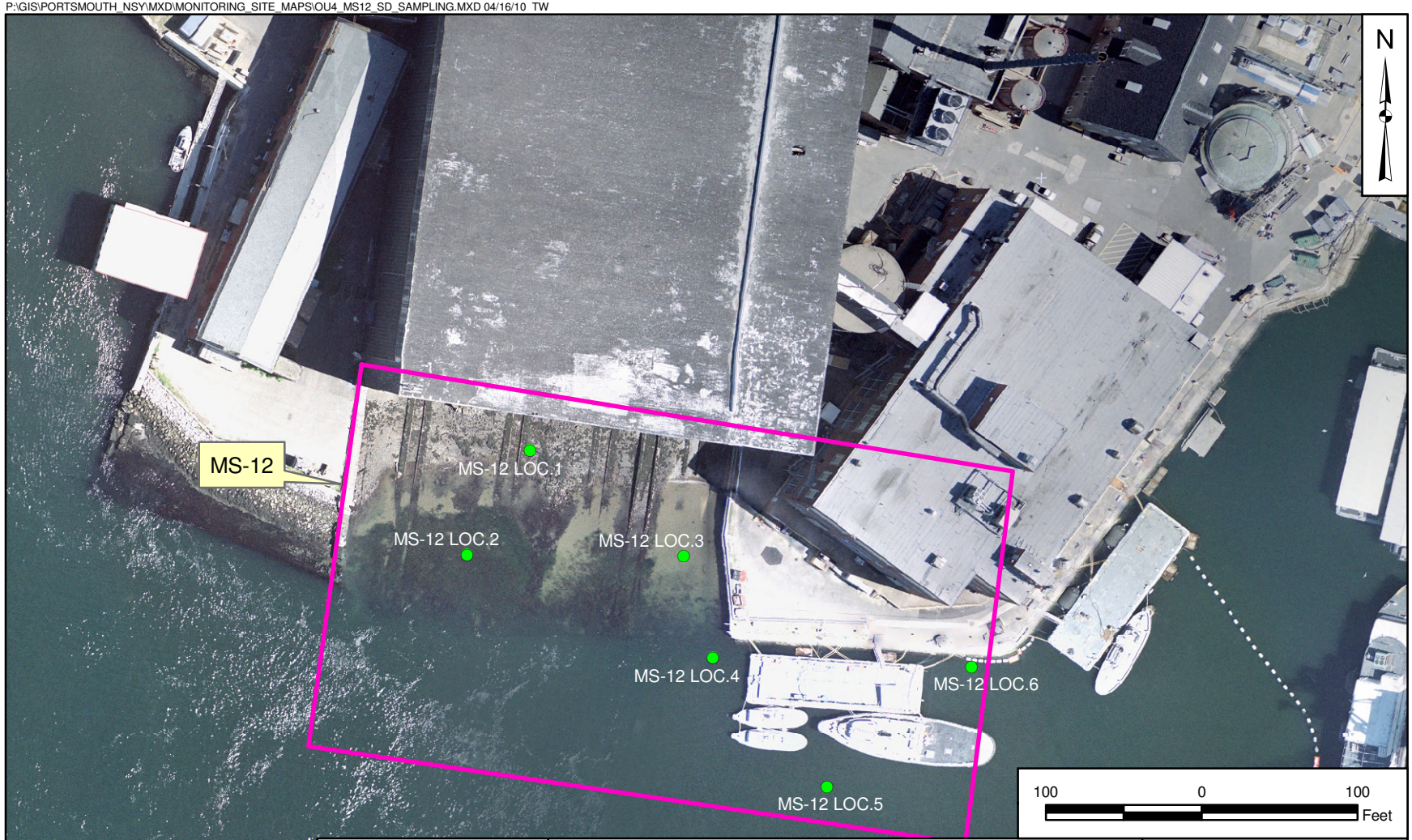
Legend

- Sediment Sampling Location
- Monitoring Station Boundary

NOTE:
2008 Aerial photograph recieved from Navy

DRAWN BY K. MOORE	DATE 12/11/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO WE29
CHECKED BY M. GRAY	DATE 12/11/09	SEDIMENT SAMPLING LOCATIONS AT MS-11 PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 9 / REV 0

P:\GIS\PORTSMOUTH_NSY\MXD\MONITORING_SITE_MAPS\OU4_MS12_SD_SAMPLING.MXD 04/16/10 TW



Legend

- Sediment Sampling Location
- Monitoring Station Boundary

NOTE:
2008 Aerial photograph recieved from Navy

DRAWN BY K. MOORE	DATE 12/11/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO WE29
CHECKED BY M. KRAUS	DATE 04/16/10	SEDIMENT SAMPLING LOCATIONS AT MS-12 PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 10 / REV 0

APPENDIX A

FIELD DOCUMENTATION FORMS

Tetra Tech NUS, Inc.

CONTAINER SAMPLE & INSPECTION SHEET

Page_____ of _____

Project Site Name: _____
Project Number: _____
Site Identification: _____
Container Number(s): _____
Sample Type: [] Grab
[] Composite

Sample ID No. _____
Sampled By: _____
C.O.C. No.: _____
Concentration: [] High
[] Medium
[] Low

CONTAINER SOURCE

DRUM:
[] Bung Top
[] Lever Lock
[] Bolted Ring
[] Other _____

TANK:
[] Plastic
[] Metal
[] Other _____

OTHER: _____

CONTAINER DESCRIPTION

COLOR: _____

CONDITION: _____

MARKINGS: _____

VOL. OF CONTENTS: _____

OTHER: _____

CONTAINER DISPOSITION

SAMPLED: _____

OPENED BUT NOT SAMPLED:
Reason _____
_____

NOT OPENED:
Reason _____
_____

CONTENTS DESCRIPTION

SINGLE PHASED: _____
_____

MULTIPHASE :

	Layer 1	Layer 2	Layer 3
Phase (Sol. or Liq.)	_____	_____	_____
Color	_____	_____	_____
Viscosity	L, M or H	L, M or H	L, M orH
% of Total Volume	_____	_____	_____

MONITOR READING:

SAMPLE and /or INSPECTION DATE & TIME:
_____HRS.
METHOD:_____

SAMPLER(S) and / or INSPECTOR(S) SIGNATURE:

ANALYSIS:

Tetra Tech NUS, Inc.

PROJECT: _____ **JOB #:** _____
LOCATION: _____ **DATE:** _____
PROJECT MANAGER: _____ **FOL:** _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1 _____			
2 _____			
3 _____			
4 _____			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place			
(OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

DAILY ACTIVITIES RECORD

PROJECT NAME: _____ **PROJECT NUMBER:** _____

CLIENT: _____ **LOCATION:** _____

DATE: _____ **ARRIVAL TIME:** _____

Tt NUS PERSONNEL: _____ **DEPARTURE TIME:** _____

CONTRACTOR: _____ **DRILLER:** _____

ITEM	QUANTITY ESTIMATE	QUANTITY TODAY	PREVIOUS TOTAL QUANTITY	CUMULATIVE QUANTITY TO DATE

COMMENTS: _____

_____

_____

_____

APPROVED BY:

_____ _____

Tt NUS REPRESENTATIVE DRILLER

DATE: _____

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name _____ CTO & Project Number _____ Task Mod. Number _____

Modification To (e.g. Work Plan) _____ Site/Sample Location _____ Date _____

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

Tetra Tech NUS, Inc.

PROJECT: _____ LOCATION: _____

JOB & CTO #: _____ MOBILIZATION DATE: _____

PROJECT MANAGER: _____ RETURN DATE: _____

FIELD PROJECT PRE-MOBILIZATION CHECKLIST

TRAVEL

___ Airline reservations
___ Hotel reservations/BOQs
___ Vehicle rental
___ Itinerary
___ Phone/pager number

DRILLING/DPT/SURVEY

Subcontractor

___ POC phone #/address
___ Drill Specification RFP
___ Contact (time & place to meet)
___ Confirm subcontract w/ TtNUS Procurement
___ Health and Safety documentation for all personnel on site
___ Copy of Drillers license
___ Well / boring permits

Utilities (2 weeks lead time)

___ Contact Site POC (Date:_____)
___ Contact Local "Call Before You Dig"
___ Utility Clearance Form

Forms

___ Boring logs / Test Pit logs
___ Well construction / development forms
___ Daily activity forms
___ IDW inventory
___ IDW drum labels
___ Chemical Inventory
___ MSDS's

EQUIPMENT MOBILIZATION

___ Equipment Requisition form completed / equipment ordered
___ 3rd Party rental / misc. equipment ordered
___ Equipment calibration forms
___ Span / calibration gas and regulator

SAMPLING

Forms

___ Sample log sheets
___ Low-flow purge data sheets
___ COC records
___ COC seals
___ Sample labels (from database group)

Laboratory

___ POC address/phone#
___ Order bottles / preservatives
___ Shipping address, also check Sat. address
___ Bottle & preservation req'ts from lab
___ _____

MISCELLANEOUS

Schedule

___ Plan field operations w/ Project manager

Documents for Field Program

___ Logbook(s)
___ Field Sampling plan
___ Health & Safety plan
___ Maps
___ H & S Guidance Manual

Authorization

___ Kick-off meeting held
___ Gov't rate letter
___ H&S/OSHA 40-hour certifcate
___ 8-Hour Refresher Training Certificate
___ Medical Clearance Letter
___ Supervisory Training Certificate
___ Health & Safety Clearance Letter
___ Full-size OSHA Poster

HYDROGEOLOGY EQUIPMENT

___ Slug test/pumping test forms
___ Groundwater elevation data sheets
___ Graph paper
___ Data Logger/transducer/data cable
___ Existing well construction & water level data
___ M-Scope, slug

SHIPPING

Forms

___ FedEx Airbills, local dropoff location & hours
___ FedEx Gov. Acct# (1771-8058-0)
___ Lab Shipping Labels
___ Warehouse Shipping Labels
___ Blank Labels

Supplies

___ Tape
___ Packing materials
___ Baggies, Large garbage bags

OTHER

___ Site POC name/phone #
___ Personnel information to POC
___ Mobilization schedule to POC
___ Site access authorizations
___ Field office / trailer arrangements made
___ Electric, phone hookups arranged
___ Steel-toed boots, safety glasses, & hard hat
___ First aid equipment
___ Insect repellent
___ _____
___ _____

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX B

FIELD STANDARD OPERATING PROCEDURES

TABLE OF CONTENTS

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1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS **2**

5.0 PROCEDURES **3**

5.1 INTRODUCTION 3
5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS 3
5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS 4
5.4 EXAMPLES OF SAMPLE NOMENCLATURE 5
5.5 FIELD QA/QC SAMPLE NOMENCLATURE 6
5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE 6

6.0 DEVIATIONS **7**

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Program Manager - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

Field Operations Leader (FOL) - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and sediment sample ID

A ~~or~~ and N 3 or 4 Characters	AAA 2 or 3 Characters	A ~~or~~ and N 3 to 6 Characters	NNNA 4 Characters
Site identifier	Sample type	Sample location	Sample ~~round~~

(2) Aqueous (groundwater or surface water) sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	NN 2 Characters	-A 1 Character
Site identifier	Sample type	Sample location	Round number	Filtered sample only

(3) Biota sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	AA 2 Characters	NNN 3 Characters
Site identifier	Sample type	Sample location	Species identifier	Sample group number

5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample round field is used to note the location, year and sampling season at which as sediment sample is collected. The first number of the code specifies the location at which the sample is collected. The next three values represent the last two digits of the year and the sampling season. The sampling season will be A or B denoting late summer or late winter/early spring of the given year.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

OU4 - Operable Unit 4
A01 - Area of Concern (AOC) 1
125 - SWMU 125
000 - Base- or facility-wide sample (e.g., upgradient well)
BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

AH - Ash Sample

AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation-Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample location - Examples of the location field are as follows:

M01 - Monitoring Station 1
001 - Monitoring well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation-derived waste drum number 96

Species identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

The sediment sample collected at Monitoring Station 1 location 2 during late summer sampling in December 2012 would be designated as OU4-SD-M01-212A.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.

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A MIS REQUEST FORM **7**

1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: _____ Request Date: _____
CTO: _____ Date Data Available for Production: _____
Project Manager: _____ Request in Support of: _____
Requestor: _____ Database Lead: _____
Program/Client: _____ GIS Lead: _____
State/EPA Region: _____ Statistics Lead: _____
Risk Lead: _____
Site Name(s) (Area, OU, etc.): _____
Sampling Date(s): _____
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other: _____

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL
Additional Instructions: _____

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other: _____
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other: _____
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

Statistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

Geostatistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (y261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (y261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

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ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical 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 chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) 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).
(4) 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 be 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 samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	Project: Site: Location:	
Sample No:		Matrix:
Date:	Time:	Preserve:
Analysis:		
Sampled by:	Laboratory:	

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE YEAR ___	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.

  - Avoidance of areas where control/custody cannot be maintained.

  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.

  - Avoidance of potentially contaminated areas.

  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

> ***Safety Reminder***
>
> When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:
>
> - All power is routed through a Ground Fault Circuit Interrupter (GFCI).
>
> - All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.
>
> In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.

  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.

  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.

  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.

  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents –
> Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

> ***SAFETY REMINDER***
> Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

> ***NOTE***
> In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

> ***CAUTION***
> Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

> ***CAUTION***
> In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 ~~Soil/~~Sediment Sampling Equipment

This section applies to ~~soil~~ sediment sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, ~~Macro Core samplers, etc.~~ and grab samplers

1. Remove all loose soil from the equipment through manual means.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.

3. Rinse the equipment with ~~tap water~~. ambient sea water for sediment samples

> **CAUTION**
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

~~4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.~~

~~5. Rinse the equipment with deionized water.~~

4. ~~6.~~ To the extent possible, allow components to air dry.

~~7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.~~

5. ~~8.~~ After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> **CAUTION**
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

> ***NOTE***
> Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

> ***NOTE***
> Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.
   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

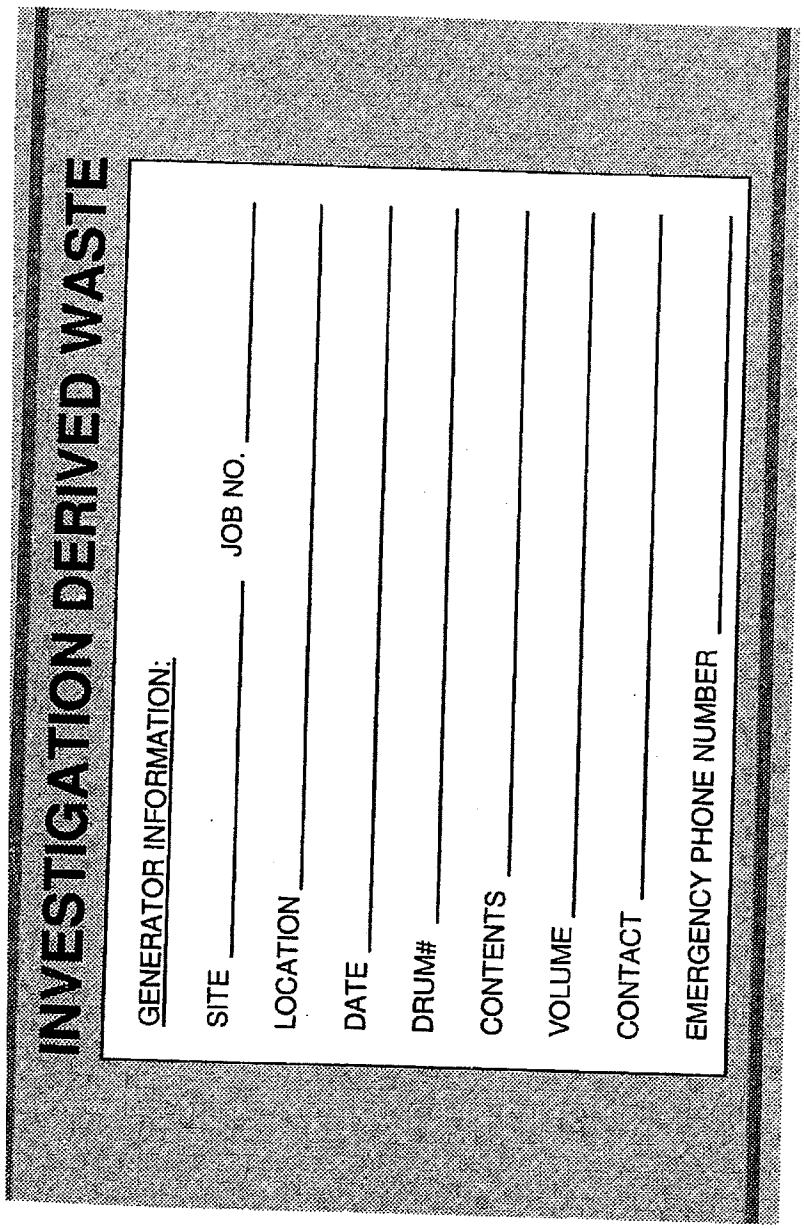
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
iDW Label



STANDARD OPERATING PROCEDURES

Interim Offshore Monitoring
Portsmouth Naval Shipyard, Kittery, Maine

1.0 STATION LOCATION

This section described the procedures for locating each station location. Coordinates for each station location will be provided by Tetra Tech NUS (TtNUS) prior to sampling dates.

1.1 EQUIPMENT LIST

- 24 ft Privateer boat with hydraulic winch and davit, fathometer, radio and required safety equipment including local charts
- Boat GPS – Survey grade Trimble AgGPS 132, accurate to within 1 meter
- Hand held DGPS – Trimble DGPS Pathfinder PRO XRS placed in a backpack with a short (1 meter) antenna, accurate to within 1 meter.
- Field log book and data sheet, aerial photographs of station location and station coordinates
- Cellular phone

1.2 METHODS

- Approach subtidal stations by boat using provided coordinates. Anchor the boat. Record station coordinates (note that because the boat may move slightly during sampling the stations coordinates may be several feet away from the actual sample location.
- Approach intertidal stations by boat or by foot using provided coordinates. Record sample coordinates. Permanent markers are available at some of the intertidal stations. If a permanent marker is available, record the distance from the marker to where the sample is collected.

2.0 SEDIMENT SAMPLING

2.1 EQUIPMENT LIST

- Boat GPS - Differential GPS Trimble AgGPS 132 with 421B Smart Antenna mounted over davit, accurate to within 1 meter
- 22 ft Privateer boat with 2 anchors hydraulic winch and davit, fathometer, radio and required safety equipment including local charts
- Hand held GPS - Differential GPS (Trimble DGPS Pathfinder PRO XRS) placed in a backpack with a short (1 meter) antenna, accurate to within 1meter.

- Van Veen and Ponar grab samplers with weights (increase penetration) and extensions (reduce penetration). A 0.1 m^2 and .04 m^2 are available. Both open from the top
- Push core system
- Stainless steel bowl
- Stainless steel scoops (Teflon scoops may be used in place of stainless steel scoops
- Sample jar (supplied by laboratory) and pre-printed sample labels
- Cooler, ice and temperature blank
- Alconox
- Squirt bottles
- Nitrile gloves
- Waste container
- Chain-of-custody forms, field log sheets, field data sheets, and log book
- Ziploc bags
- Disposable latex gloves and trash bags
- Scrub brushes and buckets
- Field log book and data sheet, aerial photographs of station location and station coordinates
- Cellular phone

2.2 METHODS

2.2.1 Subtidal Sample Collection-Ponar

- Deploy cleaned grab sampler. Sampler size will be dependent on bottom type. Estimate the depth of the sample. Accept only complete samples – those that penetrate the sediment 15 cm, where the surface is undisturbed and the sampler has completely closed.
- Add weights if the sampler does not penetrate to the required depth. Add extensions to reduce the penetration if sediment exits the top of the sampler indicating that the sampler penetrated too deep into the sediment.
- Record type of sampler used, added weights or horizontal extensions used to attain successful sample and estimate of sampler penetration into the sediment
- Carefully decant surface water through top of sampler.
- Record presence of any biota in the sampler.
- Using stainless steel scoop, remove sediment to 10 cm from the top of the sampler and place in stainless steel bowl.
- Samples will be a composite of **three** successful grab samples; move the boat slightly between grabs.
- Homogenize samples in the bowl and sub sample into labeled jars for analysis.
- Seal jars in Ziploc bags and store in cooler on wet ice.

2.2.2 Intertidal Sample Collection

- If station is approached by boat, collect sample as described above (2.2.1 Subtidal Sample Collection).
- If station is approached by foot, collect sediment sample to a depth of 10 cm using a stainless steel scoop and place in stainless steel bowl.
- Homogenize samples in the bowl and sub-sample into labeled jars for analysis.
- Seal jars in Ziploc bags and store in cooler on wet ice.

2.2.3 Push Core Sample Collection

- After arriving on station and anchoring the boat, measure water depth.
- Cut a three foot core tube and feed the tube into the core assembly.
- Lower the assembly to the bottom (mudline).
- Advance core assembly three feet,
- Retrieve the core assembly and remove the core tube. Cap and tape both ends and label the core tube

2.2.4 Decontamination

- Decontaminate grab sampler, mixing bowl and scoop between samples as follows:
    - Rinse with seawater to remove visible sediment
    - Rinse with potable water
    - Wash with scrub brush and Alconox
    - Rinse with potable water

2.3 STATION DOCUMENTATION

- For each station a field data sheet will be completed which includes the following information:
    - Date and time sample was collected
    - Benthic features – mud flat, sandy area, seeps, etc,
    - Biota present and/or biota indicators
        - Seagrasses, mussels, snails, etc.
        - Burrows
    - Debris present – metal scraps, garbage
    - Collection Methods
        - Method of locating the sample station
        - Distance to permanent marker
        - Equipment used (i.e., grab sampler, stainless steel scoop, weights or extensions added)
        - Difficulty collecting sample
    - Sediment description – color, texture, smell

    - Presence and direction of seeps
    - Indicate if duplicate samples were collected
- Sample jar labeling
    - Label jars with sample date and time (the sample number and analysis will be pre-printed on the label).
- Record sampling activities, field conditions, and weather conditions in a field log book.

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES AND CERTIFICATIONS

LABORATORY ACCREDITATION BUREAU

Certificate Number L2223

Certificate of Accreditation

Accredited to DoD ELAP and ISO/IEC 17025:2005

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 4, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 4th of November, 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation For Katahdin Analytical Services

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDE
GC/ECD	608 / 8081A,B / SOM01.2	4 4`-DDT
GC/ECD	608 / 8081A,B / SOM01.2	Aldrin
GC/ECD	608 / 8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	608 / 8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	608 / 8081A,B / SOM01.2	delta-BHC
GC/ECD	608 / 8081A,B / SOM01.2	Dieldrin
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan I
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan II
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	608 / 8081A,B / SOM01.2	Endrin
GC/ECD	608 / 8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor epoxide

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	608 / 8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	8082/8082A	3 4 4’ 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B/C	Diesel range organics (DRO)
GC/FID	8015B/C	Gasoline range organics (GRO)
GC/FID	8011 / 504	1 2-Dibromoethane (EDB)
GC/FID	8011 / 504	1 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	8260B,C / 524.2	1 1 1 2-Tetrachloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 1-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethene
GC/MS	8260B,C / 524.2	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromoethane (EDB)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloropropane
GC/MS	8260B,C / 524.2	1 3 5-Trimethylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 3-Dichlorobenzene
GC/MS	8260B,C / 524.2	1 3-Dichloropropane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C / 524.2	2 2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	2-Butanone
GC/MS	624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C / 524.2	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	2-Hexanone
GC/MS	8260B,C / 524.2	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	4-Methyl-2-pentanone
GC/MS	8260B,C / SOM01.2 / 524.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	624 / 8260B,C	Acrolein
GC/MS	624 / 8260B,C / 524.2	Acrylonitrile
GC/MS	8260B,C / 524.2	Allyl chloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Benzene
GC/MS	8260B,C / 524.2	Bromobenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Bromochloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromodichloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromoform
GC/MS	8260B,C / SOM01.2 / 524.2	Carbon disulfide
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Carbon tetrachloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2 / 524.2	cis-1 2-Dichloroethene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dibromochloromethane
GC/MS	8260B,C / 524.2	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dichlorodifluoromethane
GC/MS	8260B,C / 524.2	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / 524.2	Ethyl methacrylate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C / 524.2	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	Isopropyl benzene
GC/MS	8260B,C / SOM01.2 / 524.2	m p-xylenes
GC/MS	8260B,C / 524.2	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl bromide (Bromomethane)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C / 524.2	Methyl methacrylate
GC/MS	8260B,C / SOM01.2 / 524.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methylene chloride
GC/MS	8260B,C / 524.2	Naphthalene
GC/MS	8260B,C / 524.2	n-Butylbenzene
Gc/ms	8260B,C / 524.2	n-Propylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	o-Xylene
GC/MS	8260B,C / 524.2	p-Isopropyltoluene
GC/MS	8260B,C / 524.2	Propionitrile
GC/MS	8260B,C / 524.2	sec-butylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C / 524.2	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	8260B,C / 524.2	Tetrahydrofuran
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Toluene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 2-Dichloroethylene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C / 524.2	trans-1 4-Dichloro-2-butuene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Vinyl chloride
GC/MS	624 8260B,C	Xylene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	625 / 8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	625 / 8270C,D / SOM01.2	2-Chlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	625 / 8270C,D / SOM01.2	3 3'-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	625 / 8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	625 / 8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	7,12-Dimethylphenethylamine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthene
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthylene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline
GC/MS	625 / 8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	625 / 8270C,D	Benzidine
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	625 / 8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	625 / 8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	625 / 8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	625 / 8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	625 / 8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	625 / 8270C,D / SOM01.2	Fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Fluorene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	625 / 8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D	Isodrin
GC/MS	625 / 8270C,D / SOM01.2	Isophorone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methy methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	625 / 8270C,D / SOM01.2	Naphthalene
GC/MS	625 / 8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	625 / 8270C,D / SOM01.2	Phenanthrene
GC/MS	625 / 8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	625 / 8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
HPLC	8330/8330A/8330B	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B	2-Nitrotoluene
HPLC	8330/8330A/8330B	3-Nitrotoluene
HPLC	8330/8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B	4-Nitrotoluene
HPLC	8330/8330A/8330B	Hexahydro-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B	Nitrobenzene
HPLC	8330/8330A/8330B	Nitroglycerin
HPLC	8330/8330A/8330B	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B	Tetryl

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	245.1 / 7470A / ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	200.7 / 6010B,C / ILM05.3	Aluminum
ICP	200.7 / 6010B,C / ILM05.3	Antimony
ICP	200.7 / 6010B,C / ILM05.3	Arsenic
ICP	200.7 / 6010B,C / ILM05.3	Barium
ICP	200.7 / 6010B,C / ILM05.3	Beryllium
ICP	200.7 / 6010B,C	Boron
ICP	200.7 / 6010B,C / ILM05.3	Cadmium
ICP	200.7 / 6010B,C / ILM05.3	Calcium
ICP	200.7 / 6010B,C / ILM05.3	Chromium
ICP	200.7 / 6010B,C / ILM05.3	Cobalt
ICP	200.7 / 6010B,C / ILM05.3	Copper
ICP	200.7 / 6010B,C / ILM05.3	Iron
ICP	200.7 / 6010B,C / ILM05.3	Lead
ICP	200.7 / 6010B,C / ILM05.3	Magnesium
ICP	200.7 / 6010B,C / ILM05.3	Manganese
ICP	200.7 / 6010B,C	Molybdenum
ICP	200.7 / 6010B,C / ILM05.3	Nickel
ICP	200.7 / 6010B,C / ILM05.3	Potassium
ICP	200.7 / 6010B,C / ILM05.3	Selenium
ICP	200.7	Silicon
ICP	200.7 / 6010B,C / ILM05.3	Silver
ICP	200.7 / 6010B,C / ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	200.7 / 6010B,C / ILM05.3	Thallium
ICP	200.7 / 6010B,C	Tin
ICP	200.7 / 6010B,C	Titanium
ICP	200.7 / 6010B,C / ILM05.3	Vanadium
ICP	200.7 / 6010B,C / ILM05.3	Zinc
ICP/MS	200.8 / 6020/6020A / ILM05.3	Aluminum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Antimony
ICP/MS	200.8 / 6020/6020A / ILM05.3	Arsenic
ICP/MS	200.8 / 6020/6020A / ILM05.3	Barium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Beryllium
ICP/MS	200.8 / 6020/6020A	Boron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cadmium
ICP/MS	200.8 / 6020/6020A	Calcium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Chromium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cobalt
ICP/MS	200.8 / 6020/6020A / ILM05.3	Copper
ICP/MS	200.8 / 6020/6020A	Iron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Lead
ICP/MS	200.8 / 6020/6020A / ILM05.3	Magnesium
ICP/MS	200.8 / 6020/6020A	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	200.8 / 6020/6020A	Molybdenum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Nickel
ICP/MS	200.8 / 6020/6020A	Potassium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Selenium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silicon
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silver
ICP/MS	200.8 / 6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Thallium
ICP/MS	200.8 / 6020/6020A	Tin
ICP/MS	200.8 / 6020/6020A	Titanium
ICP/MS	200.8	Uranium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Vanadium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Zinc
IC	300.0 / 9056/9056A	Bromide
IC	300.0 / 9056/9056A	Chloride
IC	300.0 / 9056/9056A	Nitrate as N
IC	300.0 / 9056/9056A	Nitrite as N
IC	300.0 / 9056/9056A	Nitrate + Nitrite
IC	300.0 / 9056/9056A	Orthophosphate as P
IC	300.0 / 9056/9056A	Sulfate
Titration	310.2 / 2320B	Alkalinity
Caculation	2340C	Hardness
Gravimetric	1664A	Oil and Grease
Gravimetric	2540 B, C, D	Solids
ISE	120.1 / 2510 B	Conductivity
ISE	2520B	Practical Salinity
ISE	4500F- C	Fluoride
ISE	4500H+ B	pH
ISE	5210B	TBOD / CBOD
Physical	1010 A	Ignitability
Physical	9040C	pH
Titration	2340B	Hardness
Titration	4500SO_3 B	Sulfite
Titration	9034 / 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	180.1 / 2130B	Turbidity
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	335.4 / 9012B / 4500-CN G	Amenable cyanide
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO3 F	Nitrate + Nitrite

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	353.2 / 4500NO3 F	Nitrate as N
UV/VIS	353.2 / 4500NO3 F	Nitrite as N
UV/VIS	365.1 / 4500P E	Orthophosphate as P
UV/VIS	365.4	Phosphorus total
UV/VIS	376.3	AVS-SEM
UV/VIS	410.4	COD
UV/VIS	420.1 / 9065	Total Phenolics
UV/VIS	4500Cl G	Total Residual Chlorine
UV/VIS	5540C	MBAS
UV/VIS	7196A / 3500-Cr D	Chromium VI
UV/VIS	9012B / ILM05.3/ 335.4	Total Cyanide
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	3640A	Gel Permeation Clean-up
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3660B	Sulfur Clean-Up
Cleanup Methods	3665A	Sulfuric Acid Clean-Up
Organic Preparation	3510C	Separatory Funnel Extraction
Organic Preparation	3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	3010A	Hotblock
Volatile Organic Preparation	5030B,C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	8081A,B / SOM01.2	4 4`-DDE
GC/ECD	8081A,B / SOM01.2	4 4`-DDT
GC/ECD	8081A,B / SOM01.2	Aldrin
GC/ECD	8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	8081A,B / SOM01.2	delta-BHC
GC/ECD	8081A,B / SOM01.2	Dieldrin
GC/ECD	8081A,B / SOM01.2	Endosulfan I
GC/ECD	8081A,B / SOM01.2	Endosulfan II
GC/ECD	8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	8081A,B / SOM01.2	Endrin
GC/ECD	8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Heptachlor
GC/ECD	8081A,B / SOM01.2	Heptachlor epoxide
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1016 (PCB-1016)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B,C	Diesel range organics (DRO)
GC/FID	8015B,C	Gasoline range organics (GRO)
GC/FID	8011	EDB
GC/FID	8011	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C	1 1 1 2-Tetrachloroethane
GC/MS	8260B,C / SOM01.2	1 1 1-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	8260B,C / SOM01.2	1 1 2-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethylene
GC/MS	8260B,C	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2	1 2-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 2-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 2-Dichloropropane
GC/MS	8260B,C	1 3 5-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 3-Dichlorobenzene
GC/MS	8260B,C	1 3-Dichloropropane
GC/MS	8260B,C / SOM01.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C	2 2-Dichloropropane
GC/MS	8260B,C / SOM01.2	2-Butanone
GC/MS	8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2	2-Hexanone
GC/MS	8260B,C	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2	4-Methyl-2-pentanone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	8260B,C	Acrolein
GC/MS	8260B,C	Acrylonitrile
GC/MS	8260B,C	Allyl chloride
GC/MS	8260B,C / SOM01.2	Benzene
GC/MS	8260B,C	Bromobenzene
GC/MS	8260B,C / SOM01.2	Bromochloromethane
GC/MS	8260B,C / SOM01.2	Bromodichloromethane
GC/MS	8260B,C / SOM01.2	Bromoform
GC/MS	8260B,C / SOM01.2	Carbon disulfide
GC/MS	8260B,C / SOM01.2	Carbon tetrachloride
GC/MS	8260B,C / SOM01.2	Chlorobenzene
GC/MS	8260B,C / SOM01.2	Chloroethane
GC/MS	8260B,C / SOM01.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2	cis-1 2-Dichloroethene
GC/MS	8260B,C / SOM01.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	8260B,C / SOM01.2	Dibromochloromethane
GC/MS	8260B,C	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2	Dichlorodifluoromethane
GC/MS	8260B,C	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / SOM01.2	EDB
GC/MS	8260B,C	Ethyl methacrylate
GC/MS	8260B,C / SOM01.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol
GC/MS	8260B,C / SOM01.2	Isopropyl benzene
GC/MS	SOM01.2	m p-xylenes
GC/MS	8260B,C	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	8260B,C / SOM01.2	Methyl bromide (Bromomethane)
GC/MS	8260B,C / SOM01.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C	Methyl methacrylate
GC/MS	8260B,C / SOM01.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	8260B,C / SOM01.2	Methylene chloride
GC/MS	8260B,C	Naphthalene
GC/MS	8260B,C	n-Butylbenzene
GC/MS	8260B,C	n-proplybenzene
GC/MS	8260B,C	o-Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C	p-Isopropyltoluene
GC/MS	8260B,C	Propionitrile
GC/MS	8260B,C	sec-butylbenzene
GC/MS	8260B,C / SOM01.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	8260B,C / SOM01.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	8260B,C	Tetrahydrofuran
GC/MS	8260B,C / SOM01.2	Toluene
GC/MS	8260B,C / SOM01.2	trans-1 2-Dichloroethylene
GC/MS	8260B,C / SOM01.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C	Trans-1 4-Dichloro-2-butuene
GC/MS	8260B,C / SOM01.2	Trichloroethene (Trichloroethylene)
GC/MS	8260B,C / SOM01.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	8260B,C / SOM01.2	Vinyl chloride
GC/MS	8260B,C	Xylene
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	8270C,D / SOM01.2	2-Chlorophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	8270C,D / SOM01.2	Acenaphthene
GC/MS	8270C,D / SOM01.2	Acenaphthylene
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	8270C,D	Benzidine
GC/MS	8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	8270C,D / SOM01.2	Fluoranthene
GC/MS	8270C,D / SOM01.2	Fluorene
GC/MS	8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	8270C,D	Isodrin
GC/MS	8270C,D / SOM01.2	Isophorone
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methyl methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	8270C,D / SOM01.2	Naphthalene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	8270C,D/ SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	8270C,D / SOM01.2	Phenanthrene
GC/MS	8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
GC/MS	8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine
GC/MS	8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	8270C,D / SOM01.2	2 4-Dinitrophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D / SOM01.2	3 3`-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D	7,12-Dimethylphenethylamine
HPLC	8330/8330A/8330B (Analysis Only)	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	3-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	Hexahydr-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B (Analysis Only)	Nitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	Nitroglycerin
HPLC	8330/8330A/8330B (Analysis Only)	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B (Analysis Only)	Tetryl
CVAA	7471B/ ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	6010B,C /ILM05.3	Aluminum
ICP	6010B,C /ILM05.3	Antimony
ICP	6010B,C /ILM05.3	Arsenic
ICP	6010B,C /ILM05.3	Barium
ICP	6010B,C /ILM05.3	Beryllium
ICP	6010B,C	Boron
ICP	6010B,C /ILM05.3	Cadmium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP	6010B,C /ILM05.3	Calcium
ICP	6010B,C /ILM05.3	Chromium
ICP	6010B,C /ILM05.3	Cobalt
ICP	6010B,C /ILM05.3	Copper
ICP	6010B,C /ILM05.3	Iron
ICP	6010B,C /ILM05.3	Lead
ICP	6010B,C /ILM05.3	Magnesium
ICP	6010B,C /ILM05.3	Manganese
ICP	6010B,C	Molybdenum
ICP	6010B,C /ILM05.3	Nickel
ICP	6010B,C /ILM05.3	Potassium
ICP	6010B,C /ILM05.3	Selenium
ICP	200.7	Silicon
ICP	6010B,C /ILM05.3	Silver
ICP	6010B,C /ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	6010B,C /ILM05.3	Thallium
ICP	6010B,C	Tin
ICP	6010B,C	Titanium
ICP	6010B,C /ILM05.3	Vanadium
ICP	6010B,C /ILM05.3	Zinc
ICP/MS	6020/6020A / ILM05.3	Aluminum
ICP/MS	6020/6020A / ILM05.3	Antimony
ICP/MS	6020/6020A / ILM05.3	Arsenic
ICP/MS	6020/6020A / ILM05.3	Barium
ICP/MS	6020/6020A / ILM05.3	Beryllium
ICP/MS	6020/6020A	Boron
ICP/MS	6020/6020A / ILM05.3	Cadmium
ICP/MS	6020/6020A	Calcium
ICP/MS	6020/6020A / ILM05.3	Chromium
ICP/MS	6020/6020A / ILM05.3	Cobalt
ICP/MS	6020/6020A / ILM05.3	Copper
ICP/MS	6020/6020A	Iron
ICP/MS	6020/6020A / ILM05.3	Lead
ICP/MS	6020/6020A / ILM05.3	Magnesium
ICP/MS	6020/6020A	Manganese
ICP/MS	6020/6020A	Molybdenum
ICP/MS	6020/6020A / ILM05.3	Nickel
ICP/MS	6020/6020A	Potassium
ICP/MS	6020/6020A / ILM05.3	Selenium
ICP/MS	6020/6020A / ILM05.3	Silver
ICP/MS	6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	6020/6020A / ILM05.3	Thallium
ICP/MS	6020/6020A	Tin

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	6020/6020A	Titanium
ICP/MS	6020/6020A / ILM05.3	Vanadium
ICP/MS	6020/6020A / ILM05.3	Zinc
IC	9056/9056A	Chloride
IC	9056/9056A	Fluoride
IC	9056/9056A	Nitrate as N
IC	9056/9056A	Nitrite as N
IC	9056/9056A	Sulfate
Gravimetric	9070A / 9071B	Oil and Grease
Physical	1010A	Ignitability
Physical	9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
TOC	Lloyd Kahn	Total organic carbon
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	376.3	AVS-SEM
UV/VIS	3500Fe D	Ferrous Iron
Cleanup Methods	3630C	Silica Gel
UV/VIS	7196	Chromium VI
UV/VIS	7196A	Chromium VI
UV/VIS	9012B / ILM05.3	Total cyanide
Preparation	**Method**	**Type**
Preparation	1311	Toxicity Characteristic Leaching Procedure
Preparation	1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	3660B	Sulfur
Cleanup Methods	3620C	Florsil
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3640A	GPC
Organic Preparation	3540C	Soxhlet Extraction
Organic Preparation	3545A	Pressurized Fluid Extraction
Organic Preparation	3550C	Sonication
Inorganics Preparation	3050B	Hotblock
Inorganics Preparation	3060A	Alkaline Digestion
Volatile Organics Preparation	5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: November 4, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09

May 14, 2010

Jeannie Milholland
SGS Environmental Services - Wilmington
5500 Business Drive
Wilmington NC 28405

Dear Ms. Milholland:

Congratulations! Your laboratory has been approved for accreditation by the American Association for Laboratory Accreditation (A2LA) in the Environmental field of testing for the tests (or types of tests) identified in the enclosed Scope of Accreditation. An A2LA Certificate numbered 2726.01 is enclosed and may be displayed in a prominent place in your facility. Your Certificate and Scope of Accreditation have also been added to the searchable database of accredited laboratories contained on our website, www.A2LA.org.

Your laboratory is now accredited to ISO/IEC 17025:2005 through **April 30, 2012**, by virtue of the on-site assessment of your laboratory and an evaluation of all activity related to the assessment by the A2LA Accreditation Council. One year prior to your accreditation anniversary date, you must pay an annual review fee and undergo an on-site surveillance visit. In addition, A2LA must be notified in writing within 30 days at any time that significant changes occur in your laboratory's location, ownership, management, authorized representative, primary contact or major facilities.

The A2LA Board of Directors has adopted a policy that all accredited laboratories must participate in proficiency testing programs where relevant programs are available as documented in A2LA *R103 - General Requirements: Proficiency Testing for ISO/IEC 17025 Laboratories.* Your participation in the NELAC Proficiency Testing Program presently meets this requirement. Please send us copies of the summary results from this program as you receive them.

Use of your accreditation for promotional and advertising purposes is encouraged when it is in accordance with the enclosed A2LA document *P101 - Reference to A2LA Accredited Status – A2LA Advertising Policy.* For your convenience, copies of the "A2LA Accredited" symbol are enclosed. An electronic version of the combined "ILAC MRA – A2LA Accredited" symbol is available upon request. Please note that if you wish to use this combined symbol, you must present your proposed usage to A2LA for review and approval before you begin actual use of the combined symbol. When promoting or providing proof of your accreditation, please use your Scope of Accreditation, as this document details the specific tests (or types of tests) which are accredited. The certificate is to be used for display purposes only.

We are pleased to welcome you as an A2LA accredited laboratory.

Sincerely,

Peter S. Unger
President

Asst ID: 14274
Master Code: 129667
Cert No.: Environmental (2726.01)

The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

SGS ENVIRONMENTAL SERVICES – WILMINGTON, NC DIVISION

Wilmington, NC

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (QSM v4.1); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

Presented this 13th day of May 2010.

President & CEO
For the Accreditation Council
Certificate Number 2726.01
Valid to April 30, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

SGS NORTH AMERICA INC. – WILMINGTON, NC DIVISION
5500 Business Drive
Wilmington, NC 28411
Jeannie Milholland Phone: (910)-350-1903
Jeannie.milholland@sgs.com

ENVIRONMENTAL

Valid To: April 30, 2012 Certificate Number: 2726.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1)) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

ICP-OES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Misc.- Electronic Probes (pH), Hazardous Waste Characteristics Tests, Total Organic Carbon, High Resolution Gas Chromatography/High Resolution Mass Spectrometry

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	Tissue
PCBs			
PCB 1	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 2	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 3	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 4	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 5	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 6	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 7	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 8	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 9	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 10	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 11	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 12	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 13	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 14	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 15	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 16	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 17	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	Tissue
PCB 18	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 19	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 20	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 21	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 22	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 73	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 74	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 75	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 76	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 77	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 78	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 79	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 80	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 81	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 82	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 83	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 84	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 85	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 86	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 87	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 88	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 89	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 90	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 91	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 92	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 93	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 94	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 95	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 96	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 97	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 98	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 99	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 100	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 101	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 102	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 103	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 104	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 105	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 106	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 107	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 108	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 109	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 110	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 111	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	Tissue
PCB 112	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 113	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 114	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 115	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 116	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 117	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 118	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 119	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 120	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 121	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 122	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 123	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 124	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 125	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 126	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 127	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 128	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 129	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 130	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 131	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 132	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 133	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 134	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 135	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 136	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 137	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 138	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 139	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 140	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 141	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 142	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 143	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 144	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 145	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 146	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 147	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 148	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 149	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 150	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 151	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 152	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 153	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 154	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 155	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	Tissue
PCB 156	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 157	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 158	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 159	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 160	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 161	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 162	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 163	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 164	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 165	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 166	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 167	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 168	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 169	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 170	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 171	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 172	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 173	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 174	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 175	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 176	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 177	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 178	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 179	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 180	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 181	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 182	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 183	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 184	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 185	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 186	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 187	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 188	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 189	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 190	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 191	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 192	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 193	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 194	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 195	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 196	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 197	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 198	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 199	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	Tissue
PCB 200	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 201	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 202	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 203	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 204	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 205	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 206	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 207	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 208	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
PCB 209	EPA 1668A/1668B	EPA 1668A/1668B	EPA 1668A/1668B
Sample Preparation	Liquid Liquid Extraction 3520	Liquid Extraction 3520, Soxlet 3540	Soxlet 3540
Dioxins/Furans			
2,3,7,8-TCDD	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDD	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8-HxCDD	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDD	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDD	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDD	EPA 8290A	EPA 8290A	EPA 8290A
OCDD	EPA 8290A	EPA 8290A	EPA 8290A
2,3,7,8-TCDF	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDF	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,7,8-PeCDF	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8-HxCDF	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDF	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDF	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,6,7,8-HxCDF	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDF	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,9-HpCDF	EPA 8290A	EPA 8290A	EPA 8290A
OCDF	EPA 8290A	EPA 8290A	EPA 8290A
Sample Preparation	Liquid Liquid Extraction 3520	Liquid Extraction 3520, Soxlet 3540	Soxlet 3540

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste	Tissue
Dioxins/Furans				
2,3,7,8-TCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8-PeCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,7,8-HxCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,6,7,8-HxCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8,9-HxCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,6,7,8-HpCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
OCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste	Tissue
2,3,7,8-TCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8-PeCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
2,3,4,7,8-PeCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,7,8-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,6,7,8-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8,9-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
2,3,4,6,7,8-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,6,7,8-HpCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,7,8,9-HpCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
OCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
Sample Preparation	Liquid/Liquid 3520	Liquid/Liquid 3520	Soxlet 3540	Soxlet 3540

In addition, in recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, and the 2003 NELAC Chapter 5 Standard) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Parameter/Analyte	Foods
Dioxins/Furans	
2,3,7,8-TCDD	EPA 1613B
1,2,3,7,8-PeCDD	EPA 1613B
1,2,3,4,7,8-HxCDD	EPA 1613B
1,2,3,6,7,8-HxCDD	EPA 1613B
1,2,3,7,8,9-HxCDD	EPA 1613B
1,2,3,4,6,7,8-HpCDD	EPA 1613B
OCDD	EPA 1613B
2,3,7,8-TCDF	EPA 1613B
1,2,3,7,8-PeCDF	EPA 1613B
2,3,4,7,8-PeCDF	EPA 1613B
1,2,3,4,7,8-HxCDF	EPA 1613B
1,2,3,6,7,8-HxCDF	EPA 1613B
1,2,3,7,8,9-HxCDF	EPA 1613B
2,3,4,6,7,8-HxCDF	EPA 1613B
1,2,3,4,6,7,8-HpCDF	EPA 1613B
1,2,3,4,7,8,9-HpCDF	EPA 1613B
OCDF	EPA 1613B
Sample Preparation	Soxlet 3540 Liquid/liquid 3520

[1] This laboratory offers on-site testing services.
[2] Test are performed in the laboratories mobile facilities.

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: A Haley Date: 020101

Operations Manager: John C. Benton Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Dennis P. Lufkin Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 + Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited Section 7.5.1 - initial cal table	LAD	02/08	02/08

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address diffent 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

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-213-07, titled "ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-07, 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, Inc. 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.

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.

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.

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 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.

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 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.

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.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

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 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 a respirator and 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 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be onstructed 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: 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.

5.0 REAGENTS

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

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 3. 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 3. 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.

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 (R, U, or G)
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 (R, U, or G). 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 (R, U, or G).

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	30.0-80.0 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	280°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

GC/MS OPERATING CONDITIONS – DFTPP (CONT.)	
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.

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, 1.0, 2.0, 5.0 and 8.0 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 may need to be calibrated at higher concentrations. A second standard is prepared containing these compounds. The two

standards are combined as in the example below. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

Example –
For a calibration at the following levels:
Calibration mix A would be prepared containing ALL analytes at 20 ng/ul
Calibration Mix B would be prepared containing PCP, HCB and BEHP at 20 ng/ul.

Final PAH conc. (ng/uL)	Final PCP, HCB, BEHP Conc. (ng/ul)	Cal-Mix A Added (uL)	Cal-Mix B Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	1.0	10	40	950	1000
0.50	2.0	25	75	900	1000
1.0	3.0	50	100	850	1000
2.0	4.0	100	100	800	1000
5.0	5.0	250	0	750	1000
8.0	8.0	400	0	600	1000

Note: Calibration Mix B only is used to boost the PCP, HCB and BEHP concentrations in Cal. levels 1 through 4.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

GC/MS OPERATING CONDITIONS – CALIBRATION and SAMPLES	
Column temperature program	40°C for 3 min. to 300°C at 10°/min.
Final column temperature hold	300°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	2.0-6.0 minutes (time may vary dependent upon column length)
Sample volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the five 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.
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.
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 SSTD1.0 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 1.0 ng/uL.

After quantitation of the 1.0 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 1.0 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 proceed using initial calibration response factors.

7.5.4. 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.

7.6 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 judgements. 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.
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 measrued 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

The four surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10 and Pyrene-d10) must meet the current statistically derived 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 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 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.
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 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

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.

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 (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% 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.**

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.

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 are determined annually per type of instrument and filed with the Organic Department Manager and 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 8270 for other method performance parameters and requirements.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 8270C.

"USEPA Contract Laboratory Program Statement of Work for Organics Analysis," Rev. 02/88.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. June, 1998.

Katahdin SOP CA-101, Equipment Maintenance

Department of Defence Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

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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
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. 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 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

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-07	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 3

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4	Phenanthrene
Target and Surrogates:	Hexachlorobenzene (special)
Hexachloroethane (special)	Anthracene
Internal Standard: Naphthalene-d8	Fluoranthene
Target and Surrogates:	Carbazole (special)
Naphthalene	Di-n-butylphthalate (special)
1-Methylnaphthalene (dredge)	**Internal Standard: Chrysene-d12**
2-Methylnaphthalene	Target and Surrogates:
2-Methylnaphthalene-D10 (surrogate)	Pyrene
Internal Standard: Acenaphthene-d10	Benzo(a)Anthracene
Target and Surrogates:	Chrysene
Biphenyl (dredge)	Bis-(2-ethylhexyl)phthalate (special)
2,6 Dimethylnapthalene (dredge)	Pyrene-d10 (surrogate)
Acenaphthylene	**Internal Standard: Perylene-d12**
Acenaphthene	Target and Surrogates:
Fluorene	Perylene (dredge)
2-Fluorene-d10 (surrogate)	Benzo(b)fluoranthene
2,4-Dibromophenol (surrogate)	Benzo(k)fluoranthene
2-Chloronaphthalene (special)	Benzo(e)pyrene (dredge)
Internal Standard: Phenanthrene-d10	Benzo(a)pyrene
Target and Surrogates:	Indeno(1,2,3-cd)pyrene
Pentachlorophenol (special)	Dibenz(a,h)anthracene
1-Methylphenanthrene (dredge)	Benzo(ghi)perylene

`TABLE 4

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

TABLE 5

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
Naphthalene	128	129,127
2-Methylnaphthalene	142	115
Acenaphthylene	152	151,153
Acenaphthene	153	152,154
Fluorene	166	165,167
Phenanthrene	178	179,176
Anthracene	178	179,176
Fluoranthene	202	200,203
Pyrene	202	200,203
Benzo(a)anthracene	228	226
Chrysene	228	226
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Indeno(1,2,3-cd)pyrene	276	277
Dibenz(ah)anthracene	278	279
Benzo(ghi)perylene	276	277
1-Methyl naphthalene (dredge)	142	115
Biphenyl (dredge)	154	76
2,6-Dimethyl Naphthalene (dredge)	156	141
1-Methyl phenanthrene (dredge)	192	191,193
Benzo (e) pyrene (dredge)	252	125
Perylene (dredge)	252	125
Carbazole	167	166,139
Pentachlorophenol	266	264,268
Hexachlorobenzene	284	282, 286
Bis(2-ethylhexyl)phthalate	149	167
2-Chloronaphthalene	162	127, 164
Di-n-butylphthalate	149	104 150
Hexachloroethane	117	201, 199
2-methylnaphthalene-d10 (surrogate)	152	125
Fluorene-d10 (surrogate)	176	175, 177
Pyrene-d10 (surrogate)	212	210, 213
2,4-Dibromophenol (surrogate)	252	63, 143
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	134,137
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	189
Chrysene-d12 (istd.)	240	241,236
Perylene-d12 (istd.)	264	260

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.

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: 5970-X

DATE OF DFTPP INJECTION: 03/009

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	X0600	1	1	DFTP0390	2.0	JK	ok
	SSTD060X0310	X9437		2	X625A022	1.0		✓
	150	38		3				✓
	100	39		4				✓
	30	40		5				✓
	05	41		6				✓
	3510 MIX 1	42		7				ok
	2	43		8				
	3	44		9				
	4	45		10				
	5	46		11				
	6	47		12				
	7	48		13				
	8	49		14				
	9	50		15				
	BLANK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	50856
CAL. STD.	50829-30 50847-49
IS MIX	AHP0934

REVIEWED AND APPROVED BY:
DATE:

QAMS302

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

GCMS LABORATORY
REVIEWED BY/DATE:

AMP0946

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013
1 mL
FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer
STORAGE Ambient
POISON [redacted]

Dual 3/16/06 JCA

AMP0947

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012
1 mL
FOR LABORATORY USE ONLY
STORAGE Ambient
FLAMMABLE [redacted]

AMP0948

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015
1 mL
FOR LABORATORY USE ONLY
STORAGE Ambient
POISON [redacted]

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

GC/MS SVOA STANDARD PREP LOGBOOK

S0863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLh	AMP0844	8270 Mixes	300	2-22-07	4.2ml	150 ng/ml
					AMP0837	↓	350	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	100	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0659	Organophosphorus pest	300	8-14-06		
					AMP0931	Benzoic Acid	↓	3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0906	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					S0861	DEA	300	3-13-07		
					B43590	$MeCl_2$	550	—		
S0864	8270 level 1	3-15-06	7-7-06	JLh	S0863	8270 Stock	70	7-7-06	1.05ml	10 ng/ml
					B43590	$MeCl_2$	980			
S0865	8270 level 2	3-15-06	7-7-06	JLh	S0863	8270 Stock	150	7-7-06	0.90ml	25 ng/ml
					B43590	$MeCl_2$	750			
S0866	8270 level 3	3-15-06	7-7-06	JLh	S0863	8270 Stock	600	7-7-06	1.8ml	50 ng/ml
					B43590	$MeCl_2$	1200			
S0867	8270 level 4	3-15-06	7-7-06	JLh	S0864	8270 Stock	700	7-7-06	1.05ml	100 ng/ml
					B43590	$MeCl_2$	350			

Reviewed by/Date:

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-302
Revision History
Cover Page
Page 1

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Prepared By: Peter Lemay Date: 7/96

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Buxton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Dennis P. Hufnagle Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 8081A	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1.	DN	1·22·01	1/22/01
03 8081A	Changes to comply with South Carolina requirements – added linear calibration option, retention time window criteria & other minor changes to surrogate criteria.	DN	5·21·01	5·21·01
04 8081A	Changed to practice of reporting higher value. Other minor changes to Table 1 + 2, section 7.5.3 and section 7.4.3.	DN	5·21·02	5·21·02
05 8081A	Added definitions and information for the new data processing system. Replaced several figures with updated ones.	MRC	05.04.04	05.04.04
06 8081A	added alternative CV Conc. changed data checklist minor changes throughout added wording to section 8	LAD	03/05 LAD	03/05 6-13-06

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added retention time window criteria. Sect. 7.4.2 - added to shake samples before vialing.	LAD	03/06	03/06
08	Sect. 1.4 - Updated to include waste streams. Sect. 4.5 - removed balance Changed makeup gas from N2 to Ar Me. Changed T.C. from 5 to 2 peaks. Updated column confirmation. Changed corr. coef. to coeff. of determination	LAD	06/07	06/07
09	Added extraction method 3535 for AQ samples updated method references. Added Katahdin Analytical Environmental Health and Safety Manual. Changed number of peaks quantitated for Toxaphene to 3 or 5. Added S.C. clarification on marginal exceedences.	LAD	02/09	02/09
10	Changes made to sections 4.1, 7.4, 7.5, 8.0 and 10.0 for compliance with DOD QSM version 4.1	LAD	08/09	08/09

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

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-302-09**, titled **ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for Pesticides by EPA Method 8081B, as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, chlordane, 4,4'-DDT, 4,4-DDE, 4,4'-DDD, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide, toxaphene, endrin ketone, and methoxychlor. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD).

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. For aqueous samples, laboratory reagent grade water is used as a blank matrix; however a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

INDEPENDENT CALIBRATION VERIFICATION (ICV): A verification of the ratio of instrument response to analyte amount. ICV solutions are prepared from stock solutions which are independent from the stock solutions used to prepare the calibration standards.

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.

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.

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 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.

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.

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.

HP ENVIROQUANT: A data acquisition system that is used to collect chromatographic data.

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.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pesticides by method 8081, current revision. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8081, current revision, 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 Health and 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.

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.

Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the Organic Vial Waste (P).

2.0 SUMMARY OF METHOD

2.1 Method 8081 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2-5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8081 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8081 may also be performed on samples that have undergone cleanup. Method 3660, Sulfur Cleanup, by itself or in conjunction with Method 3620, Florisil Column Cleanup, may also be used to eliminate interferences in the analysis.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a problem in pesticide determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 6890 or 5890 series I or II or 6890 connected to the Turbochrom or Enviroquant data system,or equivalent.

4.1.2 Columns: Instruments are configured with a pre-column originating from the injection port which is connected to deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD).

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in a separate logbook.

5.2.3 Pesticide Working standards: Prepared by diluting the stock mix of 2000 ug/ml that contains all single component pesticides into hexane to give final concentrations of: 0.005, 0.01, 0.025, 0.05, 0.10, and 0.25 ug/ml. The mix, referred to as INDAB, also contains two surrogates: Tetrachloro-m-xylene and Decachlorobiphenyl, which are at the same concentrations as the pesticides.

5.2.4 Independent Calibration Verification Standard: Prepared as above using a standard independent of the calibration standards.

5.2.5 Multicomponent Pesticide Working standards: Toxaphene is prepared by diluting the Toxaphene stock solution to a concentration of 1.0 ug/ml. Technical chlordane is prepared similarly except to a concentration of 0.50 ug/ml.

5.2.6 Evaluation Mix: Prepared by diluting the stock solution to a concentration of 0.20 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURES

EXTRACTION - Refer to the appropriate SOP for the correct extraction procedure. In general, water samples are extracted using methods 3510, 3520 or 3535 while solid samples use methods 3540, 3545 or 3550.

7.1 INSTRUMENT CONDITIONS

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:	Makeup flow:	60 ml/min Nitrogen or Ar/Methane
	Column flow:	3.75 ml/min
	Injector Temp:	200
	Detector Temp:	300
	Oven Ramp:	160(0) - 5/min - 260(10)
	Run time:	24 min
	Injection size:	2 uL

7.2 CALIBRATION

7.2.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard mix of the INDAB mix listed in Reagents Section 5.2.2 is prepared along with a single point standard of Toxaphene and Technical Chlordane.

If the sample contains Toxaphene, a six-point calibration curve is analyzed. If the sample contains Chlordane and the analysis request is for Technical Chlordane, a six-point calibration curve is analyzed. If the analytical request is for the two components alpha-Chlordane and gamma-Chlordane, these two compounds are quantitated from the INDAB mix.

Toxaphene is calibrated using the 5 to 10 major peaks of the standard. The Target system will calculate a peak height for all 5 to 10 peaks. A calibration curve is prepared in Target using the peak heights of the 5 to 10 peaks against the concentration of the standard.

Technical Chlordane is calibrated using 3 to 5 major peaks of the standard. The Target system will calculate a peak height for all three to five peaks. A separate calibration curve for each of the 3 to 5 peaks is prepared in Target using the peak height against the concentration of the standard.

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 for each compound. A calibration curve can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = The intercept

Please note that a non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used. The linear equation is

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = The intercept

The calibration curve is calibrated the same way as the second order polynomial equation except that a five-point calibration standard mix is used.

7.2.2 The INDAB mix calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.2.3 The working calibration curve must be verified on each 12-hour shift that samples are to be analyzed by injecting the mid-point calibration standard.

7.3 RETENTION TIME WINDOWS

7.3.1 Three injections of all single component standard mixtures and multiresponsive products throughout the course of a 72-hour period.

7.3.2 The standard deviation of the three retention times is calculated for each single component standard. For multiresponsive products, a major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.3.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. For multiresponsive analytes, the analyst should use the retention time window, but should primarily rely on pattern recognition.

7.3.4 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.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.4 GAS CHROMATOGRAPHIC ANALYSIS

7.4.1 Before calibration is performed, and at the beginning of each 12 hour shift, the system is evaluated for analyte degradation by the analysis of a standard mix containing only endrin and 4,4'-DDT, often called an evaluation mix (EVAL):

COMPOUND	CONCENTRATION
Endrin	0.20 ng/uL
DDT	0.20 ng/uL

The % breakdown of DDT and the % breakdown of Endrin is calculated using the following formulas (PH = Peak Height):

$$\% \text{ Breakdown DDT} = \frac{(PH\,[DDD] + PH\,[DDE])}{(PH\,[DDD] + PH\,[DDE] + PH\,[DDT])} * 100$$

$$\% \text{ Breakdown Endrin} = \frac{(PH\,[\text{Endrin Aldehyde}] + PH\,[\text{Endrin Ketone}])}{(PH \text{ of } [\text{Endrin Aldehyde}] + PH \text{ of } [\text{Endrin Ketone}] + PH \text{ of } [\text{Endrin}]} * 100$$

The breakdown of either DDT or Endrin in the evaluation mix cannot exceed 15%. If there is breakdown of either compound exceeding 15% before starting a calibration, instrument maintenance must be performed. A calibration can not be run until the evaluation mix meets the acceptance criteria. If the exceeding breakdown occurs during the analysis sequence, then any samples analyzed after a failing evaluation mix must be reanalyzed. Reanalysis can not resume until after an acceptable evaluation mix.

7.4.2 Gently shake sample extracts before vialing for analysis.

7.4.3 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 uL injection volumes.

7.4.4 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.2 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 0.05ppm concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 15%. DoD allows a difference of ± 20%. Each sample analysis must be bracketed with an acceptable initial calibration and closing CV or an opening CV and a closing CV for each 12-hour shift. The closing CV standard is at 0.25ppm. The calibration standard must also be injected at intervals of not less than once every ten samples 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 <u>after</u> a group of samples exhibits a response for an analyte that is <u>above</u> the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts 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 <u>was</u> detected in a sample extract, 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 15% 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.

7.4.5 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.4.6 The identification of Pesticides 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 absolute retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.4.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.4.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a florisil cleanup (method 3620) and/or a sulfur cleanup (method 3660). Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.4.9 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 coefficient of determination criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, replacing the Y connector, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook. Refer to Katahdin SOP CA-101, Equipment Maintenance.

7.4.10 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibrated method.

7.4.11 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.4.11.1 Water: Concentration (ug/L) = (C) (Vt)/(Vs)

7.4.11.2 Soil / Sediment:Concentration (mg/kg) = (C) (Vt)/(Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.5 Data Review

7.5.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.7.

7.5.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the Laboratory Surrogate Acceptance Limit sheet. For DoD work, the surrogates must meet the acceptance limits in the DoD QSM.

The sample is evaluated for recoveries of the two surrogates. The recoveries of both surrogates are evaluated on both the primary and secondary column. The higher recovery from both columns is reported on the analytical report for both surrogates. The sample chromatogram is reviewed for any

interferences before determining whether to accept a sample based on the surrogate recoveries. If the surrogate recovery is affected by matrix interference, the sample result may be accepted with narration. If the recovery of one surrogate is outside of the laboratory established acceptance limit on one or both columns, and the second is acceptable, the data is narrated. If the recoveries for both surrogates are not acceptable because the recoveries are high and the sample does not contain any analytes above the PQL, the data is narrated. If the recoveries for both surrogates are low and there is no apparent matrix effect, the sample is reextracted.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

7.5.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.4.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates

the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.5.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered to be present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged indicating that the result is an estimated value. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

All flagged data must be discussed in the narrative

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV.

The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

7.6 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 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 (Figure 2) 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, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked with the twenty single component pesticides at the same concentration. The spike concentrations are:

	WATER ug/L	SOILS ug/Kg
Pesticides	0.50	16.7

The surrogate spike concentrations in the final extract are:

	WATER ug/L	SOILS ug/Kg
Tetrachloro-m-xylene(TCX)	1.0	33.3
DCB	1.0	33.3

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC 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 DoD work, the recoveries are compared to DoD QSM acceptance limits.

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

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.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

Please note that established acceptance limits that are wider than 70-130% may not be allowable for certain states, federal programs, or clients. For South Carolina, the acceptance limits for the spiked analytes will be 70-130% or narrower.

DoD work requires Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and 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 8081 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update IV, dated February, 2007, Method 8081B.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis.

Katahdin SOP CA-101, Equipment Maintenance

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF FIGURES

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TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of twenty or fewer samples	No analyte detected >PQL DoD: no analyte detected >1/2 PQL and >1/10 the amount measured in sample	(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 prep batch of twenty or fewer samples	Statistically derived limits. Note that limits wider than 70-130% are not allowable for some states, programs or clients, i.e. South Carolina. See also section 8.4 of this SOP for more information on allowable exceedances DoD: Use DoD QSM acceptance limits.	(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 of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are 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.
CCV	If calibration curve previously analyzed, analyze daily before samples and after every 10 samples.	± 15% D DoD: 20%D	(1) Evaluate the samples: If the %D>+15% and sample results are <PQL, narrate. If %D>±15% only on one channel, narrate. If %D>±15% for the closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
6 pt of INDAB mix with mid-pt cal of Toxaphene and Chlordane	Initial cal prior to sample analysis	6pt calibration coefficient of determination ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Toxaphene, or Chlordane is identified in analysis of sample, 6 pt calibration run of identified compound with reanalysis of sample.
Independent calibration verification	Once after Initial calibration	± 20 % D	Reanalyze standard Reprep standard Reprep standard from fresh stock.
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	One time per analyst initially 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
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 PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-302-10	METHOD 8081, current revision
Apparatus/ Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	7.6.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
QC - Continuing Calibration	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 PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
Instrument: GC08
Amount Injected 2ul

Reviewed by/ Date:

GC Laboratory Instrument Runlog

Method: 608 / 8081 / 8082
(circle)

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
6.13.07	SJC	8AF½ 154	WG39913-1 3510	Y	PSTA/B274A	293/294	
		155	-2				
		156	-3				
		157	-4				TCLP Blank PCB on Both
		158	SA2894-1 3550				
		159	-2				
		160	SA2750-1 3510				
		161	Hexane	N			
		162	INDAB 0.025ppm	Y			P4228
		163	SA2799-1 3510	Y			
		164	SA2883-2 3550				
		165	SA2753-1 3510				
		166	-2				PCB on B
		167	SA2799-2 3550	N			needs SC
		168	-3				
		169	-4				
		170	-5				
		171	-6	Y			
		172	-1				
		173	Hexane	N			
		174	INDAB 0.05ppm	Y			most low P4229
6.18.07		175	Hexane	N			
		176	Prime				
		177	EWL	Y			P4188
		178	INDAB 0.05ppm	N			most low P4229
		178A	INDAB 0.05ppm				
		179	0.005ppm	Y	PSTA/B275A		P4226
		180	0.01				P4227
		181	0.025				P4228
		182	0.05				P4229

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 2

DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Sample Data Summary Included (Level III & IV). _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration **(Narrate as needed).** _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

_____

First correction ☐ → Review and replace appropriate SDS Forms ☐.

Second correction ☐ → Review and replace appropriate SDS Forms ☐.

Last saved by kasgc04/13/07

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 3

PQLS FOR METHOD 8081

Parameter/Method	Analyte	Practical Quantitation Level (PQL)	
		Waters (ug/L)	Soils (ug/kg)
Organochlorine	Aldrin	0.05	1.7
Pesticides	Alpha BHC	0.05	1.7
	Beta BHC	0.05	1.7
SW3510/SW8081A (W)	Delta BHC	0.05	1.7
SW3520/SW8081A (W)	Gamma BHC (Lindane)	0.05	1.7
SW3550/SW8081A(S)	Chlordane	0.50	17
	alpha-Chlordane	0.05	1.7
	gamma-Chlordane	0.05	1.7
	4,4'-DDD	0.10	3.3
	4,4'-DDE	0.10	3.3
	4,4'-DDT	0.10	3.3
	Dieldrin	0.10	3.3
	Endosulfan I	0.05	1.7
	Endosulfan II	0.10	3.3
	Endosulfan Sulfate	0.10	3.3
	Endrin	0.10	3.3
	Endrin Aldehyde	0.10	3.3
	Endrin Ketone	0.10	3.3
	Heptachlor	0.05	1.7
	Heptachlor Epoxide	0.05	1.7
	Methoxychlor	0.50	17
	Toxaphene	1.00	33

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-334
Revision History
Cover Page
Page 1

TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Leigh Schwieterman Date: 08/04

Approved By:

Group Supervisor: Peter [illegible] Date: 8-12-04

Operations Manager: Deborah J. Nadeau Date: 8·12·04

QA Officer: Maria Crouch Date: 08.12.04

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Deleted PCB's 49, 87, 183 & 184 Changed Turbochrom to ChemStation added PCB103 and PCB 192 Surrogates. High Pt. Conc. 0.5 → 0.25, changed GC Conditions added wording to Sect. 8 minor changes throughout	LAD	013105	013105
02	Changed list of analytes from NOAA/WHO to 8082 & dredge lists. Added GPC to Sect. 2.2. Replaced lab established limits to nominal limits. Changed congeners used in LCS/MS spike. Updated Figure 3 w/ appropriate congeners	LAD	04/06	04/06
03	added injection port information, added specific waste description, added manual integration notation, removed balance from 4.5, added independent standard to calibration information; added to shake sample extracts before vialing, changed target compound detection, added LCS DOD QSM	LAD	02/08	02/08
04	V=Changes made to sections 4.0, 7.2, 7.3, 7.5, 7.7, 8.0, 10.0 and Table 1 to reflect DOD QSM compliance. Updated ruler example.	LAD	08/09	08/09

TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-334-04, titled "Analysis of PcbBs as Congeners by Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-334-04, titled "Analysis of PcbBs as Congeners by Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082".

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TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for individual PCB Congeners by EPA Method 8082 as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: PCB 1, PCB 5, PCB 8, PCB 18, PCB 28, PCB 31, PCB 44, PCB 49, PCB 52, PCB 66, PCB 87, PCB 101, PCB 105, PCB 110, PCB 118, PCB 128, PCB 138, PCB 141, PCB 151, PCB 153, PCB 170, PCB 180, PCB 183, PCB 184, PCB 187, PCB 195, PCB 206, and PCB 209. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD)

Additional PCB Congeners may also be analyzed for and quantitated using this method as part of a client's special request.

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. 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.

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.

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 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.

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 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.

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.

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.

HP ENVIROQUANT: A data acquisition system that is used to collect chromatographic data.

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.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of individual PCB Congeners by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

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 safety procedures and 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 a respirator and 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 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 waste generated during the preparation of standards should be disposed of in the Mixed Flammable Waste (O) waste stream satellite accumulation area nearest the point of generation. After the extracts have been analyzed, the autosampler vials and any expired standard vial or ampules are disposed of in the Organic Vial Waste (P). Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain individual PCB Congeners. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5µl aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup. Soil extract are also cleaned up by GPC (mandatory), solvent-exchanged into hexane, and adjusted to a final volume of 1 ml.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in individual PCB Congeners determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 6890 with split/splitless injection port with EPC connected to the Enviroquant data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects

two different columns to two detectors. The most commonly used columns are: RTX-5MS 60M x 0.25 mm ID or RTX-CLPesticides II 60M x 0.25 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron Capture Detectors (ECD).

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentrations of the working individual PCB Congener calibration standards are 0.005µg/ml, 0.01µg/ml, 0.025µg/ml, 0.05µg/ml, 0.1µg/ml, and 0.25µg/ml. The standards also contain the surrogates Tetrachloro-m-xylene (TCX), PCB 103 and PCB 192 at the same concentrations: 0.005µg/ml, 0.01µg/ml, 0.025µg/ml, 0.05µg/ml, 0.1µg/ml, and 0.25µg/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in glass 1-Liter containers for aqueous samples and 8 ounce soil jars for solid samples and stored at 4 °C ± 2 °C until extraction. Aqueous samples must be extracted within 7 days of sample collection and solid samples within 14 days of sample collection. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs (CA-500, CA-515, CA-524, and CA-536) for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 while solid samples use methods 3540, 3545, or 3550.

The extracts are concentrated and consequently cleaned up using Gel Permeation Chromatography (GPC). Aqueous samples are brought to a final volume of 0.5 mL. Soil samples should be brought to a final volume of 1 mL Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

- Makeup flow: 60 ml/min Argon/Methane or Helium
- Column flow: 0.8ml/min
- Injector Temp: 200°
- Detector Temp: 325°
- Oven Ramp: 170° → 10°/min - 270°(2) → 2°/min - 300°(6)
- Run time: 33 min
- Injection size: 2µl

7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard of the 28 individual PCB Congeners plus the surrogates, TCX, PCB 103 and PCB 192 is used.

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 for each compound. A calibration curve can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where:
y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = The intercept

Please note that a non-linear calibration model may not be allowable for certain stated, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. The linear equation is:

$$y = bx + c$$

where:
y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = The intercept

7.3.2 The calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed

7.3.3 The working calibration curve must be verified on each 12 hour shift that samples are to be analyzed by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 15%, a new calibration curve must be prepared for that analyte. DoD allows a difference of ± 20%.

7.4 Retention time windows

7.4.1 Three injections are made of all the individual PCB Congeners throughout the course of a 72 hour period.

7.4.2 The standard deviation of the three retention times is calculated for each individual PCB Congener.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms.

7.4.4 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.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 Gas chromatographic analysis

7.5.1 Gently shake sample extracts before vialing for analysis.

7.5.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5µl injection volumes.

7.5.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a mid-concentration standard (CV-calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 15%. DoD allows a difference of ± 20%. Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and an ending CV for each 12-hour shift. The calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended) 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. Another CV is analyzed or the instrument is recalibrated and then samples are injected. To avoid errors in quantitation, all samples that were injected after the standard exceeding the criterion must be reinjected, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.

The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.5.4 The identification of individual PCB Congeners is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative analyte identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.5.5 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.5.6 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665). Note: Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. All soil samples should undergo a GPC cleanup prior to analysis (Refer to section 7.1). This cleanup technique should eliminate most interferences. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.5.7 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, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook. Refer to Katahdin SOP CA-101, Equipment Maintenance.

7.6 Calculations

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibrated method.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.6.2.1 Water:

$$\text{Concentration } (\mu g/L) = (C)\,(V_t)/(V_s)$$

7.6.2.2 Soil/Sediment:

$$\text{Concentration } (mg/kg) = (C)\,(V_t)/(W_s)\,(D)$$

Where:
C = concentration calculated by Target in µg/ml
V_t = Volume of total extract including any instrument dilutions
V_s = Volume of sample extracted
W_s = Weight of sample extracted
D = Decimal total solids

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives and dilutions.

The requirement of the Organics laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

All recoveries must meet the nominal acceptance limits of 50-150%. When enough data points have been collected, laboratory acceptance limits will be

established. The most current limits will be listed on the GC Laboratory Surrogate Acceptance Limit sheet. For DoD work, the surrogates must meet the acceptance limits in the DoD QSM.

The sample is evaluated for the recovery of the surrogate. If the surrogate recovery is high and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recovery is low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recovery is low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.6.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates the reason for the manual integration. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, Manual Integration, current revision.

7.7.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgement, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration

range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered to be present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged indicating that the result is an estimated value.. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

All flagged data must be discussed in the narrative.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, or if an analyte is present but its retention time is ± 0.04 minutes or more than the retention time of the analytes in the preceding CV.

The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

7.8 Reporting

7.8.1 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 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 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 the entire QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked at the same concentration with a second source standard different from the calibration standard. A mix containing 19 of the individual PCB Congeners at 0.1ug/mL will be used for spiking. The spike concentrations in the final extract are:

Congener	Water µg/L	Soil µg/kg
1	0.1	6.67
5	0.1	6.67
18	0.1	6.67
31	0.1	6.67
44	0.1	6.67
52	0.1	6.67
66	0.1	6.67
87	0.1	6.67
101	0.1	6.67
110	0.1	6.67
138	0.1	6.67
141	0.1	6.67
151	0.1	6.67

Congener	Water µg/L	Soil µg/kg
153	0.1	6.67
170	0.1	6.67
180	0.1	6.67
183	0.1	6.67
187	0.1	6.67
206	0.1	6.67

The surrogate spiking solution is at a concentration of 0.2ug/mL. The surrogate spike concentrations in the final extract are:

	WATER µg/L	SOILS µg/kg
Tetrachloro-m-xylene (TCX)	0.2	13.3
PCB 103	0.2	13.3
PCB 192	0.2	13.3

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for individual PCB Congeners are established for both water and soil matrices. The MS/MSD acceptance limits for individual PCB Congeners still use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points can not be used (i.e. recoveries not calculable due to a matrix effect). Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For DoD work, the recoveries are compared to DoD QSM acceptance limits if available, otherwise use laboratory established acceptance limits.

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.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

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.

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.

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.

DoD work requires Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the nominal acceptance limit window, or laboratory established acceptance limit window when established, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible to document resolution.

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 are determined annually per type of instrument and 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 further details.

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Refer to the current revision of Method 8082 for other method performance parameters and Requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update III, dated December, 1996.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis.

Katahdin SOP CA-101, Equipment Maintenance

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL DoD: no analyte detected >1/2 PQL and >1/10 the amount measured in sample	(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 prep batch of twenty or fewer samples	50-150% Statistically derived limits when enough data points have been analyzed for limits to be established DoD: Use DoD QSM acceptance limits.	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.
CV	After every 10 samples or 12 hours, whichever is first: If calibration curve previously analyzed, analyze daily before samples.	± 15% D DoD: 20%D	Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.
Sample Duplicate	One sample duplicate per ten samples if requested	RPD ≤20 DoD: RPD ≤30	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Else, reanalyze
6pt calibration of all 28 individual PCB Congeners	Initial cal prior to sample analysis	6 pt calibration - correlation coefficient ≥ 0.990	(1) Repeat Initial calibration
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin Sop Ca-334-04	Method 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of … minutes. 7.3.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard of all 28 congeners is used. 7.5.3 Absolute retention time windows are established every 12 hour shift for each analyte 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.	7.6.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000). 5.7.1 The calibration contains only the 19 congeners listed in the Table of section 1.1. 7.6.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift.
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
Reagents		
Sample preserva-tion/ handling		
QC		
Apparatus/Materials		

TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
Instrument: GC01
Amount Injected 2ul

Reviewed by/ Date:

GC Laboratory Instrument Runlog
Method: SW846 8081 / 8082 / 8151
(circle) EPA 504.1 / 556 / 608
OLC03.2 / SOM01.2
OLM03.2 / OLM04.3

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
		New	twice				
5-4-09	JLP	CE00080	CON 0.05	Y	CON 002	327/328	PS179
		81	SC1543-2DL 3550	Y			1:10 10/100
		82	SC1572-1DL	Y			1:10
		83	SC1639-1DL	Y			1:10
		84	SC1543-5DL 3510	Y			1:10
		85	-7DL4				1:10
		86	-9DL4				1:10
		87	SC1490-3DL4				1:10
		88	SC1521-3	Y			3531A
		89	W662547-4 3550				
		90	-5				
		91	Hexane	N			
		92	CON 0.025				PS178
		New	twice				
5-4-09	JLP	CE00093	CON 0.05	N	CON 002	327/328	PS179 Mod
		094	CON 0.05	Y	CON 003		PS179
		095	0.005				PS176
		096	0.01				PS177
		097	0.025				PS178
		098	0.1				PS180
		099	0.25				PS181
		100	IND 0.05	Y			PS182
		101	SC1827-1RA 3510	Y			553 both
		102	SC1543-7DL5	Y			1:100 10/1000
		103	-9DL5	Y			1:500 10/1000 100/500
		104	SC1490-1DL2 3550	Y			1:500
		105	SC1521-1DL2				1:500
		106	SC1543-1DL2				1:500
		107	-2DL2				1:500

TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Sample Data Summary Included (Level III & IV). _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration (Narrate as needed). _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

_____

First correction ☐ → Review and replace appropriate SDS Forms ☐.

Second correction ☐ → Review and replace appropriate SDS Forms ☐.

Last saved by kasgc04/13/07

TITLE: ANALYSIS OF PCBs AS CONGENERS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLS FOR METHOD 8082

ANALYTE	8082 LIST (8) DREDGE LIST (D) NOAA LIST (N)	PQL (µg/L)	PQL (µg/kg)
PCB 1 - 2-chlorobiphenyl	8	0.01	0.66
PCB 5 – 2,3-dichlorobiphenyl	8	0.01	0.66
PCB 8 – 2,4'-dichlorobiphenyl	N	0.01	0.66
PCB 18 – 2,2',5-trichlorobiphenyl	8 N	0.01	0.66
PCB 28 – 2,4,4'-trichlorobiphenyl	N	0.01	0.66
PCB 31 – 2,4',5-trichlorobiphenyl	8	0.01	0.66
PCB 44 – 2,2',3,5'-tetrachlorobiphenyl	8 N	0.01	0.66
PCB 49 – 2,2',4',5-tetrachlorobiphenyl	D	0.01	0.66
PCB 52 - 2,2',5,5'-tetrachlorobiphenyl	8 N	0.01	0.66
PCB 66 - 2,3',4,4'-tetrachlorobiphenyl	8 N	0.01	0.66
PCB 87 – 2,2',3,4,5'-pentachlorobiphenyl	8 D	0.01	0.66
PCB 101 - 2,2',4,5,5'-pentachlorobiphenyl	8 N	0.01	0.66
PCB 105 - 2,3,3',4,4'-pentachlorobiphenyl	N	0.01	0.66
PCB 110 - 2,3,3',4',6-pentachlorobiphenyl	8 N	0.01	0.66
PCB 118 - 2,3',4,4',5-pentachlorobiphenyl	N	0.01	0.66
PCB 128 - 2,2',3,3',4,4'-hexachlorobiphenyl	N	0.01	0.66
PCB 138 - 2,2',3,4,4',5'-hexachlorobiphenyl	8 N	0.01	0.66
PCB 141 - 2,2',3,4,5,5'-hexachlorobiphenyl	8	0.01	0.66
PCB 151 - 2,2',3,5,5',6-hexachlorobiphenyl	8	0.01	0.66
PCB-153 - 2,2',4,4',5,5'-hexachlorobiphenyl	8 N	0.01	0.66
PCB 170 - 2,2',3,3',4,4',5-heptachlorobiphenyl	8 N	0.01	0.66
PCB 180 - 2,2',3,4,4',5,5'-heptachlorobiphenyl	8 N	0.01	0.66
PCB 183 - 2,2',3,4,4',5',6-heptachlorobiphenyl	8 D	0.01	0.66
PCB 184 -2,2',3,4,4',6,6'-heptachlorobiphenyl	D	0.01	0.66
PCB 187 - 2,2',3,4',5,5',6-heptachlorobiphenyl	8 N	0.01	0.66
PCB 195 - 2,2',3,3',4,4',5,6-octachlorobiphenyl	N	0.01	0.66
PCB 206 - 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	N	0.01	0.66
PCB 209 - 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	8	0.01	0.66

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [illegible] Date: 10/2 5/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Duncan F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·23·00	10/23/00
02	removed references to medium level extraction. New logbook figures minor changes through out	LAD	020305 ~~02~~ LAD 020305	020305
03	updated compound List changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated Solvent exchange, updated Table 1, replaced fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07
05	Updated LB example. Added temp. of nitrogen water bath, lot numbers of filter paper, lot #'s of acids need to be recorded in LB. Change "N-Lo" waste to "K" waste.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added requirement to add spike before Na_2SO_4. Changed N_2 waterbath temperature from < 37°C to < 30°C. Removed respirator references. Added KAEHS manual. Added CA-108 reference for subsampling	LAP	02/09	02/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs 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-500-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-500-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis in accordance with SW-846 Method 3550, current revision..

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should

also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

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 hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.6 Sonic disruptor – Misonix XL2015 (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.

4.7 Spatula - stainless steel

4.8 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.13 Balance – top-loading, capable of weighing to 0.1 g

4.14 Nitrogen evaporation apparatus

5.0 REAGENTS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory. Solvent rinse immediately before use by rinsing three times with pesticide grade methylene chloride.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, purified as described in section 5.1.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC/MS analysis.

5.4 Acetone and hexane - pesticide grade or equivalent

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 Pesticide Matrix spike/Lab control sample spiking solution - Prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 PCB Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260 (1660), both at 5.0 ug/mL.

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

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

LOW LEVEL EXTRACTION OF SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of 30.0 ± 0.1 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

7.1 Discard any excess water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. **Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on subsampling to ensure reproducibility.**

7.2 Weigh out a 30.0 ± 0.1 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.1 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.)

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.1 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. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.1 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 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. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.7 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero, and turn Pulsar Duty Cycle to off (or continuous mode).

7.8.2 Press POWER SWITCH to ON (up) position. Engage the Timer Switch (W-375)

7.8.3 Press and hold down the TUNE switch.

7.8.4 Turn the Output Control Knob towards setting 10. Note the position of the needle on the % output power meter. **DO NOT exceed 70%. If you reach 70% - STOP!!** Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8.5 Turn the Output Control Knob towards setting 10. Again, note the position of the needle and do not exceed 70%. Rotate the Tuning Knob until you obtain a minimum reading of 20% or below.

7.8.6 Release the TUNE switch. **CAUTION: Do NOT touch probe. The probe is still active.**

7.8.7 Turn OUTPUT CONTROL KNOB counter-clockwise to zero (or disengage timer).

7.8.8 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

Note: If the unit will not be used immediately, please turn the power switch to off.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone solution and proceed with steps 7.11 through 7.14.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. 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.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinse flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. 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 1:1 methylene chloride:acetone to complete the quantitative transfer of the extract.

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 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. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.16 For a solvent exchange, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

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 7.23 to concentrate extracts to final volume of 10.0 mLs. 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. 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 10 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than 30° 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. Note any problems or extract losses, if they occur, in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.23 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.24 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.25 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

Note: The lot number of the acid used in PCB cleanup must be recorded in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

8.2 A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Final Update IV, February 2007.

Table 1 Summary of Method Modifications
Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-06	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

P/P SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	CLP OLM04.X
	SW846 3580 (OILS/WIPES)	OTHER	
Standards	Surrogate ID: 6C0495	Spike ID: 6C0497 Pest	Spike ID: 6C0496 PCB
Solvents	Solvent Lot # (Mecl2): 61E09	Solvent Lot # (Acetone): E27E06	Solvent Lot # (Hexane):
Consumables	Filter Paper Lot # (SON) 61119160	Filter Paper Lot # (KD) H11375078	Acid Lot # 072651
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: < 20%	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
4-26-08	Km	W650766-1 W650767-1	30.01	1mL	NA	✓	✓	10mL	4-26-08	P1777 F2	Km			✓		
↓	↓	W650766-2	30.04	↓	1mL	✓		↓	↓	F3	↓					Pest K79946
		↓ -3	30.00		↓	✓				F4						
		W650767-2	29.98		1mL		✓			F5				✓		PCBK79947
		↓ -3	30.21	↓	↓		✓	↓	↓	F6	↓			✓		

Km 4/26/08

QAEX146 0000053

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
4-26-08	Km	SB7131-1	29.97	1mL	NA	✓	✓	10mL	4-26-08	F7	Km			✓		
↓	↓	↓ -2	30.03	↓	↓	✓	✓	↓	↓	F8	↓			✓		
↓	↓	↓ -3	29.99	↓	↓	✓	✓	↓	↓	F9	↓			✓		

Km 4-26-08

QAEX146 0000054

viewed By_____ Date_____

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. Radeau 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-108. Updated logbook example. Added if extract goes dry - re extract.	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

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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-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____Date:_____

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 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 CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. 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.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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.

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

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 - granular. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before each use. Cool in a desiccator and store in a glass bottle with a Teflon-lined cap.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) - slowly add 500 mL of H_2SO_4 (sp gr 1.84) to 500 mL reagent water.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

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. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - 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	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

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 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	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.

5.5.3 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.

5.5.4 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 3 at a concentration of 2 ug/mL for base/neutral. 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.

5.5.5 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.

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 appicable
- 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.

The internal chain-of-custody must be signed when removing and replacing samples in storage locations.

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). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.1.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies 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.

7.1.6 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 $\leq$ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.1.7 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.8 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) 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.

7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).

7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).

7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS 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.

7.1.10.1 If the request is for SVOA -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.11 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.

7.1.12 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.

7.1.13 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.

7.1.14 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.

7.1.15 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.

7.1.16 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the "N-Hi" satellite.

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 should 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. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This 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 base/neutral and acid (SVOA) 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.

7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.

7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.

7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS 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 the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, 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 $\geq$ 11 with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be $\geq$ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.10 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.11 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. Following each shake, allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into an amber collection bottle.

7.2.12 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.

7.2.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.

7.2.14 Repeat the extraction for a third time as described in 7.2.14.

7.2.15 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at $\geq$ 11. If the pH has changed back to neutral range, it must be readjusted to $\geq$ 11 and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value $\geq$ 11, the pH is then adjusted to $\leq$ 2 with 1:1 H_2SO_4. Add enough 1:1 H_2SO_4 to adjust the pH to $\leq$ 2 with stirring. Use glass stirring rods to stir.

7.2.16 Add 60 mL methylene chloride and extract the samples three times in the same manner described in 7.2.11 – 7.2.13. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.17 Sample waste should be poured into the "n-lo" sattalite.

7.2.18 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.1 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.

7.3.2 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 fluted 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

7.3.3 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.

7.3.4 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.

7.3.5 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.

7.3.6 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.

7.3.7 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.8 Using methylene chloride for a quantitative transfer, adjust the final volume of each extract to 1 mL. Use the 1 mL oil-filled reference vial for volume comparison.

7.3.9 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, Methods 3510 and 3520 (current revisions), SW-846 Third Edition, Updates I, II, IIA, and IIB, Revised January 1995, US EPA.

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-06	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-502-06	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 ± 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

SVBLM
Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8270 ✓	EPA 625	CLP OLM04.2	CLP OLC02.1	CLP SOM01.2	
Standards	Surrogate ID (1): SV2350		Spike ID (1): SV852		Spike ID (3):	
	Surrogate ID (2): SV2349		Spike ID (2): SV2351			
Solvents/Acid/Base	Solvent Lot # (Mec12): H90631		H_2SO_4 # 635026		NaOH Lot # H22101	
Consumables	Filter Paper Lot # K11472365		$NaSO_4$ Lot # 27969003		Vial Lot # 00094428	
Nitrogen Water Bath Temperature 35°C			pH (1st Extraction) ≥11		pH (2nd Extraction) ≤2	
Prep Start Time: 0930	Prep End Time: 1230	CLLE Acid Start:	CLLE Acid End:		CLLE B/N Start:	CLLE B/N End:

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SW	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	W670483-1 W670483-1	1000	1mL each	NR	✓	✓	1mL	10/26/09	SV492 B8	CB	R_{spike} R112490
↓	↓	W670483-2	↓	1mL	1mL		↓	↓		B9	↓	R_{surr} R112487
		-3	980	↓	↓		↓			B10		W6SC6526-11k
		-4	1000				✓			B1		MSD -11L
		W670484-2	1000			✓				C2		
↓	↓	-3	↓	↓	↓	↓		↓	↓	C3	↓	
					CB	10/26/09						

0000089

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SW	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	SC6526-1a	1060	1mL	NR		✓	1mL	10/26/09	SV492 C4	CB	
↓	↓	-3c	1010	↓	↓		↓	↓	↓	C5	↓	
		-5d	1030							C6		
		-7c	1060							C7		less double sand.
		-9c	1030							C8		
		-11h	1030							C9		MSD
		-13c	1020							C10		
		-15d	1010							D1		
		-17c	1060							D2		
		-19d	↓							D3		
		-21c	↓							D4		
		-23c	1020							D5		
↓		-25c	1040				✓			D6		
		SC6525-2e	1060			✓				D7		
		-3c	↓			↓				D8		
		-4e	↓							D9		
		-5d	1040							D10		
↓	↓	-6d	1040	↓	↓	↓		↓	↓	E1	↓	

QAEX173

Reviewed By_____ Date_____

0000090

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

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	

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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: J C Burton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·24·00

General Manager: Dennis P. Lufgan 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-1008 reference for additional subsampling information.	LAD	02/09	02/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

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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 3550B 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 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

A 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 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.1 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

5.0 REAGENTS

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. Solvent rinse immediately prior to use by rinsing three times with pesticide

grade methylene chloride. (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 400 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 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - 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	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

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 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	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.

5.6 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.

5.7 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

5.8 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. 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

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeation chromatography (GPC). 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.

Sign chain-of-custody when removing and replacing samples in storage locations, and 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 Decant and discard any water layer on a sediment sample. Mix with a stainless steel spatula 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.

7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a 30.0 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Refer to 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. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 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 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 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.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 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.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.7 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.

7.6 Record all weights to one decimal place in the extraction logbook.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to 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.

7.7.1 If the request is for SVOA or SVOA Appendix IX, use the SVOA surrogate solution (sect. 5.4).

7.7.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.7.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

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 **prior** to 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 and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.8.4 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 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.9.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.9.2 Press and hold down the power switch to on.

7.9.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.9.4 Release the TUNE switch.

7.9.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.9.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

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 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 to loosen up the mixture prior to extracting. Rinse the spatula 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 with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. 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 ti the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using 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. 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 fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written ti 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 < 30°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

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

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.

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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 are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, 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, Method 3550C, USEPA SW-846, Third Edition, Update IV, February 2007.

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-07	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, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8270 ✓	SW846 3580 (OILS/WIPES)	OTHER
Standards	Surrogate ID (1): SV2239	Spike ID (1): SV2231	
	Surrogate ID (2):	Spike ID (2):	
Solvents	Solvent Lot # (MeCl2): G22E23	Solvent Lot # (Acetone): E46E42	
Consumables	Filter Paper Lot # (SON) M1119164	Filter Paper Lot # (KD) J1137505	
Misc.	Nitrogen Bath Temperature: 39°C	Sonicator Horns Tuned:	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	WG53548-1	30.01	1mL	NR	1mL	7-17-08	SV324 C4	KF	R53707
		-2	30.03		1mL			C5		
		-3	29.97					C6		
						KF	7-17-08			

QAEX147 0000067

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	SB3798-2	30.00	1mL	NR	1mL	7-17-08	SV324 C7	KF	
		SB3801-1	29.99			2mL		FP710 E3		Cloud chips
		-2	29.96					E4		
		-3	30.03					E5		Cloud chips
		SB3845-1	30.01			1mL		SV324 C8		Reg-SB3217-6
						KF	7-17-08			

Reviewed By_____ Date_____

QAEX147 0000068

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

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-515
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCBurton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Dennis P. Hulgan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Miner changes throughout. Clarifications to procedure section.	DN	10·23·00	
02	Addition of SPE Procedure. Minor changes through out. Added wording to Sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. Updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07
05	Updated logbook example. Added logbook requirements	LAD	09/08	09/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added information for determining initial volume. Added reference to CA-108. Added clarification for LCS/D and MS/D sets for PEST/PCB analysis. Minor changes to reflect current techniques	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs 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-515-05,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-515-05,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel, continuous liquid-liquid, and solid phase extraction methods (EPA Methods 3510, 3520, 3535A, and EPA Method 608 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 aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the 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 personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the

appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Supervisor to oversee that members of their group follow this SOP, 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 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 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste 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 etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the "N-Hi" or "N-low" satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest

the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "O" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel, continuous liquid-liquid apparatus or Automated Extractor System (SPE), following EPA Methods 3510, 3520, 3535A and EPA Method 608. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using all extraction methods, if the extraction lab wishes to use either or all techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which 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 which 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 the solvent to be used for extraction.

7.18 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

7.19 Concentrator tube - 10 mL, graduated

7.20 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

7.21 Snyder column - Kuderna-Danish, three ball macro

7.22 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

7.23 Short Stem Funnels

7.24 250 mL amber collection bottles with Teflon-lined caps

7.25 12 mL and/or 16 mL glass vials with Teflon-lined caps

7.26 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

7.27 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

7.28 Nitrogen evaporation apparatus.

7.29 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent). Cleaned by Soxhlet.

7.30 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

7.31 Vials, 60 mL with PTFE – lined screw caps.

7.32 Horizon SPE-DEX 4790 Automated Extractor System.

7.33 Atlantic DVB disks, or equivalent.

7.34 1-L amber bottles

5.0 REAGENTS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, "Baker-analyzed", or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.

5.4 Sulfuric Acid Solution (1:1) - Add 500 mL concentrated sulfuric acid (certified ASC grade or better) slowly to 500 mL laboratory reagent grade water. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4'-DDT	0.5
4,4'-DDD	0.5
4,4'-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5

ANALYTE (cont.)	ug/mL
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

1.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

Holding time for extraction of aqueous samples for Methods 3510, 3520, and 3535 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed might be project/program specific.

2.0 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 appicable
- 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.

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Rinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

4.3 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.

4.1 Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.1 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.2 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis. An LCS is required for every daily extraction batch of twenty or fewer samples and 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.

7.3 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples or every 14 days and each analysis(refer to the logbook page, "date QC expires"). Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

4.1 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

4.2 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.

4.3 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.

4.4 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD(s).

4.5 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.

4.6 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

4.7 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.10 - 7.12). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

4.8 Repeat the extraction for a third time as described in 7.13.

1.1 Proceed to Section 7.53 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.17 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

1.1 Add 2-3 boiling stones to the round bottom flask and approximately 500 - 600 mL of methylene chloride to the CLLE body.

1.2 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

4.1 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

4.2 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. 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. When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis.

4.3 Mark the sample levels on the sample bottles. Transfer the samples to the CLLE bodies.

4.4 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

4.5 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples, whichever occurs first. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

4.6 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

4.7 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. 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.

4.8 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

4.9 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

4.10 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 at least 18 hours, total extract time may go up to 20 hours. Turn off the mantles and let samples cool.

4.11 Proceed to Section 7. 53 for sample extract concentration procedures.

EXTRACTION WITH AUTOMATED EXTRACTOR SYSTEM (SPE)

Alternatively, samples may be extracted using the Horizon Automated Extractor System (Figure 2)

Purging the Extractor Vessels

4.1 Check and fill all necessary solvent bottles (acetone, laboratory reagent grade water and hexane) as needed. Check and empty the two waste containers as needed.

4.2 Turn on nitrogen tank to 60 psi. Turn the instrument pressure on top of the controller to 50 psi. Turn the solvent bottle pressure to 10 psi.

4.3 Turn on the Horizon controller (switch in the back).

4.4 Check the lubrication oil on the air pump. Fill as needed. Turn the air pump on.

4.5 Clean the glass sensors that are located on the back of the dispensing stems of the extractors using a Kim Wipe. This is to remove any residue that may interfere with the sensors.

4.6 Attach 19/22 adapters to 40-mL vials and attach beneath the disk holder platforms of the extractors. Assembly per owner's manual and place empty Horizon disk holder assemblies on top of the disk holder platforms. There should be roughly 1 cm separating the speedisk from the extractor downtube.

4.7 Check to be sure that all extractors have empty sample bottles loaded on top. If not, use a Horizon cap on a one liter empty bottle and firmly place the bottlenose down into the extractor.

4.8 Press *select* on the control panel to designate an extractor (1, 2, 3, 4 or "." for all), then press *enter*.

4.9 Type 8081.9, and press enter to select pesticide/PCB purge method. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete.

4.10 Repeat this process 2-3 times before using the Horizon autoextractors.

ANALYSIS OF SAMPLES WITH AUTOEXTRACTOR

4.1 Label a 60 ml vial with the sample to be extracted. Attach 19/22 adapter to vial and attach beneath the disk holder platforms of the extractors.

4.2 Place an Atlantic DVB disk (or equivalent) into a Horizon disk holder assembly and assemble per owner's manual. Place the disk holder assemble on top of the disk holder platform. There should be roughly 1 cm separating the speedisk from the extractor downtube

4.3 Mark the volume level of liquid in each sample on the outside using a grease pencil.

4.4 Add 1 L laboratory reagent grade water to 1 L amber bottle. This is the method blank for this batch. A method blank is required for every extraction batch of twenty or fewer samples.

4.5 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis, pesticide and/or PCB. Add 1 L of laboratory reagent grade water to a 1 L amber bottle. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required to meet client specific or program specific requirements. This information will be disseminated from the project manager or department manager.

4.6 Add 1.0 mL of Pesticide/PCB surrogate spike to each sample including the blank, LCS/LCSD and MS/MSD, if required. Recap samples and shake well.

4.7 Add 1.0 mL of pesticide or PCB matrix spike to the appropriate LCS/LCSD and MS/MSD samples. Recap and shake well.

4.8 Remove cap and add 5.0 mL of 1:1 H_2SO_4 to each sample including the blank, LCS/LCSD and MS/MSD set immediately prior to extracting the sample.

4.9 Remove the cap from each sample bottle and cover with tin foil. Screw a Horizon adapter cap over the tin foil. Invert the bottle and check for leaks.

4.10 Load the sample bottle on the holder and twist ¾ of a rotation. Stop twisting when air bubbles rise to the top of the sample bottle. Do not twist completely around. The foil may loosen and jam the valve.

4.11 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.

4.12 Type in 8081.3 for the method and press enter. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red

LED will blink when the method is complete. The extract will be collected in the 60 ml vial.

4.13 Sample is now ready to reduce to 10 mL final volume.

NOTE: The instrument methods stated above apply specifically to the Atlantic DVB disk. Instrument methods may need to be modified with the usage of different filters and/or to increase recoveries. See instrument logbook for current methods in use.

CONCENTRATION OF WATER SAMPLE EXTRACTS

4.1 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.

4.2 For methylene chloride extracts, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

4.3 Transfer the methylene chloride or hexane 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 bottle three times with ~ 2 – 3 mLs of methylene chloride(or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor) and allow to drain.

4.4 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) 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 (or hexane for samples extracted with the Autoextractor).

4.5 Place the K-D in a hot water bath (85-90°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 ≈ 5-6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of

hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

4.6 Reduce the extracts to ≈ 1 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). 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 needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

4.7 If at any point in the concentration procedure the concentrator tube goes dry – reextract the sample immediately.

4.8 Transfer the label from the concentrator tube to the vial. 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.

4.9 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. Therefore, all sample extracts for combined 8081/8082 analyses must be split. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

4.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency

must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

40 CFR 136, Appendix A, "Test Procedures for Analysis of Organic Pollutants," Method 608, June, 1998 edition.

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	2. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	3. extract collection in amber bottle or Erlenmeyer flask 4. extract dried using Na_2SO_4 in short stem funnels 5. no apparatus height specification for concentration on water bath 6. sample removed from water bath when volume reaches ~10 mL 7. hexane added directly to K-D body at start of concentration process	2. extract collection in Erlenmeyer flask 3. extract dried using Na_2SO_4 in drying columns 4. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 5. sample removed from water bath when volume reaches 1-2 mL 6. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 3

SUMMARY OF METHOD MODIFICATIONS (METHOD 3535, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	1. Horizon SPE-DEX 4790 Automated Extractor System.	1. Empore solid-phase extraction system
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. no methanol addition 2. extraction using Horizon SPE-DEX 4790 Automated Extractor System. 3. extract dried using Na_2SO_4 in short stem funnels 4. no apparatus height specification for concentration on water bath 5. sample removed from water bath when volume reaches ~10 mL 6. hexane added directly to K-D body at start of concentration process	1. 5mL methanol added to all samples and blanks 2. extraction using Empore solid-phase extraction system 3. extract dried using Na_2SO_4 in drying columns 4. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 5. sample removed from water bath when volume reaches 1mL 6. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PIP
Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	EPA 608	CLP OLM04.2	CLP OLC2.1	Other:
Standards	Surrogate ID: G606654		Spike ID: G606665		Spike ID: G606663	
Solvents	Solvent Lot # (Meci2): H40631		Solvent Lot # (Hexane): H22615		Solvent Lot # (Acetone): ——	
Consumables	Filter Paper Lot # K11672365		Acid Lot # G35026		Na_2SO_4 Lot # 27969003	
Nitrogen Bath Temperature: 37°C		Vial Lot #: 00097809 / 00098146				
Prep Start Time: 1400		Prep End Time: 1500		CLLE Start Time:		CLLE End Time:

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
10/22/09	OB	W470360-1 W470361-1	1000	1mL	NR	✓	✓	10	10-23-09	F4	GN			✓		Repeat of R112304
		W470360-2			1mL					F5						Repeat of R112305
		-3								F6						
		-4	200		NR	✓				F7						Transfer 1mL aliquot to 10mL in DI
		W470361-2	1000		1mL		✓			F8				✓		
		-3								F9				✓		
																10-23-09 GN

EX-002 – Revision 1 – 10/15/09

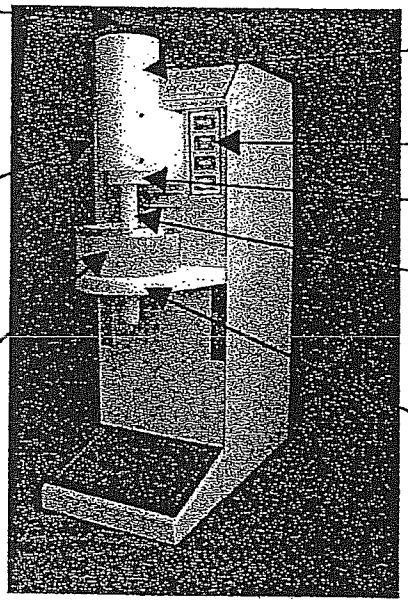
QAEX177 0000010

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
10/22/09	OB	SC6328-1	200	1mL	NR	✓		10mL	10-23-09	F10	GN					Duplicate of site sp. FILP overnight to 10mL in DI
		SC6340-2								F11						
		-6								F12						
		-8								PFSEL A1						
		SC6345-11								A2						
		SC6413-1								A3						
		SC6433-15a	1060				✓			A4				✓		
		-16j	1040							A5						
		SC6457-2L	1060							A6						
		-3L	970							A7						
		-4L	1040							A8						
																10-23-09 GN

EX-002 – Revision 1 – 10/15/09 QAEX177 0000011

FIGURE 2

HORIZON AUTOEXTRACTOR SYSTEM DIAGRAM



TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J. Benton Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·15·00

General Manager: Duncan T. Hufnah Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11·15·00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced Figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated Logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in Logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-524-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of

samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste

generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

b) 500 mL flat-bottom boiling flask

c) 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, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.3 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.4 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.5 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.6 Matrix Spike/Lab Control Sample spiking solution

5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone
Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

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

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps and pre-baked glass wool with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. 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 Please refer to Katahdin Analytical Services SOP CA-108, current revision, "Basic Laboratory Techniques" for more information of subsampling.

7.3 Weigh out a 30.00 ± 0.05 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.05 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. 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 pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% 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. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

7.14 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. Samples that undergo GPC are not solvent exchanged into hexane.

7.15 If samples are not to be GPC'd follow Steps 7.16 through 7.23 to concentrate extracts to final volume of 10.0 mLs

7.16 For a solvent exchange, (for samples not being GPC'd), add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent 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 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.

7.19 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, hexane, if exchange is taking place. 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, hexane, if exchange is taking place.

7.20 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus to ensure all methylene chloride has been evaporated. 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. <u>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.</u> 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..

7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "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.

7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis, unless it has been GPC'd. All sample extracts for 8081 pesticide analysis should undergo further cleanup using the GPC unless time is a factor. All sample extracts for combined 8081/8082 analyses must be split unless GPC'd. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB 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
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

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 are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, 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, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-06	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PCB
SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) X	SW846 3545 (ASE)
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0608	Spike ID:
Solvents	Solvent Lot # (MeCl2): H17E31	Solvent Lot # (Acetone): G5[illegible]	Solvent Lot # (Hexane): L46E07
Consumables	Filter Paper Lot # (SON) J115[illegible]481	Filter Paper Lot # (KD) E116723G3	Acid Lot # H04032
	Sodium Sulfate Lot #: 72969002	Powder 27979001	
Misc.	Nitrogen Bath Temperature: 35° C	Sonicator Horns Tuned: [illegible]	Balance ID: Mettler PJ400

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest.	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
7-21-09	AC	W66647-1	29.96	1mL	NR		X				✓	7/23/09	CB	10mL	Hood B2	R104594
↓	↓	↓ -2	29.99		1mL		X				↓	↓	↓	↓	B8	
↓	↓	↓ -3	29.96	↓	↓		X				↓	↓	↓	↓	B9	
								CB 7/27/09								

0000129

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest.	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
7-21-09	AC	SC3972-4	29.96	1mL	NR		X				✓	7/23/09	CB	10mL	Hood B10	acid remains
↓	↓	SC4000-5	30.02		↓		X				↓	↓	↑	↓	B11	PT polychlorinated biphenyls 2525
		SC4005-1	30.01				X								B12	acid remains
		SC4044-2	29.97				X								C1	
↓	↓	↓ -3	29.97	↓	↓		X				↓	↓	↓	↓	C2	
								CB 7/27/09								

QAEX162

0000130

Reviewed By_____ Date_____

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: [illegible] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·16·00

General Manager: [illegible] Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	On	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 LAD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~mixes~~ 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 adipate compounds to Fig. 2. Added recording of consomable'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

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

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 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".

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

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

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried 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.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:
- a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
- b) 500 mL flat-bottom boiling flask
- c) 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 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS

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.

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 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - 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	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

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 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	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.

5.6 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.

5.7 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. 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.

5.8 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

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Sign chain-of-custody when removing and replacing samples in storage locations, and 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 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 Sediment/soil samples - Decant and discard any water layer on a sediment sample. 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 a 30.00 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.05 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 30.00 ± 0.05 g 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 30.00 ± 0.05 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 30.00 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 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 appropriate base/neutral and acid surrogate spiking solution listed below 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.

7.8.1 If the request is for SVOA, use the SVOA surrogate solution (sect. 5.4)

7.8.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.8.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

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/SIM, add 1 mL of the SVOA spiking solution and 1 mL of the SIM Spiking solution to appropriate LCS/LCSD and/or MS/MSD. (sect's 5.6 and 5.7).

7.9.4 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 55-60% 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 solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF 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. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. 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 (75-85°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 ≈ 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 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.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

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 are determined annually per type of instrument and filed with the Organic Department Manager and 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, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-06	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

SV SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2324	Spike ID (1): SV2320	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (Mecl2): H196008	Solvent Lot # (Acetone): —	Sodium Sulfate (granular) Lot # ~~2769~~ 279690D2
	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) K1672363	Sodium Sulfate (powder) Lot # ~~2769~~ 279690D1
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler R3400

QAEX169

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol (mL)	Spk. Vol. (mL)	Fraction		Pre-GPC			Post-GPC				Comments
						BN	AE	Date Conc.	Conc. Init.	Final Vol. (mL)	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc.	
6-23-09	KF	1665386-1	30.00	1mL	1ml	✓		6-26-09	KF	5mL	6-29-09	KF	1mL	SV451 B6	R102843
		-2	29.98		1ml									B7	
		-3	29.96											B8	
		GPC Blank 6-29-09												B9	
										KF	6-26-09				

0000007

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol (mL)	Spk. Vol. (mL)	Fraction		Pre-GPC			Post-GPC				Comments
						BN	AE	Date Conc.	Conc. Init.	Final Vol. (mL)	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc.	
6-23-09	KF	SC3258-11F	30.04	1mL	1ml	✓		6-26-09	KF	5mL	6-30-09	GN	1mL	SV451 B10	
		-12F	30.02											C1	
		-13F	30.00											C2	
		-14F	30.01											C3	
		-15F	29.96											C4	
		-16F	30.05											C5	
		-17F	30.02											C6	
		-18F	29.99											C7	
		-19F	29.99											C8	
		-20F	30.00											C9	
		SC3259-3F	30.99											C10	
		-4F	29.96											D1	
		-5F	30.00											D2	
		SC3305-3F	30.01											D3	
										KF	6-26-09				

QAEX169

0000008

Reviewed By_____ Date_____

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

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Prepared By: Galen Nickerson Date: 6-23-06

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Radeau Date: 6-23-06

QA Officer: Leslie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1.4 - Added solvent and acid waste streams. Changed solvent in sect. 7.4.2 from hexane to MeCl2. Updated logbook example. Added recording of solvent, Na2SO4, hydromix, filterpaper Lot #'s as well as the temperature of the Na waterbath in the logbook.	LAD	07/08	07/08
02	Updated section 4 with current materials. Updated section 7 with current techniques. Changed when Hexane is added during the solvent exchange process. Changed all weights to record to 0.05g. Updated logbook example. Added CA-108 reference for subsampling.	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-537-02,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-537-02,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

Method 3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges, waste solids and tissues samples. This Pressurized Fluid Extraction (PFE) method uses elevated temperature (100°C or 175°C) and pressure (1500 - 2000 psi) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of organochlorine pesticides and PCBs. Organochlorine pesticides and PCBs may then be analyzed by a variety of chromatographic procedures.

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. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks,

standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide and PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide and PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that 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 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

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Samples are prepared for extraction by weighing out a specific quantity of sample and mixing said quantity with pelletized diatomaceous earth (Hydromatrix) to remove moisture. The sample is then loaded into an extraction cell and placed on the Accelerated Solvent Extractor (ASE).

The extraction cell containing the sample is heated to the analyte-specific extraction temperature (see section 7.0), pressurized with hexane and extracted for a period of time outlined in section 7.0.

The solvent is collected from the extraction cells into 60 mL vials and allowed to cool. The solvent is then concentrated and, as needed, exchanged into solvent compatible with the cleanup or determinative step being employed.

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. Pressurized fluid extraction device: Dionex Accelerated Solvent Extractor, Model number ASE 200 with appropriately sized extraction cells. The cells will accommodate 15 g of sample and are made of stainless steel which is capable of withstanding the pressure requirements necessary for this procedure.

4.2. Analytical Balance: Mettler PJ4000 balance capable of weighing to 0.01 g

4.3. Vials for the collection of extracts: Dionex 60 ml pre-cleaned, open to screw cap with PTFE-lined silicone septum.

4.4. Filter disk for extraction vessels: Dionex 1.91 cm, Type D28.

4.5. 400 ml beakers

4.6 Ottawa Sand (Fisher P/N 523-3)

4.7 Kuderna-Danish (K-D) apparatus

4.7.1 Concentrator tube - 10-mL

4.7.2 Evaporation flask - 500-mL

4.7.3 Snyder column - Three-ball macro

4.8 Nitrogen evaporation (N-EVAP) apparatus.

4.9 Stainless steel spatula.

4.10 Porcelain mortar and pestle.

4.11 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride.

4.12 Vials - Glass, 12 mL and 4 mL capacity

4.13 500 ul syringes

4.14 Funnel powder, glass.

4.15 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.16 Water bath or steam bath capable of maintaining a temperature of at least 85°C.

5.0 REAGENTS

5.1 Aluminum oxide (Alumina, acid), Brockman activity I, 60-325 mesh

5.2 Drying agents

5.2.1 Pelletized diatomaceous earth (Hydromatrix) - Fisher P/N.

5.2.2 Sodium sulfate crystals, Na_2SO_4.

5.3 Extraction solvents

5.3.2 Methylene Chloride - Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL and evaluating by both GC/MS and GC/FID analyses.

5.3.3 Hexane - Pesticide grade or equivalent. Lot must be verified by concentrating 200-300 mLs to 1 mL followed by GC/ECD analysis.

5.3.4 Acetone – Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL followed by GC/MS, GC/ECD, and GC/FID analyses.

5.3.5 Organochlorine pesticides and PCBs may be extracted with acetone/methylene chloride (1:1,v/v) or hexane (100%).

5.3.6 Other solvent systems may be employed, provided that the analyst can demonstrate adequate performance for the analytes of interest in the sample matrix

CAUTION: For best results with very wet samples (e.g., ≥ 30% moisture), reduce or eliminate the quantity of hydrophilic solvent used.

5.4 Pre-purified nitrogen is used to purge and/or pressurize the extraction cell.

5.5 Spiking Solutions

5.5.1 Pesticide/PCB surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone. Store the solution at –10 to -20 °in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.5.2 Pesticide/PCB matrix spike/Lab control sample spiking solution - Prepare separate spiking solutions for Pesticides and one for PCBs in pesticide grade methanol and acetone, respectively, that contain all target analytes listed below:

Pesticide Spiking Solution

Analyte	ug/mL	Analyte	ug/mL
4,4'-DDD	0.5	Endosulfan I	0.5
4,4'-DDE	0.5	Endosulfan II	0.5
4,4'-DDT	0.5	Endosulfan Sulfate	0.5
Aldrin	0.5	Endrin	0.5
alpha-BHC	0.5	Endrin Aldehyde	0.5
beta-BHC	0.5	Endrin Ketone	0.5
delta-BHC	0.5	gamma-BHC (Lindane)	0.5
Dieldrin	0.5	Heptachlor	0.5
alpha-Chlordane	0.5	Heptachlor Epoxide	0.5
gamma-Chlordane	0.5	Methoxychlor	0.5

PCB Spiking Solution

Analyte	ug/mL
Aroclors 1016/1260	5.0

Store the solution at –10 to -20 °C in a Teflon sealed container. The solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Holding time for extraction of sediment/soil and tissue samples for Method 3545 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

7.1 Preparing the Accelerated Solvent Extractor

7.1.1 Before extractions can begin, the system must first go through its daily maintenance checks. To begin, turn on the system and nitrogen gas tank. Check the system pressure to ensure that the solvent pressure is at 10 psi, air is at 50 psi, and compression is at 130 psi. Next, check the solvent bottle and fill with the appropriate solvent if necessary. At this time the rinse and waste vials should be emptied and four 60 ml vials need to be placed in the rinse positions of the sample collection carousel. Record the solvent lot number in the extraction logbook.

7.1.2 The system must now be rinsed before the analysis can begin. Press the rinse button on the touch pad. The system will immediately begin its rinse cycle. This rinse procedure should be repeated at least three times before any samples are extracted to ensure that all contaminants are removed from the system.

7.1.3 To prepare extraction cells, disassemble the ASE extractor cells and clean each piece with soapy water. Next rinse each piece with acetone to ensure that all water is removed. Next, rinse the body and two screw caps three times with methylene chloride. Be sure you are using a solvent whose lot has been checked. Inspect each screw cap to ensure that the o-rings are not crushed and in need of replacement. O-rings should be replaced after approximately 50 extractions to ensure proper fit. Cell bodies should also be

checked for nicks and dings at each end. If any dings are present the cell should be placed out of rotation until it can be repaired. If any instrument maintenance is necessary record the maintenance performed in the instrument maintenance logbook.

7.1.4 Once the extraction cells have been rinsed replace the bottom cell screw cap. The bottom of the cell body is the end that is imprinted with Dionex. Next, place 2 19.8 mm filter disks into the top of the cell body and push it to the bottom of the cell with the plunger that was supplied with the system. Record the lot numbers of the filter disks in the extraction logbook.

If extracting pesticides/PCBs from tissue samples weigh out 5.00 ±1.00 g of aluminum oxide (alumina, acid) into cell and place a second filter on the top of alumina. Avoiding matrix affect interference for tissue sample, the aluminum oxide is used to remove lipid. Record the lot numbers of the aluminum oxide and filters in the extraction logbook.

7.1.5 Label each extraction cell and clean, rinsed, 60 ml collection vial with the appropriate sample ID with a black marker.

7.2 Sample preparation

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. 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 grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. Materials such as glass, rubber, metal, etc. may not require mixing with Hydromatrix to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to ASE extraction.

7.2.3 Tissue samples are blended prior to handling in extraction laboratory.

7.2.4 Refer to Katahdin Analytical Services SOP CA-108, current revision, "Basic Laboratory Technique" for more information regarding subsampling.

7.3 Sample Handling

The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.3.1 Weigh out 15.00 ± 0.05g portion of sample into a 400 ml beaker. For tissue sample, weigh out a 5.00 ± 0.05g portion of sample into a 400 ml beaker. Record sample weight to the nearest 0.05g in the appropriate extraction logbook.

7.3.2 Add enough Hydromatrix to the sample to produce a "free-flowing" mixture, around 5g. For tissue sample, pulverize the mixture (sample-hydromatrix) using the porcelain mortar-pestle apparatus. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. The amount of Hydromatrix will depend upon the moisture content of the sample (e.g., low moisture content will require less Hydromatrix). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record the Hydromatrix lot number in the extraction logbook.

7.3.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out a 5.00 ± 0. 05g portion of Ottawa sand for tissue sample batch or 15.00 ± 0.05g portion for sediment/soil sample batch into a 400 ml beaker. Record the weight to the nearest 0.05 g in the appropriate extraction logbook. Add enough Hydromatrix to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. If no MS/MSD is to be prepared a laboratory control sample duplicate must be prepared. To prepare an LCS (and LCSD), weigh out a 15.00 ± 0.05g portion of Ottawa sand into a 400 ml beaker; for tissue sample, weigh out a 5.00 ± 0. 05 g of Ottawa sand into a 400 mL beaker. Record the weight to the nearest 0. 05 g in the appropriate extraction logbook. Add enough Hydromatrix, approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker

with aluminum foil. **If extracting both pesticide and PCB separate LCS/D and MS/D need to be prepared.**

7.3.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out a 15.00 ± 0.05g portion of designee sample into a 400 ml beaker. For tissue sample, weigh out a 5.0 ± 0.05g portion of designee sample into a 400 mL beaker. Record the weight to the nearest 0.05g in the appropriate extraction logbook. Add enough Hydromatrix approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.6 Once all the QC and field samples have been weighed and mixed with a portion of Hydromatrix, find the appropriately labeled extraction cell and place the cell funnel, rinsed three times in methylene chloride, on the open end. This funnel was supplied with the system. Pour the contents of the beaker into the extraction cell. Carefully scrape all the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. If there is head space remaining in the extraction cell fill the remaining space with more Hydromatrix by pouring the sample back into the beaker and mixing the new Hydromatrix into the sample. This will minimize the amount of solvent used. Again, pour the sample back into the same extraction cell and cover with a screw cap. Rinse the cell funnel with extraction solvent between each sample.

7.3.7 To all tissue samples, method blank, LCS, and MS/MSD add 0.2 mL of Pest/PCB Surrogate spiking solution using a pre-rinsed 500 uL gas tight syringe. To all sediment/soil samples and QC being extracted add 0.5 mL of Pest/PCB surrogate spiking solution using a pre-rinsed 0.5 mL gas-tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is complete. To LCS and MS add 0.5 mL of appropriate spike solution to each.

7.3.8 Replace all screw caps and ensure that each is properly tightened. Place each extraction cell into the ASE sample carousel. Place the corresponding collection vial into the appropriate location on the sample collection carousel. Be certain that the cell and vial are in the same number position of the carousel. In order to avoid a possible carryover, samples should be placed in order of their cleanness.

7.3.9 Press the menu button on the ASE touch pad. Choose option number one and press enter. Select method number one for Pesticide and PCB analysis, press enter. Press start on the
touch pad. The extraction process will begin and takes approximately 14 minutes per sample to complete.

NOTE: Recommended extraction conditions:

<u>For Pest/PCB (saved in the instrument as method one)</u>
Oven temperature: 100°C
Pressure: 1500 - 2000 psi
Static time: 5 min (after 5 min pre-heat equilibration)
Flush volume: 60% of the cell volume
Nitrogen purge: 90 sec at 150 psi (purge time may be extended for larger cells)
Static Cycles: 1

Optimize the conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500 - 2000 psi should suffice.

Once established, the same pressure should be used for all samples extracted for the same analysis type.

As stated above, the recommended conditions have been saved as methods on the instrument. If it becomes necessary to re-program conditions, follow the above guidelines.

7.3.10 Once the extraction process is complete and the instrument is idle remove the sample extracts and label with appropriate sticker. They are now ready to be stored in the interim extract storage refrigerator unless the samples are to be concentrated the same day.

7.3.11 Remove the extraction cells from the ASE and empty their contents into the appropriate solid waste container.

7.3.12 The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferents and the determinative method to be employed. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

7.4 Concentration of Extracts

7.4.1 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.

7.4.2 If samples are not to be GPC'd follow steps 7.4.3 through 7.4.9 to concentrate extracts to final volume of 5.0 mLs for soil/sediment samples and 2.0 mL for tissue samples

7.4.3 Before assembling, rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with hexane if using 100 % hexane for Pesticide/PCB extraction, if not rinse three times with methylene chloride. 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. Rinse the sodium sulfate in the assembled funnels with ≈20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Visually examine each 60mL vial of sample. If there is excessive moisture in the sample, biphasic layering will be seen. If this occurs, add approximately 1 inch of sodium sulfate crystals to the vial. This should remove most of the moisture. Record the lot number of the sodium sulfate in the extraction logbook.

7.4.4 For a solvent exchange, no GPC, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.4.5 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. After pouring all of the extract volume through the sodium sulfate, rinse the extract vial three times with 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.

7.4.6 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of hexane.

7.4.7 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.4.8 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 hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the 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.

7.4.9 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 5 mL in a 12mL vial using the appropriate "reference vial" for volume comparison. For Pesticides/PCB from tissue samples, adjust the volume of hexane extract to 2.0 mL in a 4.0 mL vial using the appropriate "reference vial" for volume comparison.

7.4.10 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.

7.4.11 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. Sample extracts for 8081 pesticide do not undergo further cleanup unless requested by the client. All soil/sediment sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method

blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spike samples are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

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 Pesticides and PCBs for quality control acceptance criteria.

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 are determined annually per type of instrument and filed with the Organic Department Manager and 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.

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3545A, SW-846, Third Edition, Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, Revised February 2007, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-537-02	METHOD 3545, current revision
Apparatus/Materials		
Reagents	Katahdin receives a certificate with each lot indicating sodium sulfate crystals were dried by the manufacturer at prescribed conditions.	Section 5.3.3 Drying agents should be purified by heating at 400 °C for 4 hours…
Sample preservation/ handling	Samples are not ground prior to mixing with the drying agent.	Section 7.3 Grind a sufficient weight of the dried sample…
Procedures	Section 7.3.9 Nitrogen purge for Pesticide/PCB and DRO: 90 sec at 150 psi Oven temperature for DRO: 175°C	Section 7.8 Recommended extraction conditions for all extractions: Nitrogen purge: 60 sec at 150 psi. Oven temperature: 100 °C
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PCB
ASE

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX)	SW846 3545 (ASE) X
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0608	Spike ID:
Solvents	Solvent Lot # (Mecl2): —	Solvent Lot # (Acetone): G51E06	Solvent Lot # (Hexane): G46E07
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) J1S5884	Acid Lot # H04032
	Sodium Sulfate Lot #: 279 69 002		
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler PJ400

QAEX162

						Fraction		Pre - GPC				Post - GPC				
Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Pest	PCB	Date Conc.	Conc. Init.	Final Vol. (mL)	Acid Wash	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	WG65085-1	14.96	0.5mL	NR		X	—			✓	6-16-09	KF	5mL	KF 6-23 C2	R102347
		-2	14.98		0.5mL		X								C3	
		-3	15.00				X				✓	6-17-09	AC		C12	
								KF 6-16-09								

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						Fraction		Pre - GPC				Post - GPC				
Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Pest	PCB	Date Conc.	Conc. Init.	Final Vol. (mL)	Acid Wash	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	SC3169-1	14.97	0.5mL	NR		X	—			✓	6-16-09	KF	5mL	KF 6-23 C4	
		-3	14.97												C5	
		-5	15.02												C6	
		-7	14.99												C7	
		-9	14.99												C8	
		-11	15.04									6-17-09	AC		C9	
		-13	15.02												C10	
		-15	15.03												C11	
								KF 6-16-09								

QAEX162

0000076

Reviewed By_____ Date_____

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP 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. Madeau Date: 1·22·01

General Manager: Dennis F. Hefner 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.	DH	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.	DH	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

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-604-04**, 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, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-04**, 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, Inc. 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 beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Chemical Hygiene 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 a respirator and all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

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

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**

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 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 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.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

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.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs 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 3010A.

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Table 2 Summary of Method Modifications
Table 3 Procedure Condensation
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Figure 2 Preparation of Matrix Spikes, LCSs, and Spiking Solutions: Method 3010
Figure 3 Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

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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

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-04	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.

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FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: C20022 JT Baker HCL: C07046 Ashland H2O2: NA

Method: 3010

Standards/Spiking Information:
I.V. CLPP-SPK-1 (ID/Vol): MS1362 / 0.05 mL NA : NA
CLPP-SPK-INT1 (ID/Vol): MW9975 / 0.5 mL Hot Plate No.: A
CLPP-SPK-INT2 (ID/Vol): MW9930 / 0.5 mL Temp.: 95 °C
U-Spike (ID/Vol): MS1332 / 0.05 mL

REVIEWED
EM 06-27-06
KATAHDIN ANALYTICAL METALS SECTION

Spiking Witnessed by: N/A

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LC2WWF27ICW1	WF27ICW1	0.05	L	0.05	L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A	N/A	N/A
LCSWWF27ICW1	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
PBWWF27ICW1	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
WW3165-001	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-002	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-003	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-004	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-005	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-006	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-007	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-008	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-009	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-010	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-011	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-012	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-013	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-014	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-015	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-016	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-017	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-018	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-019	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-020	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						

DJJ 6/27/06

Digestion performed by: DJJ On: 6/27/06

Page: WF061 Revision: 00

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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
	1000 mg/L Uranium Standard	Inorganic Ventures	0.005

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	5.0
	1000 mg/L As	High Purity Standards	5.0
	1000 mg/L Pb	High Purity Standards	5.0
	1000 mg/L Cd	High Purity Standards	2.5
	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
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

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 IN SOLUTION, mg/L							
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000					
Antimony	0.500	0.500		100	50			
Arsenic	0.500	0.500		4	50			
Barium	2.000	2.000	2000					
Beryllium	0.050	0.050	50					
Boron	0.500	0.500		50		50	500	
Cadmium	0.250	0.250		5	25			
Calcium	2.500	2.500			250			
Chromium	0.200	0.200	200					
Cobalt	0.500	0.500	500					
Copper	0.250	0.250	250					
Iron	1.000	1.000	1000					
Lead	0.500	0.500		2	50			
Magnesium	5.000	5.000			500			
Manganese	0.500	0.500	500					
Molybdenum	0.300	0.300		30		30	300	
Nickel	0.500	0.500	500					
Potassium	10.000	10.000			1000			
Selenium	0.500	0.500		5	50			
Silicon	5.230	5.230				523	230	
Silver	0.050	0.050	50					
Sodium	7.500	7.500			750			
Strontium	0.500	0.500		50		50		
Thallium	0.500	0.500		5	50			
Tin	0.500	0.500		50		50		
Titanium	1.000	1.000		100		100	1000	
Uranium	0.100	0.100						1000
Vanadium	0.500	0.500	500					
Zinc	0.500	0.500	500					

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-605
Revision History
Cover Page
Page 1

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

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. Lufkin Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3+4 to reflect current practice.	DN	8.29.02	8.29.02
03 3050B	New Title to include ILMO5.3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 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 2.0ml, 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

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-605-04**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-605-04**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. 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.

LCSS – Laboratory Control Sample for Solids – 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.

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.

Personnel are required to read the Katahdin Hazrdous Waste Management Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and 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 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 Mnagement Plan and Safety Manual.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

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°C ± 5°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 recordedeach 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

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°C ±2°C) 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. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet. Refer to Katahdin Analytical Services SOP CA-108, current revision "Basic Laboratory Technique" for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95°C ± 5 °C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95°C ± 5°C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or

snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does

not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Depatment 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 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Table 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

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.

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 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

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Table 2 Summary of Method Modifications – Method 3050
Table 3 Preparation of Matrix Spikes and Spiking Solutions
Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
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Figure 2 Example Page from Metals Sample Preparation Logbook
Figure 3 Example Certificate of Analysis for Solid Reference Material

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 Solids (LCSS)	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	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.

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-02	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.

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	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00
	1000 mg/L Uranium Std.	High Purity Standards	0.01

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	5.0
	1000 mg/L As	High Purity Standards	5.0
	1000 mg/L Pb	High Purity Standards	5.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	5.0
	1000 mg/L K	High Purity Standards	10.0
	1000 mg/L Na	High Purity Standards	7.5
	1000 mg/L Mg	High Purity Standards	5.0
	1000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

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

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike (ICP-AES)	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3 SPK-3	2007 ICS- 1	1000 mg/L U
Aluminum	2.000	2000						
Antimony	0.500		100	50				
Arsenic	0.500		4	50		500		
Barium	2.000	2000						
Beryllium	0.050	50						
Boron	0.500		50		50		500	
Cadmium	0.250		5	25		250		
Calcium	2.500			250				
Chromium	0.200	200						
Cobalt	0.500	500						
Copper	0.250	250						
Iron	1.000	1000						
Lead	0.500		2	50		500		
Magnesium	5.000			500				
Manganese	0.500	500						
Molybdenum	0.300		30		30		300	
Nickel	0.500	500						
Potassium	10.000			1000				
Selenium	0.500		5	50		500		
Silicon	5.230				523		230	
Silver	0.050	50						
Sodium	7.500			750				
Strontium	0.500		50		50			
Thallium	0.500		5	250		500		
Tin	0.500		50		50			
Titanium	1.000		100		100		1000	
Uranium	0.100							1000
Vanadium	0.500	500						
Zinc	0.500	500						

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 perfoming 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

Reagent Information:
JT Baker HNO3: H12022 JT Baker HCL: H64040 KMG H2O2: 62022 Method: 3050H

LCS / Spike LCS/Spiking Information:
☐ ☑ I.V. CLPP-SPK-1 (ID/Vol): MS1569 / 0.1 mL Hot Plate/Block ID: B Fisher Filter Paper: K1267273 3B
☐ ☑ CLPP-SPK-INT1 (ID/Vol): MW11904 / 1.0 mL Start Temp.: 95 °C
☐ ☑ CLPP-SPK-INT2 (ID/Vol): MW11864 / 1.0 mL End Temp.: 95 °C
☐ ☑ Uranium Spike (ID/Vol): MS1594 / ~~0.01~~ 0.1 mL ASD Thermometer ID/Pos.: ALC B / 1:4
☐ ☐ CLPP-SPK-4 (ID/Vol): N/A / mL 7/31/09
LCSS: MS1578 Balance ID: Ohaus Galaxy 400 / Mettler AE200

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KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LC2SZG311CS0	ZG311CS0	0.5074	g	0.10	L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A	N/A	N/A
LCSSZG311CS0	ZG311CS0	0.5034	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
PBSZG311CS0	ZG311CS0	1.00	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
SC4328-001	ZG311CS0	1.79	g		L	SL	IC	AJB	07/31/2009						A
SC4328-001P	ZG311CS0	1.05	g		L	SL	IC	AJB	07/31/2009						
SC4328-001S	ZG311CS0	1.04	g		L	SL	IC	AJB	07/31/2009						
SC4348-001	ZG311CS0	1.35	g		L	SL	IC	AJB	07/31/2009						
SC4348-002	ZG311CS0	1.24	g		L	SL	IC	AJB	07/31/2009						
SC4348-003 7/31/09	ZG311CS0	1.11	g		L	SL	IC	AJB	07/31/2009						
SC4357-001 ASD	ZG311CS0	1.46	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~002~~ 002	ZG311CS0	1.17	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~003~~ 003	ZG311CS0	1.52	g	0.10	L	SL	IC	AJB	07/31/2009						A

AJB 7/31/09

Digestion performed by: AJB Date: 7/31/09

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™

M51475

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. - 198
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

KATAHDIN ANALYTICAL SERVICES, INC.
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. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: Dennis P. 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.	DN	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 concs. in Tables 3,4,5,6 to reflect current practices.	DN	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

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-09**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-09**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

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 group supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Group Supervisor 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 I 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

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 3 and 4 for a listing of stock standards required, and to Table 7 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 4 for a listing of intermediate standards required and for preparation instructions. Refer to Table 6 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 3 for a listing of working standards and for preparation instructions. Refer to Table 5 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]	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 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 8 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 multielement calibration standard (S1 in Table 3) 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 Table 1 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 Table 1 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 Table 1 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 Table 1 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.

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.

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 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 a 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 ± PQL if the PQL is greater than 0.01 mg/L, within ± 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 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%. 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.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are 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 and LODs are determined annually per

type of instrument and filed with the Metals Supervisor and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09

Katahdin SOP CA-101, Equipment Maintance

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 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, Method 6010C.

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) ≥ 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. DoD: no analytes detected > LOD	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem. 3) DoD: Do not report results without valid ICB
	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. 3) DoD: Do not use results for failing elements unless CCV > 110% and the sample is below reporting limit.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL. DoD: no analytes detected > LOD	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem. 3) DoD: Do not report results without valid CCB
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value. DoD: Recovery within 20% of true value	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem. 3) DoD: No samples may be run without valid PQL
	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. DoD: Absolute value for all non-spiked analytes < LOD, unless verified trace impurity	1) Do not use results for failing elements. 2) Investigate and correct problem.

TABLE 1 (cont.)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	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. DoD: No analytes detected >1/2 RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit. (whichever is greater) For common contaminants, no analytes detected >RL	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration. 3) DoD: Report flagged results for samples greater than 10x target analyte in blank. Report results for target analytes less than ½ RL or less than RL for common contaminants. Redigest and reanalyze all other associated samples.
	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. DoD: Recovery within ± 20% of true value; for Ag in solid matrix, recovery within 75 – 120%.	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. DoD: Use QC acceptance criteria specified for LCS.	1) Flag results. 2) DoD: Perform additional project specific quality control tests.
	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. DoD: Use QC acceptance criteria specified by DoD for LCS. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results. 2) DOD: Perform additional project specific quality control tests.

TABLE 1 (cont.)

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 (LOQ if DoD), 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	DoD: 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.
	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	0% - 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

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-608-09	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 3

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 4)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	QCP-CICV-3	Inorganic Ventures	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn	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 4)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 4)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 4)	1.0

TABLE 4

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 B,Li,Sn,Sr, W, U	H-P or IV	1.0 each
	10000 mg/L K, Na	H-P or IV	1.0 each
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	1000 mg/L Si	High Purity Standards	2.0
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se, Mo	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,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
	10000 mg/L Fe	High Purity Standards	0.1
ICSAB-INT	10000 mg/L K,Na	H-P or IV	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, W, U	High Purity Standards	1.0 each
	1000 mg/L Si	High Purity Standards	4.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L								
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**MN_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	0.1		0.5	0.5			
Cadmium	1	0.4	0.01		1.0	0.5			
Calcium	25	10	0.05	500	500	12.5			
Chromium	1	0.4	0.015		0.5	0.5			
Cobalt	1	0.4	0.03		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	0.05	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.04		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.015		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.1		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			
Tungsten	1	0.4	0.1		0.5	0.5			
Uranium	1	0.4	0.1		0.5	0.5			
Vanadium	1	0.4	0.025		0.5	0.5			
Zinc	1	0.4	0.025		1.0	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

	CONCENTRATION IN SOLUTION, mg/L			
Element	**ICP Intermed STD**		**PQL-INT**	**ICSAB-INT**
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			10	10
Cadmium			1.0	
Calcium	240		5.0	
Chromium			1.5	
Cobalt			3.0	
Copper			2.5	
Iron	240		10	
Lead			0.5	
Lithium	10		10	10
Magnesium	240		5.0	
Manganese			0.5	
Molybdenum			1.0	10
Nickel			4.0	
Potassium	150		100	400
Selenium			1.0	
Silicon	250		20	40
Silver			1.5	
Sodium	240		100	400
Strontium	10		10	10
Thallium			1.5	
Tin	10		10	10
Titanium			1.5	10
Tungsten	10		10	10
Uranium	10		10	10
Vanadium			2.5	
Zinc			2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	IV-28	QCS-26	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	QCP-CICV-3
Aluminum	100	100		5000		
Antimony	100	100			60	
Arsenic	100	100			10	500
Barium	100	100			50	
Beryllium	100	100			50	
Boron	100	100	500			
Cadmium	100	100			100	250
Calcium	100	100		5000		
Chromium	100	100			50	
Cobalt	100	100			50	
Copper	100	100			50	
Iron	100	100		2000		
Lead	100	100			5	500
Lithium	100					
Magnesium	100	100		5000		
Manganese	100	100			50	
Molybdenum	100	100	300			
Nickel	100	100			100	
Potassium	1000	1000				
Selenium	100	100			5	500
Silicon	50	50	230			
Silver	100	100			20	
Sodium	100	100				
Strontium	100					
Thallium	100	100			10	500
Tin						
Titanium	100	100	1000			
Vanadium	100	100			50	
Zinc	100	100			100	

TABLE 8

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	

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, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-611
Revision History
Cover Page
Page 1

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: John C. Butler Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Hufnah 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 QC 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**

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-611-06, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-06, 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, Inc. 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, and sludges under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade 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 laboratory reagent grade 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.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

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.

PB - Preparation Blank - Laboratory reagent grade 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.

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.

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 there 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.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

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, for use as digestion vessels.

4.2 Water bath 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 Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory reagent grade water – mercury-free water which has been treated by the Culligan Water System and the Millipore Mill-QÔ Water System and meets the specifications of ASTM Type II water

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 Aqua regia: Prepare an appropriate amount immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3 in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood.

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°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 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 a silver paint marker, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.6 Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled digestion bottles. The mercury concentrations of these calibration standards will be, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0 ug/L, 0.2 ug/L and 0.5 ug/L standards are analyzed during analysis as the CCB, PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.7 Using a calibrated adjustable pipette, prepare the initial calibration verification (ICV) standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to an appropriately labeled digestion bottle. 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) by weighing appropriate masses of solid reference material into labeled digestion bottles. The

mercury concentration of these LCSSs 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.

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 All calibration standards, QC samples, and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP.

SAMPLE PREPARATION AND DIGESTION

7.12 Weigh three approximate 0.2 g portions (a total of approximately 0.6 g) of untreated sample from different parts of the sample container and place them in the bottom of a labeled digestion bottle. The purpose of using three portions is to obtain a representative sample from the sample container.

7.13 Add 5 mL of laboratory reagent grade water and 5 mL of aqua regia to each sample, standard, and QC sample. Place bottles in a water bath located in a fume hood and heat for 2 minutes at 95° C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 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. 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° 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 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.

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 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.

7.21.2 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.

7.21.3 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

7.22 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:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{(C) \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

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 (%)

7.23 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. 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 (LCSS), consisting of solid reference material 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 are supplied by the vendor of the solid reference material. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS 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.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are 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 and LODs are determined annually per type of instrument and filed with the Metals Supervisor and 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

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09

Katahdin SOP CA-101, Equipment Maintenance, current revision.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 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, Method 7471B.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

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TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL. DoD: Less than LOD	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or 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 or run, after every 10 samples, and at end of the run	Less than PQL. DoD: Less than LOD	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. DoD: No analytes detected >1/2 RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit. (whichever is greater) For common contaminants, no analytes detected >RL	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration. 3) DoD: Report flagged results for samples greater than 10x target analyte in blank. Report results for target analytes less than ½ RL or less than RL for common contaminants. Redigest and reanalyze all other associated samples.
	Laboratory Control Sample (LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within vendor-supplied acceptance limits. DoD: 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. DoD: Recovery within ± 20% of true value.	Flag results.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

TABLE 1

QC REQUIREMENTS (CONTINUED)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	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)DoD: Recovery within ± 20% of true value. 3)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 Annually.	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.

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-06	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 Verification	1)Known reference sample (ICV) analyzed daily. 2)Calibration verified after every 10 samples with CCV.	1)Known reference sample analyzed quarterly. 2)Calibration verified after every 20 samples.
QC - Calibration Blanks and Method Blanks	Acceptance Criterion: < PQL	Acceptance criteria: Low enough not to interfere with data quality objectives, or <10% of PQL, or <10% of regulatory limit, or <10% of lowest associated sample

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: G02058 JT Baker HCL: E45047 JT Baker H2SO4: N/A
JT Baker KMNO4: M2684 JT Baker K2S2O8: N/A JT Baker NH2OH-HCl: M8663
Method: 7471
LCSS: MS1475

Standards Information:
1ppm A = MW41032
1ppm B = MW41007
LCSS = MS1475
Spike(S/P) = 100uL of 1ppm A to 100mL
TCLPMS(M) = 50uL of 1ppm N/A to 25mL
ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL
S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

REVIEWED
EW 03-25-08
KATAHDIN ANALYTICAL METALS SECTION

Digestion Start Time (@ 95 °C): 1620 Digestion End Time (@ 95 °C): 1650

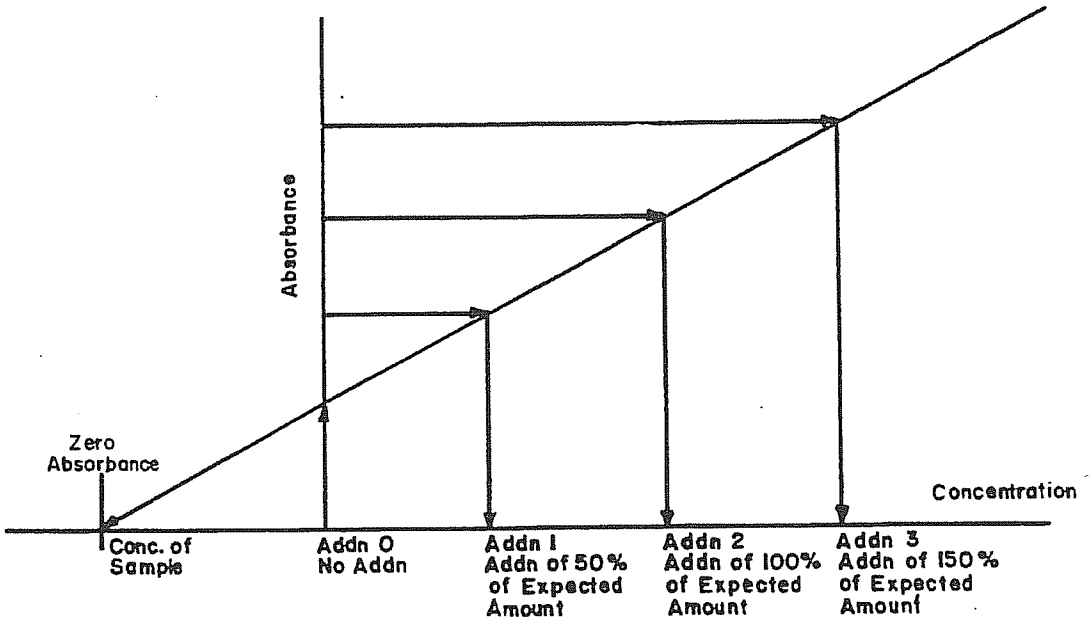
Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LCSSYC21HGS0	YC21HGS0	0.2663	g	0.100	L	SL	HG	HHH	03/21/2008	N/A	N/A	N/A	N/A		
MDL7471H-001	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-002	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-003	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-004	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-005	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-006	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-007	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-008	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-009	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-010	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
PBSYC21HGS0	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008	N/A	N/A	N/A	N/A		
SB1155-001	YC21HGS0	0.61	g		L	SL	HG	HHH	03/21/2008						G1
SB1155-001P	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						↓
SB1155-001S	YC21HGS0	0.63	g		L	SL	HG	HHH	03/21/2008						↓
SB1155-003	YC21HGS0	0.69	g		L	SL	HG	HHH	03/21/2008						↓
SB1378-001	YC21HGS0	0.68	g		L	SL	HG	HHH	03/21/2008						A
HHH ~~SB1378-002~~	~~YC21HGS0~~	~~0.60~~	~~g~~		~~L~~	~~SL~~	~~HG~~	~~HHH~~	~~03/21/2008~~						~~A~~

Digestion performed by: HHH On: 3-21-08 Page: YC073 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™

M51475

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. - 198
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

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-615
Revision History
Cover Page
Page 1

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 F. Hufnagel Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DN	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.	DN	08/09	08/09

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-615-04,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-04,** 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, Inc. 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.

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 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.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

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.

PB - Preparation Blank - Laboratory grade 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.

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.

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 VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95° C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC 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

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 VOA vials. 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 a silver paint marker, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to 6 standards digestion bottles (250 mL media bottles). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled media bottles containing 100 mL of laboratory grade reagent water. The mercury concentrations of these calibration standards are, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 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.

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled "ICV". Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV 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 All QC samples and blanks 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.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, 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.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95° C upon addition of the digestion vials. After the temperature of the bath has risen back to 95° C, continue heating the samples at 95° C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath 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 95° C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. 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".

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:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

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:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

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.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are 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 and LODs are determined annually per type of instrument and filed with the Metals Supervisor and 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 245.1 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

Table 1 QC Requirements
Table 2 Method Modifications
Figure 1 Example Mercury Preparation Logbook Page
Figure 2 Standard Additions Plot

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 ≥ 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. DoD: Less than LOD	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 20% 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. DoD: Less than LOD	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. DoD: No analytes detected >1/2 RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit. (whichever is greater) For common contaminants, no analytes detected >RL	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration. 3) DoD: Report flagged results for samples greater than 10x target analyte in blank. Report results for target analytes less than ½ RL or less than RL for common contaminants. Redigest and reanalyze all other associated samples.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	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)	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.	Flag results

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 1, CONTINUED

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	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.		

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-04	USEPA METHOD 7470
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	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
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	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: G41034 — JT Baker HCL: N/A — JT Baker H2SO4: G20022 — Method: 7470
JT Baker KMNO4: MR781 — JT Baker K2S2O8: MR780 — JT Baker NH2OH-HCl: MR776

Standards/Spiking Information:
1ppm A : ~~[illegible]~~ MW 11565
1ppm B : Dwm 2-25-09 MW 11566
LCSW = 125uL of 1ppm A to 25mL
Spike(S/P) = 25uL of 1ppm A to 25mL

ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL

S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

Balance ID: N/A
Water Bath ID : B
Digestion Start Time (@ 96 °C): 13:30
Thermometer ID : ACC-5
Digestion End Time(@ 95 °C): 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWZB24HGW0	ZB24HGW0	0.025	L	0.025	L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
PBWZB24HGW0	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
SC0797-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0805-007T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-013T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-002 Dwm 0.25mL	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0836-001	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						D
SC0846-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0858-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0858-002T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0868-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0868-015	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0834-013TP			L		L										
0.375			L		L										

Dwm 2-25-09

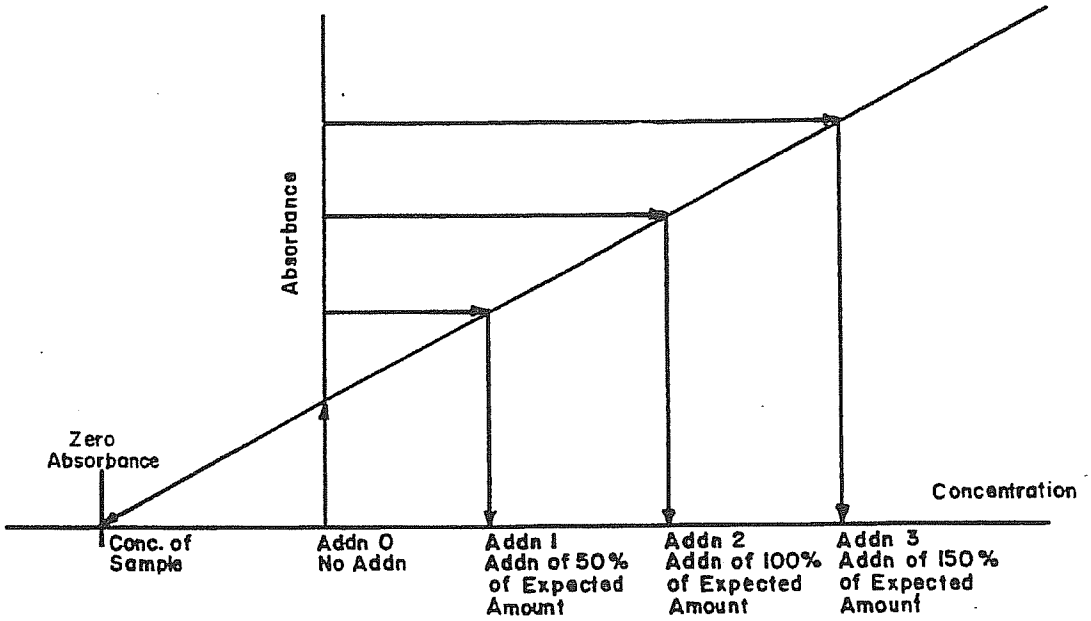
Digestion performed by: Dwm On: 2-24-09

Page: ZB071 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: 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: J. 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.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

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.

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-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. 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, Inc. (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., Department Manager, Operations 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 Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC 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 QAO and Vice President/President.

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 Vice President/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.

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

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 4). 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" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require 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 6). 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 7 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.

7.8 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.

7.9 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 16 hours after preservation before sample preparation can begin.

7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.

7.10.1 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.

7.10.2 A vacuum pump is attached.

7.10.3 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.

7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.11 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.

7.12 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 9).

7.13 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.

7.14 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 Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client

requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15 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).

7.16 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 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.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date that samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date that samples were collected in the format YY-Month-DDTIME.
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 long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
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.
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.

Type - Container type will automatically be entered.

Bot - Enter number of containers per test for printing of labels.

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager-	Initials of Katahdin person overseeing the project.
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 and regulatory agency (ie., IV NFESC).
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-	Used for federal programs.
EDD Format-	Specific KAS EDD format.

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".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.17.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.18 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDXL)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".

Note: As stated in "create containers" 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:

SC9001-001(a), SC9001-001(b), SC9001-001(c)

Example: One job containing two client samples with 2 different containers for each:

SC9002-001(a), SC9002-001(b), SC9002-002(a), SC9002-002(b)

7.19 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.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate

binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

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.

7.20.1 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.

7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.20.3 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.

7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" 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 walk-in.

7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or 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 with respect to native samples, extracts and digestates within the laboratory. 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.

7.21 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.

7.22 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 13).

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

7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.

7.24 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 14) 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. When the logbook for Immediate Internal COC's is used, the standard internal COC's do not have to be signed at a later date. The Immediate Internal COC Logbook must always be consulted if there is ever a question about whether an internal COC has been completed.

7.25 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.

7.26 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

7.27 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 annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual 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.

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, Inc., Environmental Health & Safety Manual, current revision.

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TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 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	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	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	1,11	28 days
pH (Laboratory)	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	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	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-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	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	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
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	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	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
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 (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GC ORGANIC ANALYSES					
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
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
GC/MS ORGANIC ANALYSES					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	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(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
MICROBIOLOGICAL ANALYSES					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES		**4 oz=100 g**			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
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	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
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	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
ELEMENTAL ANALYSES					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
ELEMENTAL ANALYSES **(cont.)**		**4 oz=100 g**			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	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
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
GC/MS ANALYSES					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	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-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
RCRA - HAZARDOUS WASTE CHARAC.					
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	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H_2SO_4 to pH<2
4 = HNO_3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M $Na_2S_2O_3$ or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = $Na_2S_2O_3$, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

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

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF HOMEOWNER KATAHDIN 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

Homeowner 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 must 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	Lead	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 Copper, Iron Manganese Sodium
													FHA/MSH Standard plus Lead 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 <6 degrees celcius. 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.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarbrough, 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:

Shipping: UPS Fed-Ex Airbill No.:

Temperature:

Food & Microbiological Services

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (A/H/S)	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:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date:_____

Reviewed By:_____ Date:_____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon. BOD	Color	Nitrate	Nitrite	OPO4	Set. Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc. **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):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If not, has the cooling process begun (i.e. ice or packs present) and sample collection times <2hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: **Aqueous:** No bubble larger than a pea **Soil/Sediment:**					
Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
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					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

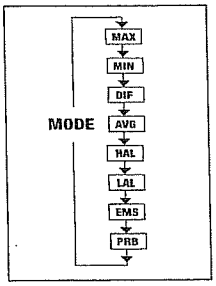
TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

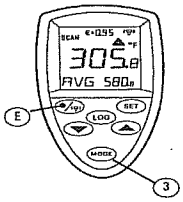
FIGURE 7

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

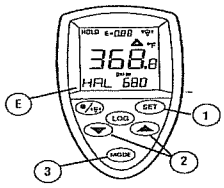
MODE Button Functions
Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information 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.

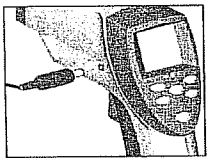
*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

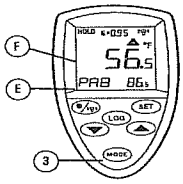




Selecting a Function
To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.







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.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No.	Site ID	Filtration Requested By:		Filtered and Preserved By:		
List Individually	(Optional)	Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

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
WW6757-9A	0.22	12-19-06		12/14/06	~~1830~~ 1400	DM
↓ -10A	0.18	↓		↓	↓	↓
WW6758-1A	4.80	↓		↓	↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓		↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	10.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-09-07		01/09/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM

REVIEWED BY: DATE:

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:
PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services
Login Chain of Custody Report (lno1)
Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services

Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: 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 SW8260FULL5ML	09-FEB-07			2		
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 SW8260FULL5ML	09-FEB-07			2		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

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?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930 →	0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0900 →	1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0900	9/20/06 1000	CP	yes no
CCAR		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900 →	1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1015	CP	yes no
Highliner		WW4992 ②	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PERCHLORE PH, SP.COND	9/21/06 1100	9/21/06 1257	↓	yes no
W&C		WW5001	BOD	9/21/06 1300		CP	yes no
Arlens		WW5016	NO3	9/22/06 1300 NO3	09/22/06 1100	KR	yes no
RAISOM		WW5010	↓	↓ ↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/22/06 1100		CP	yes no

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KATAHDIN ANALYTICAL SERVICES, INC.
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: Jal C. Burton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log-book and 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

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-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. 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 Vice President.

It is the responsibility of the Operations Manager/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 Vice President.

It is the responsibility of the Laboratory Vice 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.

5.0 REAGENTS

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 handing 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 samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

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 Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6 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.

7.9.7 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.

7.9.8 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.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

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.

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 7 and 8. 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 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.

7.12 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 Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

Figure 1 Example of Sample Disposal Logbook
Figure 2 Example of KIMS Generated Waste Disposal Report
Figure 3 Example Of Hazardous Waste Area Daily Check Documentation
Figure 4 Characteristic Toxic Hazardous Waste and TCLP concentrations
Figure 5 Example of Elementary Neutralization Logbook

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

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				
SAS783-1	WC	10-17-07	✓										1-22-08	GN
SAS786-1		10-17-07	✓											
SAS787-1,2,4		10-17-07	✓											
SAS790-1		10-19-07		✓										
SAS793-1		10-17-07	✓											
SAS795-1-9		10-23-07	✓											
SAS797-1		10-23-07	✓											
SAS798-1,2		10-25-07			✓									
SAS799-1-5		10-31-07	✓											
SAS804-1,2		10-25-07	✓											
SAS809-1,2		10-23-07	1			2				2				
SAS810-1-4	✓	10-23-07	✓										✓	

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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

Week of: 1-28, 20 08

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
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)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-28-08	[illegible] 1-29-08	[illegible] 1-30-08	[illegible] 1-31-08	[illegible] 2-01-08

Deficiency noted:					
Corrective action:					
By (Name/Date):					

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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

FIGURE 4, cont'd

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
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: 3-4-09		**Time:** 12:00	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

25
240

Date: 3-10-09		**Time:** 13:45	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

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SGS North America Inc.
Standard Operating Procedure

Standard Operating Procedures
Sample Processing

Issue date: 05/08/09
Revision: 29

SGS North America Inc.
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Updated By	050809 Date
Guy Dickinson for Kendall Sutler Name, Laboratory Manager	05-08-2009 Date
Jeanette Michaelcard Name, QA/QC Manager	5-8-2009 Date

Purpose

To describe the procedures followed for the conditioning, extraction, splitting/archiving, spiking, concentration and cleanup of samples.

Summary

The primary objective of sample processing is to quantitatively remove the analytes of interest from the matrix into an appropriate solvent, and then to remove the solvent, leaving a dried extract that can then be reconstituted prior to analysis. Samples are first conditioned using homogenization for biological samples and grinding for solids with a particle size greater than 1 mm. If appropriate, a percent solids or percent lipids determination will be made during sample conditioning. The samples are then aliquotted and fortified with extraction standard. The samples are extracted using one of a variety of procedures depending on the matrix. These include Soxhlet-Dean Stark (SDS), continuous liquid-liquid extraction (CLLE), and specialized extraction procedures for wipes, milk, oils, and synthetic precipitation. If necessary, extracts may be split after extraction, with a portion of the extract archived for future use. The resulting extracts are concentrated using mini Snyder columns (K-D) or heated nitrogen blow down (Turbovap). Concentrated extracts are subjected to cleanup procedures in order to remove potential interferences. The cleanup procedures involve column chromatography using different packing materials as appropriate. The cleanup procedures result in small volumes of extracts which are then reduced to dryness.

3.1 Sample Conditioning

3.1.1 Homogenization

3.1.1.1 Equipment/Supplies

- Heavy-duty meat grinder, freezer, tissue macerator, butcher knife, commercial food containers, suitable personal protection equipment (PPE), waste receptacle, cut-resistant gloves.

3.1.1.2 Procedure

- Prior to processing tissue samples, it must be determined as to the exact tissue that is to be analyzed. Analytical requests include whole fish, whole fish less skin, fillets, specific organs or various portions of the above. Once identified, the appropriate tissue must be frozen.
- When practical, samples are ground and homogenized while frozen.
- If specific portions must be dissected from the whole specimen the remaining tissue can be refrozen. When the entire sample, usually fish, requires analysis, reduce the sample down to manageable means using a butcher knife.

Note: Great care must be exercised when reducing a frozen fish with a knife. Cut-resistant gloves must be worn to protect the hands as well as to hold the fish in place.

- When analyzing specific portions, such as a fillet, dissect the required portion under stringent safety guidelines to protect the analyst from injury as well as to ensure the integrity of the sample.
- Once reduced in size, process the tissue through a meat grinder and collect into a suitable container.
- To ensure complete homogeneity process the ground sample two more times.

Note: Further reduction can be achieved by processing a portion of the specimen with a commercially available tissue macerator. However, continuous reprocessing of the sample through a meat grinder will often achieve the desired results.

- Record all observations.

3.1.2 Grinding

3.1.2.1 Equipment/Supplies

- Mortar and pestle, heavy-duty meat grinder, freezer, mesh sieves of various pore sizes, suitable PPE, waste receptacle.

3.1.2.2 Procedure

- After donning the appropriate PPE, open the sample in the hood.
- Remove any large objects such as rocks.
- If necessary pass the sample through a series of sieves to help facilitate the partitioning of the various particle sizes.
- Particles requiring grinding can in most instances be reduced by means of a mortar and pestle.
- In some cases reducing the temperature of the sample will aide in the grinding process.
- Some matrices such as certain papers, grains and amorphous solids need to be reduced using a Wiley Mill or other suitable mechanism.
- After sufficient reduction in the gross particle size, recombine and mix the various fractions of the sample.
- Record all observations.

3.1.3 Determination of Particle Size

3.1.3.1 Equipment/Supplies

- Clean stainless steel forceps, fume hood, aluminum foil, suitable PPE, waste receptacle.

3.1.3.2 Procedure

- Spread a piece of clean aluminum foil in an appropriately ventilated fume hood.
- After donning the appropriate PPE, open the sample in the hood.

- With the aide of forceps spread a representative portion of sample on the aluminum foil.
- Estimate the size of the particles in the sample.
- If the estimate determines that the average particle size is greater than 1 mm, then the sample must be ground before extraction.
- Record all observations.

3.1.4 Percent Solids (by Modified ASTM D2216)

3.1.4.1 Equipment/Supplies

- Drying oven, dessicator, balance, aluminum weigh boats, and glass fiber filters.

3.1.4.1.1. Solids

- Label each weigh boat required with the unique sample ID number.
- Thoroughly homogenize the sample, and grind the sample if necessary.
- Weigh out 5-20 g of sample into the weigh boat.
- Record the tare and sample weights taken and make note of any unique characteristics.
- Note the time and date, then dry the sample in a 110°C ± 5°C oven for 12 hours.
- After at least one hour, the sample may be weighted back in order to get an estimated result, as calculated below, that can be used to approximate the minimum extraction amount. If this is performed, note the time and date and record the appropriate data. Then place the sample back in the oven.
- Remove the sample and allow to cool in a dessicator.
- Note the time and date then reweigh the sample and record the dried weight.
- Calculate the % solids as follows:

$$\%solids = \frac{weight\ of\ sample\ after\ drying}{weight\ of\ sample\ before\ drying} \times 100\%$$

- Continue to re-weigh the sample, waiting at least one hour between weightings, until its mass changes by less than 1%. Use the final dried weight in the calculation of percent solids provided for sample reporting.

3.1.4.1.2. Aqueous

- Desiccate and weigh a glass fiber filter to three significant figures.
- Filter 10.0 ± 0.02 mL of well-mixed sample through the filter.
- Dry the filter a minimum of 12 H at 110°C + 5°C, and cool in a dessicator.
- Calculate percent solids by dividing the weight of solids trapped by 10, then times 100 for percent.

3.1.5 Percent Lipids

3.1.5.1 Equipment/Supplies

- balance

3.1.5.2 Procedure

- Extract the sample via the appropriate method (see Sec. 3.2.2).
- Concentrate the sample extract to "dryness" inside a tared container.
- Calculate the lipids content to the nearest three significant figures:

$$\text{Percent Lipids} = \frac{\text{Weight of Residue (in g)}}{\text{Weight of Wet Sample (in g)}} \times 100$$

3.1.6 High Moisture Procedures

- Soxhlet extraction: High moisture samples may be extracted using the Soxhlet/Dean-Stark (SDS) method (sec. 3.2.2). The Dean-Stark adapter continuously removes extracted moisture that may be drained off during the extraction.
- Filter/dual extraction: Samples that are very high in moisture content may be vacuum filtered. The filtrate may then be extracted by CLLE (sec. 3.2.5), and the filter cake may be extracted by SDS. The two extracts can then be recombined and treated as one sample. The following equation is used to calculate percent solids after filtering and drying the filter.

$$\%solids = \frac{weight\ of\ sample\ after\ drying - weight\ filter}{10g} \times 100\%$$

- Freeze Drying: Samples with less than 30% solids should be considered for freeze drying (FD). This instrument consists of a Labconco manifold (#7522700), vacuum pump (#117) and an Edwards pirani gauge (#501). 150ml – 2000mL glassware is used depending on the amount of sample to be dried. A 10g equivalent is weighted into a FD vessel of sufficient size to allow a thin shell of sample to be frozen to the walls of the vessel. Cap the vessel and store it in a freezer at a 45° angle. Prepare the FD manifold by adding dry ice to the trap using the gloves provided and filling it with ethanol. This chamber is maintained at –78°C. Attach the vacuum hose and turn the pump on. To form the shell remove the chilled samples from the freezer and roll them in the dry ice/ ethanol bath. This shell freezing increases the surface area of sample exposed to vacuum, thus creating more consistent and faster drying. After the shell has formed attach the vessel to the manifold and slowly open its valve until the vessel is under vacuum. Once all samples have been added the vacuum should be maintained at 10^{-2} torr. Repeat the shell drying for each sample. Refill the trap as needed, about every 5 hours. After 10 hours, remove each sample and check to see if they have dried completely. If so, one can store them for extraction. If not, reattach them to the manifold for another 2 hours, repeat as necessary until dry.

3.1.7 Sub Sampling

3.1.7.1 Equipment/Supplies
- Beaker, shaker, spatula.

3.1.7.2 Procedure

3.1.7.2.1. Water
- Shake sample to homogenize. Particulates must be incorporated and evenly distributed.
- A powered shaker may be used.

3.1.7.2.2. Soil
- Stirring the entire sample in its container is sufficient for wet samples.
- For dry samples, degenerate fractional shoveling is used to correctly take a representative subsample.
- Remove the sample containers cap and place it upside-down near the container and obtain a clean small beaker for the reject pile.
- Care should be taken when non-indigenous soils are handled. One must place foil down over the workspace to catch any spills. This foil and any items used in contact with the soil must be packed and properly labeled with purple stickers to insure proper disposal.
- Discard any foreign objects such as sticks, leaves, and rocks.
- The "top" layer of soil in the container is placed in the cap to prevent grab sampling errors. Mix the remaining sample in its jar.
- Proceed to place every third scoop in the subsample pile (usually a tared beaker or thimble). The other scoops go into the reject pile (the beaker obtained earlier).
- Once the desired mass of subsample has been acquired from the jar, return the reject pile to the jar. Using the cap, replace the "top" layer of soil and re-seal the jar prior to storage.
- Reference: Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, EPA/600/R-03/027, by Gerlach and Nocerino November 2003

3.2 Sample Extraction

3.2.1 Waste Dilution

3.2.1.1 Equipment/Supplies
- 60 mL vial, balance

3.2.1.2 Solvent
- Hexanes

3.2.1.3 Procedure
- Weigh out 1 gram of oil into a 60 mL vial.
- Spike the sample with the appropriate amount of extraction standard as listed in the spike profile document.
- Add 5 mL of hexanes to the vial, making sure the sample is dissolved in the solvent.
- Proceed directly to PCU cleanup.
- If the matrix is known to be exceedingly difficult, an extra dilution may be performed. After weighing out the sample, add 20 mL of hexanes before spiking. Split the diluted sample into equal

portions, spike one portion with standards, and archive the remaining portions. Then proceed to PCU cleanup.

3.2.2 Soxhlet/Dean-Stark (SDS)

3.2.2.1 Equipment/Supplies

- Complete SDS assembly, heating mantle, chiller, stainless steel spatula, thimble.

3.2.2.2 Extraction Solvents

- Dioxin solids: Toluene
- PCB solids: Methylene chloride
- All tissues: Methylene chloride

3.2.2.3 Procedure

3.2.2.3.1. Pre-extraction

For methods requiring pre-extraction, the following steps are followed:

- Add 2-4 Teflon™ chips inside the 500-mL round-bottom flask.
- Add 300 mL of extraction solvent to the flask.
- Place the thimble holder onto the flask.
- Add a thimble to the holder.
- Assemble the condenser.
- Turn on the water recirculator, which is set to 5°C.
- Turn on the heating mantle to position "64" for toluene, "50" for DCM.
- Allow the reflux to go on for at least three hours.
- Turn off the heating element, and allow the unit to cool down.
- Transfer the solvent to solvent waste.

3.2.2.3.2. Extraction

- Add 300 mL of extraction solvent to the round-bottom flask.
- Add 2-4 fresh Teflon™ chips.
- For dioxin methods, add 0.5 mL purified tridecane to the flask.
- Affix the thimble holder and insert the thimble containing the sample aliquot mixed with sodium sulfate.
- Spike the sample with the extraction standards as listed on the spike profile and allow to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- Spike the appropriate samples with matrix spike as listed on the spike profile and allow to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked.**
- Assemble the DS and condenser components (DS used for toluene extractions only).
- Turn on the water recirculator, then the heater. The heater should be set to "50" for methylene chloride, "64" for toluene.
- Insulate the round-bottom flask with foil.
- Allow the extraction to continue for 16 to 24 H.
- During the extraction, verify that the solvent cycles normally.
- Drain water from the Dean-Stark as appropriate to prevent water from returning to the boiling flask.

3.2.2.3.3. Concentration of the extract

- At the end of the reflux cycle time, proceed with the sample concentration as described below.
- **Do not turn off the heater yet.**
- Drain the toluene present in the DS side arm (if used) directly to solvent waste.
- Open the stopcock on the thimble holder and drain the extraction solvent from the soxhlet until approximately 20 mL remains inside the RB flask.
- Turn off the heater when the solvent reaches the 20-mL mark. **Do not go to "near dryness".**
- Remove the heating mantle and allow the flask to cool down.
- Transfer the sample to a 60 mL vial for Turbovap concentration (3.6.3).

3.2.3 Extraction of XAD Air Samples by SDS

3.2.3.1 Equipment/Supplies

- Complete SDS assembly, heating mantle, chiller, XAD, thimble, boiling chips.

3.2.3.2 Solvent

- Toluene, tridecane

3.2.3.3 Procedure

- Add 2-4 fresh boiling chips to each round bottom flask.

- Add 350 mL of toluene and 0.5 mL of purified tridecane.
- Adapt the thimble holder and insert thimble containing XAD.
- Spike the sample with extraction standard as listed on the spike profile and allow it to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- Spike the appropriate samples with matrix spike as listed on the spike profile and allow it to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked.**
- Assemble the DS and condenser components.
- Cover the opened extremity of the condenser with foil.
- Turn the chiller on, which is set to 5°C.
- Turn on the heating mantle, which is set at 65.
- Allow the extraction to continue for 16 to 24 hours.
- During the extraction, verify that the solvent cycles normally.
- Drain the water as appropriate to prevent water from returning to the boiling flask.
- At the end of the reflux cycle time, proceed with the sample concentration as described below.
- **Do not turn off the heater yet.**
- Drain the toluene present in the DS side arm directly to solvent waste.
- Drain the toluene present in the thimble holder to solvent waste.
- Continue to drain the toluene until approximately 20 mL of toluene remains inside the RB flask.
- Turn off the heater when the toluene reaches the 20 mL mark. **Do not go to "near dryness".**
- Remove the heating mantle and allow the flask to cool down.
- Transfer sample to 60 mL vial. Split sample and archive as necessary.
- Concentrate sample in turbovap (3.6.3).

3.2.4 Separatory Funnel Extraction

3.2.4.1 Equipment/Supplies

- 2-liter separatory funnel, glass funnels, 250-mL beakers, 90-mm filter set-up with a 1-liter vacuum container, Na_2SO_4, glass wool, 2 µm x 90-mm GMF 150 filters, wide range pH paper, 40 mL vials.

3.2.4.2 Solvent

- Methylene chloride, H_2SO_4.

3.2.4.3 Procedure

- Prepare drying funnels by packing the neck of a glass funnel with glass wool.
- Add approximately 10-15 g of Na_2SO_4 on top of the glass wool and position over an appropriately labeled collection vial.
- Add 0.1 mL of tridecane if necessary.
- Place the sample bottle on the balance and zero the scale.
- Add the contents of the sample bottle to the separatory funnel, reweigh the bottle and record weight of the displaced sample volume.
- Test the pH of the sample and record the pH on the extraction sheet.
- Add 5 mL H_2SO_4 to the sample to reduce pH to <2.
- Add extraction standard to the sample, shake vigorously and allow it to dissolve.
- Add 60 mL of CH_2Cl_2 to the separatory funnel.
- Shake for at least 2 min, periodically purging the separatory funnel of any excess pressure.
- Allow the sample-solvent solution to settle out and form a distinct interface.
- Drain the solvent through the sodium sulfate funnel into the collection vial.
- Repeat the extraction twice more, collecting all the solvent into the same vial.
- Concentrate aliquots of the sample in the Turbovap (3.6.3).
- Prepare a 25 mL sodium sulfate column as above and dry the extract.
- If necessary, decant the water present before adding the sample to the drying column.

3.2.5 Continuous Liquid-Liquid Extraction

3.2.5.1 Equipment/Supplies

- 1000 mL extraction body, drying adapter, concentrator, CLLE rack with condenser and water lines, water heater, water pump, chiller, Na_2SO_4, boiling chips, balance, deionized water, tridecane

3.2.5.2 Solvents

- Dichloromethane, H_2SO_4

3.2.5.3 Procedure

- Remove samples from designated storage area and allow to come to room temperature.
- Make sure the individual water supply valves on the CLLE rack are closed.
- Turn on the main pump valve to "extraction." This valve is located inside the cabinet.
- Open the water return valve, located on the extraction rack near the tank.
- Add several boiling chips to each concentrator.
- Attach the extraction body, drying adapter and concentrator to the CLLE rack.
- Attach the water supply and return lines to the concentrator, and open the valves.

- Turn on the pump power switch on the side of the cabinet to "extraction."
- Turn on the water heater and allow the temperature to come up to 160°C.
- Make sure the chiller is running at the proper temperature (10°C).
- Add 250 mL of dichloromethane to each extraction body.
- Check and record the pH of the sample.
- Place sample jar on the balance and zero the scale.
- Add the contents of the sample bottle to the extraction body.
- Reweigh the sample jar on the zeroed scale and record the weight of the displaced sample volume.
- Rinse the jar with 50 mL of methylene chloride and add to the extraction body.
- Add 2 mL of 5:1 H_2SO_4 to each sample to lower the pH (<2).
- Spike the sample with extraction standards as listed on the spike profile.
- Attach the condenser to the extraction body and open the stopcock.
- Allow the samples to extract for 4-6 hours.
- When the extraction is complete, close the stopcock and allow the extract to concentrate down to approximately 1 mL.
- Turn off the water heater.
- Turn off the power to the water pump.
- Close the main return valve.
- Switch the main pump valve to "drain."
- Turn on the power to the "drain" pump.
- Crack open the individual water return valves to allow air in, and water to drain out.
- Run the "drain" pump until the water has completely drained.
- Turn off the power to the "drain" pump.
- Close the individual water supply and return valves.
- Turn off the chiller.
- Transfer the extract to a 60 mL vial containing the cleanup standards and 100 uL tridecane.
- Properly dispose of the remaining sample water and dichloromethane.
- Concentrate the extract to near dryness in the Turbovap (3.6.3).

3.2.6 Milk Extraction

3.2.6.1 Equipment/Supplies

- Centrifuge, 40-mL vials, 4-oz jar, glass funnel.

3.2.6.2 Solvents/Reagents

- Hexane, Ether, Ethanol, Potassium Oxalate, sodium sulfate, Conc. H_2SO_4.

3.2.6.3 Procedure

- Add 90 mL of milk to a 4-oz jar.
- Add ES.
- Shake well and let stand for 10 min.
- Transfer the milk inside six 40-mL vials (15 g per vial).
- Add to each vial 2 mL of potassium oxalate solution (1800 mg in 12 mL water; or 20 mg per gram of milk).
- Shake.
- Add 15 mL ethanol to each vial.
- Shake.
- Add 5 mL hexane.
- Centrifuge for 15 min. @ 2000 rpm.
- Draw the hexane directly into another 4-oz jar through a sodium sulfate funnel, combining all six vials.
- Repeat the hexane/centrifuge steps two more times.
- Finally, add 5 mL diethyl ether.
- Shake.
- Centrifuge.
- Draw the organic layer and combine with the hexane layers.
- Concentrate the hexane/ether inside the Turbovap.
- Dissolve the residue in hexane (25 mL).
- Treat the hexane two times with conc. H_2SO_4 using a 40-mL vial and 10 mL of acid.
- Centrifuge as needed.
- Pull off the hexane layer and Turbovap to near dryness.

3.2.7 Fish Oil

3.2.7.1 Equipment/Supplies

- 2-liter flask, sodium sulfate columns, vessels to receive the extract (approx. 300mL), 10mL disposable pipettes, Na_2SO_4, glass wool, acidic silica gel.

3.2.7.2 Solvent

- Hexane.

3.2.7.3 Procedure

- Position columns over an appropriately labeled collection vessel containing 500µL of tridecane.
- Pipet 10g of sample into a flask. Record the actual amount. Make any notes pertaining to the sample matrix at this time (color, viscosity, etc.).
- Tilt the flask to coat the bottom with sample so that the nonane based additions of extraction standard and/or matrix spike, as listed in the spike profile, do not land on glass. (Blanks and OPRs should receive 50ml of hexane and 100 uL of corn oil). Tilt the flask again as each spike is absorbed by the oil. Record all spike information.
- Add hexane to bring volume to 200ml. Swirl the flask to incorporate the oil.
- Begin to add the silica gel a little at a time, swirling each time to prevent clumps. As much as 100g of silica gel may be needed to treat 10g of oil. A thick, dark paste will form. The static extraction time should be at least 2 minutes, swirl the flask vigorously every 30 seconds. About a minute after the last swirl the hexane will supernate and can be decanted. Pour the hexane thru the sodium sulfate columns. This decantation should go as dry as possible.
- Add 50mL of hexane, swirl and decant, again, as dry as possible. Two additional extractions, each with 10 to 20mL of hexane should be performed.

3.2.7.4 Comments

The vessel used to collect the sample under each sodium sulfate column is usually a rotovap round bottom flask. These are large enough to hold the volume of hexane used. Rotovapping the extracts down to the tridecane is recommended over evaporation. Since so much acidic silica gel is used, it is a good idea to make a fresh lot for each batch so as not to deplete our regular supply.

3.2.8 Soy/Corn Oil

3.2.8.1 Equipment/Supplies

- Recyclable glass columns, paper clips, glass wool, celite, Carbopak B, 250 mL beakers, 60 mL vials

3.2.8.2 Solvent

- Hexane
- Toluene

3.2.8.3 Procedure

- Weigh out the required amount of sample into a 250 mL beaker. Use an equal amount of hexane for the LMB, OPR and OPRD.
- Spike the samples with the extraction standards as listed on the spike profile. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- If the sample is significantly more viscous than hexane, dilute the sample with hexane (usually 1:1).
- Pack a glass column with glass wool, 3-4 cm celite, 1 gram Carbopak B, and a glass wool plug. Secure the glass wool at each end with a paper clip to prevent slipping.
- Secure the column in a clamp with the celite end up and place a "waste" beaker underneath it.
- Wet the column with hexane until it starts to drip into the waste beaker.
- Transfer the sample onto the column.
- When the entire sample has been transferred, rinse the sample beaker with 20 mL hexane and transfer to the column.
- Continue to rinse the column with hexane until all the oil has been rinsed off the column (approximately 20 mL).
- Remove the waste beaker and flip the column over so that the carbon end is up.
- Place a 60 mL vial under the column and elute with 50 mL toluene.
- Repeat with 4 more 50 mL aliquots of toluene.
- Concentrate the toluene and combine into one vial. Continue concentrating to dryness and exchange with hexane, then continue to cleanup steps.

Note: This extraction procedure was designed to accommodate large amounts of oil (50-100 g). For smaller amounts of oil, the sample may be added directly to a PCU cleanup column.

3.2.9 Wipe Extraction

3.2.9.1 Equipment/Supplies

- 4 oz. amber glass jars, filter paper, table shaker and Pasteur pipette.

3.2.9.2 Solvent

- Toluene
- Tridecane

3.2.9.3 Procedure

- Add one piece of filter paper to each jar to be used for lab QC.
- Samples received in containers suitable for use as extraction jars may be left in those containers. Samples received in foil must be transferred to jars with one solvent rinse of the foil.

- Spike the wipe with the extraction standards as listed on the spike profile and allow to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- Spike the appropriate samples with matrix spike as listed on the spike profile and allow to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked.**
- Add toluene to cover the wipe, seal, and shake for 1 hour.
- Label 60mL vial with sample ID number and add 500 uLtridecane. **Record the tridecane lot# and volume added.**
- Remove the jars from the shaker and individually transfer the solvent to the matching vial.
- Refill each jar with toluene, seal, and shake for 30 minutes.
- Observe the solvent level in each vial and concentrate as to facilitate adding the second extraction's solvent.
- Label another set of vials to hold the archived portion of each sample.
- Remove the jars from the shaker and individually transfer the solvent to the vial containing the solvent from the first extraction.
- Split the extracts equally into the archive vials. Store the archive fraction.
- Spike the vial containing the half of the extract to be processed for analysis with the cleanup standards as listed on the spike profile. **Record the CS lot#, its concentration, and the volume (µL) spiked.**
- Concentrate the extract in preparation for cleanup.

3.2.10 Synthetic Precipitation Leaching Procedure

3.2.10.1 Equipment/Supplies

- 2.2L extraction vessels, rotary agitator, filtration device, pH meter, balance, graduated cylinder, disposable lid-liners for extraction vessels, pre-cleaned 0.6-0.8µm Pyrex filters.

3.2.10.2 Reagents

- 60/40 sulfuric/nitric acid mixture, 1M sodium carbonate solution, DI water, acetone.

3.2.10.3 Procedure

- For Eastern Fluid use 20µL of the acid mix and 200µL of the Na_2CO_3 solution per liter of DI water. Stir the fluid well and make any fine pH adjustments down using 5µL of the acid mix or up using 20µL of the Na_2CO_3 solution. The pH of this fluid should be between 4.15 and 4.25.
- Only particles less than 9.5 mm in diameter should be used for the extraction. If the sample contains particles larger than this, they may either be discarded from the aliquot to be extracted or crushed to meet the requirements.
- A dry weight should be reported for each sample that is leached, unless the sample appears to have less than 10% moisture. The amount of sample used for the extraction can then be adjusted based on the amount of moisture contained in the sample.
- If a sample has a dry weight of less than 90%, the following formula must be used to calculate the minimum amount of sample to be extracted: (50g * 100/% solids) = min. amount.
- Once the appropriate weight has been calculated, use the following formula to calculate the appropriate amount of extraction fluid to add to the sample: (20 * dry weight % * actual weight used) / 100 = mL of extraction fluid
- If a sample has a dry weight equal to or greater than 90%, 50g of sample and 1000mL of extraction fluid may be assumed for the extraction.
- After adding both sample and extraction fluid to the appropriate vessel, lid-liners are placed over the opening, and the cap is screwed on over the liners. Be sure the caps are secured tightly to prevent leakage.
- Samples are tumbled using a rotary agitator for 18 ± 2 hours.
- Record the temperature of the ambient air around the rotary agitator (ref. form DC13).
- Record the times at which the agitator was both started and stopped.
- Separate the material in the extractor vessel into its liquid and solid phases by filtering through a Pyrex filter. The filter may be changed to facilitate filtration.
- All asterisked (*) data on form DC13 shall also be stored electronically. Review electronic data in the 'prep' table for accuracy and completeness.

3.2.11 Tocopherol Oil

3.2.11.1 Equipment/Supplies

- 2-liter flask, sodium sulfate, vessels to receive the extract (approx. 300mL), 10mL disposable pipettes, Na_2SO_4, glass wool, florisil.

3.2.11.2 Solvent

- Hexane.
- DCM

3.2.11.3 Procedure

- Weigh out the required amount of sample (nominally 20g for Dioxin analysis) into a 250 mL flask and add 50 mL of Hexane. Use an equal amount of hexane for the LMB, OPR and OPRD.

- Spike the samples with the extraction standards as listed on the spike profile. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- The use of mild heat and sonication may be required to speed the dissolving of the oil in the solvent.
- Pack a giant glass column with glass wool, 2 cm salt, 3" of florisil, and 1 cm of salt.
- Secure the column in a clamp.
- Position a waste collection beaker.
- Wet the column with 30 mL hexane until it starts to drip into the waste beaker.
- Transfer the sample onto the column.
- When the entire sample has been transferred, rinse the sample beaker with 2 mL hexane and transfer to the column.
- Continue to rinse the column with hexane until all the oil has been rinsed off the column (approximately 60 mL).
- Remove the waste beaker and position a sample collection flask.
- Elute the sample with 240 mL dichloromethane.
- Concentrate the DCM and combine into one 60 mL vial.
- Add 50 µL of keeper to each vial. Continue concentrating to dryness and exchange with hexane, then continue to cleanup steps.

Note: This extraction procedure was designed to accommodate large amounts of oil (5-20 g). For smaller amounts of oil, the sample may be added directly to a PCU cleanup column.

3.3 Splitting/Archiving Extracts

3.3.1 Supplies

- Graduated tube, centrifuge tube, Pasteur pipette, glass wool.

3.3.2 Procedure

- Label the graduated and centrifuge tubes as required.
- Using a Pasteur pipette and a glass wool plug, filter the tridecane residue obtained from after concentration directly to the graduated or calibrated tube (e.g., 3.5- and 7-mL marks).
- Add approximately 1 mL of hexane to the sample container, swirl the solvent and transfer to the Pasteur pipette.
- Add another 1 mL hexane inside the RB flask, swirl, and wash the RB flask walls using the pipette.
- Transfer the rinse to the Pasteur pipette.
- Collect all rinses directly in the graduated tube.
- Make sure the volume reaches the 7-mL mark.
- Transfer 3.5 mL to the centrifuge tube or any container suitable for the fractionation.
- Seal the graduated tube using a Teflon™-lined cap and store the sample inside the refrigerator.

Notes:

- To prevent sample spills, always secure the sample tube and the Pasteur pipette to a rod using easily adjustable clamps.
- In the presence of particulates, it is necessary to rinse the particulates with toluene, and repeat the concentration and solvent exchange steps. **Do not Split/Archive an inhomogeneous sample.**

3.4 Spiking

3.4.1 Equipment/Supplies

- 10 µl – 1000 µl automated pipette.

3.4.2 Procedure

- In each case, add the appropriate amount of standards for the method in use. A 'witness' must be present.
- Note: the composition and amount of standards used may be subject to change based on method requirements and individual client needs. Refer to the spike profile document and project folder for any changes.
- If the sample to be spiked is depleted or if only a limited volume remains, the witness must verify the ID of the sample prior to spiking. The extractionist is responsible for identifying such samples to the witness.
- If the samples to be spiked are air samples (i.e. M23 or TO9A) then the witness must complete a separate form detailing further duties.

3.5 Pipette Calibration

3.5.1 Equipment/Supplies

- 10 µl – 1000 µl automated pipette.
- Analytical Balance
- D.I. water

3.5.2 Procedure

- Pipettes are to be calibrated quarterly.
- The pipettes are calibrated at the high and low volume settings that are provided for each pipette model in the manual.
- D.I. water is transferred into the analytical balance and the results recorded in the Wet Lab Maintenance Logbook five times for each volume setting.

- The average of the five replicates must fall within the acceptable range provided in the pipette manual for each model and volume.
- Pipettes that pass both volume settings will be labeled with the data of calibration and the initials of the calibrator.
- Pipettes that fail will be removed from service until an acceptable calibration is performed.
- If the calibration passes one calibration range but not the other, that pipette may be used for the passing volume only.
- New or repaired pipettes must pass fully before entering service.

3.6 Concentration

3.6.1 Turbovap

3.6.1.1 Equipment

- Zymark Turbovap, vial racks.

3.6.1.2 Procedure

- Set the water bath to the appropriate temperature for the solvent used and allow the bath to come to temperature (approximately 45°C for hexane and methylene chloride, 65°C for toluene).
- Clean the nitrogen needles with CH_2Cl_2 before use.
- Place the 40- or 60-mL sample vials into the TurboVap. (Note: Be sure the correct vial rack is used.)
- Set the nitrogen at 1-2 psi, and set the time to allow the samples to evaporate down 5-10 mL. This will help prevent cross contamination. Once the samples have evaporated enough that solvent will not splash out of the vials, set the nitrogen at 5 psi and set the time to allow the sample to evaporate to near dryness. The TurboVap will beep to signal that time is up. Additional time may be added if samples are not completely evaporated.
- Once the samples have evaporated, remove the vials and rinse the nitrogen needles with toluene.

3.7 Sample Cleanup

3.7.1 Conventional

3.7.1.1 Equipment/Supplies

- Pipettes (25 mL and 10 mL), Turbovap, acid- and base-coated silica, alumina, Florisil, carbon/celite.

3.7.1.2 Solvent

- Hexane, methylene chloride (MC), toluene.

3.7.1.3 Procedure

- Assemble the acid/base silica column in a 25 mL pipette as follows from bottom: glass wool, 1 g SiO_2, 4 g NaOH coated silica, 1 g SiO_2, 8 g H_2SO_4 coated silica, 2 g SiO_2, 4 g Na_2SO_4.
- Wash column with 50 mL hexane.
- Load sample and two 1-mL rinses of hexane.
- Elute with 100 mL hexane.
- The eluate is concentrated to near dryness and is now ready for the alumina column.
- The alumina column is prepared in a 10 mL pipette using 6 g acidic alumina and 4 g sodium sulfate.
- Load sample and two hexane rinses onto alumina column.
- Wash with 2%.
- Elute with MC to collect dioxins/furans.
- Turbovap to near dryness.
- Assemble the carbon column in a 10 mL pipette using 6 g carbon/celite with glass wool plugs.
- Load the sample onto the column with hexane, and allow the hexane to run completely through the column.
- Invert the column and elute with toluene.
- Turbovap the sample in toluene to dryness.

3.7.2 PCU-F

3.7.2.1 Equipment/Supplies

- Recyclable glass columns, Turbovap, acid- and base-coated silica, sodium sulfate, Florisil (store supplies inside dessicator).

3.7.2.2 Solvent

- Hexane, DCM.

3.7.2.3 Procedure

- Assemble the acid/base silica column.
- Assemble the florisil column.

  Note: For tissue samples (e.g., fish) use the anthropogenic isolation column.
- Assemble waste collection setup.

- Wet columns with hexane.
- Fill 2% line.
- Flush center fitting.
- Load sample plus two vial rinses.
- Elute acid base column with hexane.
- Elute florisil with 2%.
- Position collection vial.
- Elute sample with 100% CH_2Cl_2.

Note: To perform method, start PCU software, load method APC-2000.txt, and hit start. When method is complete, concentrate samples using Turbovap.

3.7.3 PCU-A

3.7.3.1 Equipment/Supplies

- Recyclable glass columns, TurboVap, acid- and base-coated silica, sodium sulfate, alumina (store supplies inside dessicator).

3.7.3.2 Solvent

- Hexane, Methylene Chloride.

3.7.3.3 Procedure

- Assemble the acid/base silica column.
- Assemble the alumina column.

Note: For tissue samples (e.g., fish) use the anthropogenic isolation column.

- Assemble waste collection setup.
- Wet columns with hexane.
- Fill 2% line.
- Flush center fitting.
- Load sample plus two vial rinses.
- Elute acid base column with hexane.
- Elute alumina with 2%.
- Position collection vial.
- Elute sample with 100% CH_2Cl_2.

Note: To perform method, start PCU software, load method PCU-abal.txt, and hit start. When method is complete, concentrate samples using Turbovap.

3.7.4 Acid Treatment for Lipids

3.7.4.1 Equipment/Supplies

- Separatory funnel (250 mL), vacuum concentrator (VC), glass funnel, rotovap flask, conc. H_2SO_4, sodium sulfate.

3.7.4.2 Solvent

- Hexane.

3.7.4.3 Procedure

- This section assumes that the extraction was completed with methylene chloride and the solvent **completely** removed under reduced vacuum. The lipids content of the sample is determined before engaging in the step that follows.
- Use 200 mL hexane per 10 g of lipids/fat.
- Add the hexane to the fat and allow the fat to dissolve completely.
- Transfer to a 250-mL separatory funnel and add two 5-mL rinses.
- If the volume of hexane is above 200 mL, repeat what follows on 100-mL (or less) portions.
- Add carefully 50 mL of conc. H_2SO_4 (or 50 mL per 100 mL hexane or 10 g fat).
- Mix gently the two phases; do not shake hard to prevent the formation of emulsions. Tipping the separatory funnel and releasing vapors is done gently several times. Let the system settle before drawing the lower acid layer to wastes. Repeat this step two more times with another 2 x 50-mL of acid. All collected acid layers are discarded slowly in the sink by allowing tap water to run abundantly.
- The hexane layer is passed through a sodium sulfate funnel directly into a rotovap flask.

3.7.5 Florisil Column Cleanup

3.7.5.1 Equipment/Supplies

- Recyclable glass columns, Turbovap, sodium sulfate, Florisil (store supplies inside dessicator).

3.7.5.2 Solvent

- Hexane, Methylene Chloride.

3.7.5.3 Procedure

- Assemble the bottom column as follows from bottom: filter, florisil.

- Assemble the top column as follows from bottom: filter, sodium sulfate.
- Position waste collection assembly.
- Wet columns with 2%.
- Wet columns with hexane.
- Load sample plus two vial rinses.
- Elute florisil column with hexane.
- Elute with 2%.
- Position collection vial.
- Elute sample with 100% CH_2Cl_2.

Note: To perform method, start PCU software, load method fonly3.txt, and hit start. When method is complete, concentrate samples using Turbovap.

3.7.6 Alumina Column Cleanup

3.7.6.1 Equipment/Supplies

- Recyclable glass columns, Turbovap sodium sulfate, alumina (store supplies inside dessicator).

3.7.6.2 Solvent

- Hexane, Methylene Chloride.

3.7.6.3 Procedure

- Assemble bottom column as follows from bottom: filter, alumina.
- Assemble top column as follows from bottom: filter, sodium sulfate.
- Position waste collection assembly.
- Fill 2% line.
- Wet columns with hexane.
- Load sample plus two vial rinses.
- Elute alumina column with hexane.
- Elute with 2%.
- Position collection vial.
- Elute sample with 100% CH_2Cl_2.

Note: To perform method, start PCU software, load method a-only.txt, and hit start. When the method is complete, concentrate samples using Turbovap.

3.7.7 Carbon Cleanup

3.7.7.1 Equipment/Supplies

- Disposable glass pipettes, glass wool, celite, carbon/celite mixture

3.7.7.2 Solvent

- Methylene Chloride, Toluene, Binary mixture (50% Methylene Chloride:50% Hexane), Tertiary mixture (75% Methylene Chloride:20% Methanol:5% Toluene)

3.7.7.3 Procedure

- Assemble the carbon column by placing a glass wool plug in the disposable pipette, add 5 mm of celite, 5 mm carbon/celite mixture, then another plug of glass wool.
- Secure the column in a clamp and place a "waste" jar underneath the column. The column should be positioned so that the celite end is up.
- Rinse the column with 4 mL of toluene. Allow the solvent to run completely through the column. Continue to rinse the column with 2 mL of tertiary, 4 mL of binary, and 2 ml methylene chloride. Be sure to let the solvent run completely through the column between each rinse, but do not let the column go dry.
- Load the sample onto the column with three 1-2 mL rinses of methylene chloride.
- Rinse the column with 10 mL binary, then 5 mL tertiary.
- Flip the column over so that the celite end is down. Remove the waste jar and position a collection vial underneath the column.
- Elute the sample off the column with 20 mL toluene.

3.7.8 Drinking Water Cleanup

3.7.8.1 Equipment/Supplies

- 25 mL drying column, H_2SO_4 treated silica gel, glass wool, sodium sulfate, 60mL vial.

3.7.8.2 Solvent

- Methylene chloride

3.7.8.3 Procedure

- Assemble in a 25 ml column as follows from the bottom: glass wool, 5mm of neutral silica, 20mm of florisil, 5mm neutral silica, 65mm of H2SO4 coated silica and 15mm sodium sulfate.
- Pre-clean the column with two 10mL rinses of DCM and drain into the appropriate solvent waste container.

- Place a 60mL vial under column.
- Load sample with methylene chloride.
- Rinse concentrator with three 2mL rinses of methylene chloride.
- Rinse column twice with 10mL of methylene chloride.
- Turbovap the sample to .5mL and transfer to GC vial.

3.7.9 Wipe Cleanup

3.7.9.1 Equipment/Supplies

- Silica gel
- Acid Treated silica gel
- Florisil
- 10mL disposable pipette
- Glass wool

3.7.9.2 Solvents

- Dichloromethane
- Hexane

3.7.9.3 Procedure

- Ensure that a keeper is present in the samples. Blow them down and exchange into hexane.
- Add a glass wool plug to each column to be prepared and attach it to the rack.
- Fit a funnel into the end of each column to facilitate the addition of cleanup media.
- Add a "Pinch" of untreated silica. (Measuring scoops provided.)
- Add a "Dash" of florisil.
- Add a "Pinch" of acid treated silica.
- Add a "Pinch" of sodium sulfate.
- Position waste collection vessel(s).
- Wet the column with hexane, and then add the sample.
- Add 2mL of Hexane and allow the eluent to flow to waste.
- Add 4 x 5mL aliquots of 2%DCM/Hexane.
- Position sample collection vial.
- Add 4 x 5mL of DCM.
- Turbovap the sample to 0.5 mL and transfer to GC vial.

3.7.10 Florisil Cleanup for Method 1668A

3.7.10.1 Summary

PCB sample extract cleanup is accomplished by passing the extract through the column under controlled conditions described below.

3.7.10.2 Equipment/Supplies

- Florisil column (sec.2.12)
- Labeled 60mL reusable vials with snyder column
- Disposable glass pipettes
- Water bath
- Fume hood
- Tridecane
- Teflon boiling chips

3.7.10.3 Solvent

- Hexane
- 6% ether:hexane

3.7.10.4 Procedure

- Position waste collection vial under florisil column.
- Wet column with 20mL hexane and allow solvent to flow to waste.
- Wet column with 10mL hexane and allow solvent to flow to waste.
- Transfer sample from concentrator to column using disposable pipet.
- Rinse the sample vial twice with 1mL portions of hexane and apply to column.
- Position properly labeled vial under column.
- Elute sample from column with 50mL of 6% ether:hexane.
- Position 2nd labeled vial under florisil and elute with another 50mL of 6% ether:hexane.
- Add boiling chips to collection vials.
- Both vials should be concentrated using snyder column and water bath inside a fume hood.
- Vials should be combined and transfered to a labeled GC vial containing 20µL of tridecane.

3.7.11 Acid silica cleanup for 1668A (mini silica)

3.7.11.1 Summary

PCB sample extract cleanup is accomplished by passing the extract through the column under controlled conditions described below.

3.7.11.2 Equipment/Supplies

- 25mL acid silica cleanup column (sec. 2.13)
- Disposable pipettes
- Water bath
- Fume hood
- Tridecane
- Turbo evaporator
- 60mL reusable vials with snyder column

3.7.11.3 Solvent

- DCM

3.7.11.4 Procedure

- Position waste collection vial under silica column.
- Rinse the column with 10mL of DCM and allow to flow to waste.
- Rinse the column with 5mL of Hexane and allow to flow to waste.
- Position properly labeled vial under column.
- Load sample directly from concentrator.
- Rinse concentrator with 2x 1mL rinses of DCM and apply to column.
- Flush column with 3mL of Hexane.
- Vial should be concentrated using the turbovap.
- Sample should be transferred to a labeled GC vial containing 20µL of tridecane.

3.7.12Acid silica with florisil cleanup for dioxins

3.7.12.1 Summary

Dioxin sample extract cleanup is accomplished by passing the extract through the column under controlled conditions described below.

3.7.12.2 Equipment/Supplies

- 25mL drying column
- Florisil
- Acid coated silica gel
- Silica gel
- Sodium sulfate
- Glass wool
- Disposable pipettes
- Turbo evaporator
- 60mL vials.

3.7.12.3 Solvent

- Hexane
- Dichloromethane

3.7.12.4 Procedure

- Assemble the column as follows from bottom: glass wool, 5mm silica, 15mm florisil, 5mm silica, 80mm acid silica, 10mm sodium sulfate.
- Position waste collection vial under silica column.
- Rinse the column with 2x 10mL of dichloromethane and allow to flow to waste.
- Rinse the column with 2x 10mL of hexane and allow to flow to waste.
- Load the sample from the 60mL vial.
- Rinse vial with 2x 1mL rinses of hexane and apply to column.
- Flush column with 2x 20mL of hexane.
- Position properly labeled 60mL vial under column.
- Elute sample with 50mL of dichloromethane.
- Vial should be concentrated using the turbovap.
- Sample should be transferred to a labeled GC.

SGS North America Inc.
Standard Operating Procedure

Standard Operating Procedures
For Analysis and Reporting (Method 1613)

Issue date: 04/21/09
Revision: 13

SGS North America Inc.
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Updated By	042209 Date
Name, Laboratory Manager	4/23/09 Date
Jeanne Michelland Name, QA/QC Manager	4-22-2009 Date

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Purpose

To describe the processes used in operating the HRGC/HRMS system, as well as the procedures followed in the generation, interpretation and review of laboratory data for Method 1613.

Summary

This SOP details how to analyze and report samples by EPA Method 1613. HRGC/HRMS is used to detect and quantitate PCDD/Fs. Samples arrive at the MS lab having been extracted and fractionated using procedures in Section 3. Analyses are grouped into 12-hour sequences that include analyses of samples and standards mixtures. Upon completion of the sequence, the analyst reviews the data associated with both standards and samples in order to confirm the validity of the sequence and to determine any potential need for re-analysis or re-extraction. The analyst generates quantitation reports and chromatograms using sophisticated software. These reports are used to generate forms that summarize the results of the analysis.

4.1 Operation of HRGC/HRMS

4.1.1 Equipment

- HP6890 GC, Micromass Autospec Ultima high resolution mass spectrometer, vortex mixer, 10-100 uL pipette

4.1.2 Procedure

- Recall the GC temperature/pressure/flow program.
- Recall the MS experiment (see Table 1).
- Perform any necessary maintenance.
- Tune the MS resolution to 100 ppm at 5% height.
- Acquire location data to calibrate the MS and print a copy of each function's MS resolution.
- Inject the window defining/GC resolution/continuing calibration mix (RETCON). Evaluate descriptor-switching times for accuracy. If any window defining peaks have shifted outside the descriptor windows, adjust the switching times before injecting any samples. This injection is also used to verify that there is less than or equal to 25% peak to valley for the two close eluters of 2,3,7,8-TCDD. Print a copy of the GC resolution check. If the valleys are within specifications, proceed to calibrate or verify a previous calibration. If not, further investigation and/or maintenance may be required. Re-inject this solution after maintenance to check for improvement.
- Now that the GC/MS resolution and descriptor switching times have been verified, a series of five initial calibration standards may be injected and reviewed for method requirements. If an initial calibration already exists, a RETCON may be analyzed to verify continuing calibration. If the curve or the RETCON passes method requirements, sample analysis may begin.
- Reconstitution of a sample is accomplished by adding nonane containing the injection standards, capping the vial, and mixing well with a vortex mixer. The amount of standard to be added can be found in the spike profile document.
- Samples are injected under conditions identical to those used to establish calibration.
- A "back-end" print out of the MS resolution must be performed.
- The calibration data from a sequence is filed in a folder cabinet under the day it was analyzed and includes the all GC/MS resolution checks, window verification, valley verification, front end Retcons, run logs and window defining mix (WDM) retention time sheets.
- Each sample hardcopy should include the quant report, totals pages, deviations, chromatograms, and report forms.
- Columns: DB-225, 30 m, id 0.25 mm, 0.25 μm; DB-5MS, 60 m, id 0.25 mm, 0.25 μm.

Table 1: Mass Descriptors used for Selected Ion Recording HRMS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	100	20
1	2	305.8987	100	10
1	3	315.9419	40	10
1	4	316.9824	20	10
1	5	316.9824	(Lock)	50
1	6	317.9389	40	10
1	7	319.8965	100	10
1	8	321.8936	100	10
1	9	327.8847	40	10
1	10	331.9368	40	10
1	11	333.9339	40	10
1	12	375.8364	30	20
2	1	339.8597	100	20
2	2	341.8568	100	10
2	3	351.9000	40	10
2	4	353.8970	40	10
2	5	355.8546	100	10
2	6	357.8517	100	10
2	7	366.9792	20	10
2	8	366.9792	(Lock)	50
2	9	367.8949	40	10
2	10	369.8919	40	10
2	11	409.7974	30	20
3	1	373.8207	100	20
3	2	375.8178	100	10
3	3	380.9760	20	10
3	4	380.9760	(Lock)	50
3	5	383.8639	40	10
3	6	385.8610	40	10
3	7	389.8156	100	10
3	8	391.8127	100	10
3	9	401.8559	40	10
3	10	403.8530	40	10
3	11	445.7555	30	20
4	1	407.7818	100	20
4	2	409.7788	100	10
4	3	417.8253	40	10
4	4	419.8220	40	10
4	5	423.7767	100	10
4	6	425.7737	100	10
4	7	430.9728	20	10
4	8	430.9728	(Lock)	50
4	9	435.8169	40	10
4	10	437.8140	40	10
4	11	479.7165	30	20
5	1	441.7427	100	20
5	2	443.7398	100	10
5	3	454.9728	20	10
5	4	454.9728	(Lock)	50
5	5	457.7377	100	10
5	6	459.7348	100	10
5	7	469.7780	40	10
5	8	471.7750	40	10
5	9	513.6775	30	20

4.2 Data Generation, Interpretation and Review

SGS North America, Inc. defines a batch of samples as no more than 20 samples processed within a 12-hour shift. One LMB and one OPR are processed per analytical batch, following the same procedures as the field samples. Generally, soil is replaced by salt (Na_2SO_4), effluent by deionized water and biological tissues by vegetable oil. An invalid LMB or OPR requires a re-extraction of the affected samples.

4.2.1 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. The use of high purity reagents and solvents helps minimize interference problems. All new materials used in the analysis shall be demonstrated to be free from interferences by running an initial reagent blank. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the analytes. The elimination of interferences is essential. Cleanup steps are used to reduce or eliminate these interferences and thereby permit reliable determination of the analyte as close as possible to the specified sensitivity.

4.2.2 Method Detection Limit Study and IPR Schedule

On an annual schedule, the laboratory shall perform Method Detection Limit studies (MDLs) for each matrix analyzed, as well as for each extraction method utilized per matrix. All MDL studies will be conducted following the guidelines set forth in 40 CFR, Part 136, appendix B and must be lower than one-third the regulatory compliance level or one third the Minimum Levels (ML) set forth in Table 2 of the reference method. In addition, at the time of the MDL a continuing calibration will be analyzed at a concentration other than the regularly utilized CS3, to further demonstrate instrument performance.

Each analyst is required to successfully complete an Initial Demonstration of Precision and Accuracy (IPR) before working on samples. An IPR is conducted for each matrix and extraction technique utilized. IPRs are repeated with each relevant change to a technique.

4.2.3 Initial Calibrations

The percent relative standard deviations for the mean response factors from the seventeen unlabeled standards must not exceed +/- 20%. The percent relative standard deviations from the labeled standards (i. e. extraction standards, cleanup standards and sampling standards) must not exceed +/- 35%. The signal to noise ratio for all signals present must be $\geq$ 10. The ion abundance ratios must be within specified control limits (see Table 2). SGS North America Inc. uses the concentrations in Table 3 to construct the initial calibration. The concentration of the lowest calibration point is a modification of the referenced method. It is lower than required.

Table 2. Theoretical Ion Abundance Ratios and Their Control Limits

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6[a]	0.51	0.43	0.59
7	1.04	0.88	1.20
7[b]	0.44	0.37	0.51
8	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF
[b] Used only for ^{13}C-HpCDF

A new initial calibration is required when the continuing calibration criteria below are not met. Routine maintenance may be performed to correct any failures. Any major maintenance to the analytical system such as slit cleaning, analyzer lens cleaning, magnet shifts, and detector disk changes warrant a new ICAL. At a minimum, a new initial calibration must be performed annually.

Table 3. Initial Calibration Concentrations

Analyte	CS-1	CS-2	CS-3	CS-4	CS-5
	Concentration (pg/µL)				
Unlabeled					
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
Extraction Standards					
^{13}C-2378-TCDD	100	100	100	100	100
^{13}C-2378-TCDF	100	100	100	100	100
^{13}C-12378-PeCDD	100	100	100	100	100
^{13}C-12378-PeCDF	100	100	100	100	100
^{13}C-23478-PeCDF	100	100	100	100	100
^{13}C-123678-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-1234678-HpCDD	100	100	100	100	100
^{13}C-1234678-HpCDF	100	100	100	100	100
^{13}C-1234789-HpCDF	100	100	100	100	100
^{13}C-OCDD	200	200	200	200	200
Cleanup Standards					
^{37}Cl-2378-TCDD	0.25	2	10	40	200
Injection Standards					
^{13}C-1234-TCDD	100	100	100	100	100
^{13}C-123789-HxCDD	100	100	100	100	100

4.2.4 Continuing Calibrations

Check that all paperwork is present. A CCal package should contain the documentation listed below.

- Pass: Run log. HRMS Resolution Checks. WDM retention time sheet. WDM chromatograms. GC performance for 2,3,7,8-TCDD. CCal quantitation page. CCal chromatograms. Injection preparation log.
- Fail: The analyst listed on the run log can provide any missing paperwork.

Review the Run log.

- Pass: Check that the 12-hour windows have not been exceeded between the front end Ccal and the last sample of the sequence.
- Fail: Re-analysis of affected samples.

Review the HRMS Resolution checks.

- Pass: Verify 100ppm width at 5% height for PFK mass 318 or higher. Compare the resolution check times to those on the run log to be sure they bracket each sequence.
- Fail: If instrument resolution drops below 10,000 at any point during a 12-hour sequence, the samples from the sequence should be reanalyzed.

Review the Window Defining Mix and GC Performance Documentation.

- Pass: Check that the sample numbers on the WDM sheets match those on the run log. Check that the retention times are correct for the WDM chromatograms.
- Check that the valley between 2,3,7,8-TCDD and its close eluters does not exceed 25%.
- Fail: Any missing peaks in the window-defining sample should be re-identified with a survey scan. Determine proper switching times. These must be entered into the HRMS ion function descriptors before analysis may resume. If the GC performance valley is greater than 25% instrument maintenance may be required. When a valley fails all samples must be reinjected.

Review the CCal Quantitation and Chromatograms.

- Pass: Check that all ion ratios are in specification. Verify that all compounds are within the concentration limits set by the method (see Table 4) for all front end Ccals.
- Fail: Routine instrument maintenance such as installing new injection port hardware, inner source cleaning, retuning, column clipping etc. will usually correct a calibration failure. If these measures do not work, then the laboratory will demonstrate performance after corrective action with either two consecutive acceptable CCALs or a new initial calibration.

Compound Name	CCAL (pg/µL)	Limits (pg/µL)	Compound Name	CCAL (pg/µL)	Limits (pg/µL)
2,3,7,8-TCDD	10	7.8 - 12.9	$^{13}C_{12}$-2,3,7,8-TCDD	100	82 - 121
1,2,3,7,8-PeCDD	50	39 - 65	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	62 - 160
1,2,3,4,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	85 - 117
1,2,3,6,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	85 - 118
1,2,3,7,8,9-HxCDD	50	41 - 61	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	72 - 138
1,2,3,4,6,7,8-HpCDD	50	43 - 58	$^{13}C_{12}$-OCDD	200	96 - 415
OCDD	100	79 - 126	$^{13}C_{12}$-2,3,7,8-TCDF	100	71 - 140
2,3,7,8-TCDF	10	8.4 - 12	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	76 - 130
1,2,3,7,8-PeCDF	50	41 - 60	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	77 - 130
2,3,4,7,8-PeCDF	50	41 - 61	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	76 - 131
1,2,3,4,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	70 - 143
1,2,3,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	74 - 135
2,3,4,6,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	73 - 137
1,2,3,7,8,9-HxCDF	50	44 - 57	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	78 - 129
1,2,3,4,6,7,8-HpCDF	50	45 - 55	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	77 - 129
1,2,3,4,7,8,9-HpCDF	50	43 - 58	$^{37}Cl_4$-2,3,7,8-TCDD	10	7.9 - 12.7
OCDF	100	63 - 159			

Table 4. Continuing Calibration Limits

Review the Injection Prep log sheet.

- Pass: Check that all samples have been spiked with 2 ng injection standard. Verify that final volume is 20 uL. Be sure that any dilutions or other comments are noted.
- Fail: Calculations of sample concentrations should reflect any deviations from normal injection prep parameters.

4.2.5 Quality Control Work Groups

The following elements should be present in a complete work group file:

- LMB topsheets
- LMB totals sheets

- LMB chromatograms
- OPR topsheets
- OPR chromatograms
- Extraction log sheet
- Cleanup log sheet
- Cleanup observation forms
- Dry weight sheet (where applicable)
- Any additional information (ex. re-extract request sheet)

The following procedure should be used for reviewing a work group:

- Review the header information on the LMB topsheets. Verify that the method and client sample ID (LMB or OPR) are correct.
- Review the footer information on the LMB and OPR topsheets. Verify that the following information is correct: SGS North America Inc. sample ID or OPR project number, extraction date, analysis date, method, matrix, sample weight/volume, percent solids/lipids, pH, work group number, sample datafile, retcheck datafile, beginning cal datafile and ICal datafile.
- Verify that no target analytes are present in the LMB above Method 1613's Minimum Levels. If target analytes are above this limit, the associated samples must have concentrations that exceed 10 times the LMB concentration for the specified analyte. Otherwise, samples must be re-extracted.
- Review the totals data for the LMB. Be sure that any ghosting peaks are removed from the totals concentrations and the associated detection limits are elevated to reflect the subtracted peaks.
- Verify that extraction and cleanup standard recoveries are within method specifications (see Table 5) for the LMB and OPR. These recoveries are found on the topsheets. Validate any failures based upon signal to noise and acceptable detection limits. If the lab validation fails a corrective action is required. Corrective actions may include re-extraction, re-cleanup, lower sample volume, extract dilution, etc.
- Verify that the recoveries in the OPR meet SGS North America Inc.'s recovery limits, found in Table 6.

Compound Name	Amount Spiked (pg/µL)	Limits %
$^{13}C_{12}$-2,3,7,8-TCDD	100	25 - 164
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25 - 181
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32 - 141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28 - 130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23 - 140
$^{13}C_{12}$-OCDD	200	17 - 157
$^{13}C_{12}$-2,3,7,8-TCDF	100	24 - 169
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24 - 185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21 - 178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26 - 152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26 - 123
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	29 - 147
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	28 - 136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28 - 143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26 - 138
$^{37}Cl_4$-2,3,7,8-TCDD	10	35 - 197

Table 5. Labeled Standard Recovery Limits

Analyte	Amount Spiked (pg/µL)	Limit (pg/µL)
2378-TCDD	10	6.7-15.8
12378-PeCDD	50	35-71
123478-HxCDD	50	35-82
123678-HxCDD	50	38-67
123789-HxCDD	50	32-81
1234678-HpCDD	50	35-70
OCDD	100	78-144
2378-TCDF	10	7.5-15.8
12378-PeCDF	50	40-67
23478-PeCDF	50	34-80
123478-HxCDF	50	36-67
123678-HxCDF	50	42-65
123789-HxCDF	50	39-65
234678-HxCDF	50	35-78
1234678-HpCDF	50	41-61
1234789-HpCDF	50	39-69
OCDF	100	63-170
^{13}C-2378-TCDD	100	20-175
^{13}C-12378-PeCDD	100	21-227
^{13}C-123478-HxCDD	100	21-193
^{13}C-123678-HxCDD	100	25-163
^{13}C-1234678-HpCDD	100	26-166
^{13}C-OCDD	200	26-397
^{13}C-2378-TCDF	100	22-152
^{13}C-12378-PeCDF	100	21-192
^{13}C-23478-PeCDF	100	13-328
^{13}C-123478-HxCDF	100	19-202
^{13}C-123678-HxCDF	100	21-159
^{13}C-123789-HxCDF	100	17-205
^{13}C-234678-HxCDF	100	22-176
^{13}C-1234678-HpCDF	100	21-158
^{13}C-1234789-HpCDF	100	20-186
^{37}Cl-2378-TCDD	10	3.1-19.1

Table 6. OPR Recovery Limits

4.2.6 Data Review

4.2.6.1 Procedure

For quick reference, refer to the chart below.

4.2.6.2 Initial Review

- Summary form generation and organization of Ical, Ccal, Samples and QC data is conducted by the analyst.
- Analysts review data upon generation and elicit peer review.
- Data is then released to a secondary review level with the analyst's signature.
- Assemble project samples and QC data.

4.2.6.3 Final Review

- Review client information and documentation.
- Complete Data Review Checklist (Section 4, Appendix B) and sign reports.
- Samples are released to clients with the data reviewer's signature after the complete package is reviewed by a QA Manager or Lab Director.

SGS Quality Control Requirements for Method 1613			
QC Check	**Frequency**	**Limits**	**Corrective Action**
Method Blank	One per extraction batch.	PCDD/F< ML or <10% of level in sample.	Affected samples must be re-extracted.
Initial Calibration (ICAL)	As needed to maintain acceptable CCAL, after major maintenance, or at a minimum one per year.	20% RPD for native and 35% RPD for labeled species.	An acceptable ICAL must be established before sample analysis may begin.
Mass Resolution	Before and after each 12 hour analytical sequence.	Mass resolution must be at 10,000 as estimated from a printout of a PFK peak.	MS maintenance.
Descriptor Defining Isomers	At the beginning of each 12h analytical sequence.	First and last eluters must be present within the switching times.	Perform a survey scan to identify the correct switching times.
GC Performance Isomers	At the beginning of each 12h analytical sequence.	Must be <25% valley.	GC maintenance.
OPR (LCS)	One per extraction batch.	See Table 6	Re-extract batch if low and detected. Evaluate data quality for high and non-detected.
OPRD (LCSD)	One per extraction batch.	See Table 6, ≤ 20%RSD.	If needed and fails, re-extract batch.
Extraction Standards (ES)	Every sample must receive ES before extraction.	See Table 5.	Evaluate data quality, and if needed re-extract sample.
Continuing Calibration (CCAL)	At the beginning of sample analysis every 12h.	See Table 4.	Acceptable CCAL must be established before sample analysis may begin.
Lab Sample Duplicate (DUP)	Per client request.	<25%Difference.	Flag failures.
Matrix spike/duplicate (MS/MSD)	One pair per method, per matrix, per extraction technique, per 30 days, per 20 samples.	±25% recovery, 20% RPD.	Flag failures, report OPRD.

4.2.7 Calculations

4.2.7.1 Target compound calculation

- PCDD/PCDF (ppt) = $\frac{\text{(Sum Ion Abun. of analyte)(ES Amount)}}{\text{(Sum Ion Abun. of Int. Std)(RRF from ICal)(Amt. of Sample)}}$

- EDL = $\frac{3\,\text{(Sum Height of Noise)(Std. Amount)}}{\text{(Sum Height of Int. STD.)(RF from ICal)(Amt. of Sample)}}$

The instrumentation software calculates the noise level. However, manual noise determination may be employed at the reviewer's discretion in order to more accurately report peaks of interest.

4.2.7.2 Extraction Standard Recovery Calculation

- % Recovery = $\frac{\text{(Sum Ion Abun. of ES)(JS Amount)}}{\text{(Sum Ion Abun. of JS)(ES RRF from ICal)(ES Amount)}}$

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard

4.2.8 Requests for Re-extraction

Review all supporting data, including spike profiles, extraction logs, clean-up logs, injection prep logs, observation forms, and the sample tracking forms in the folder. The project or work group folder may contain exceptions or changes to routine spiking procedures.

Check the sample for problems relating to analysis. These problems include response factors that may introduce quantitative errors, interference that could be diluted out, or any interference that causes de-tuning or chromatographic conditions that could lead to quantitative errors.

The Laboratory Supervisor or Director should be consulted when re-extraction is considered.

If re-extraction is necessary, complete the Re-Extraction Form, which indicates the sample id, re-extraction due date, and reason for re-extraction (ref. form DC18). Set the sample status to PR:NEED in the database.

4.3 Safety

4.3.1 Each employee must read, understand and follow the safety guidelines in the Chemical Hygiene Plan.

4.3.2 For further safety precautions, please refer to the referenced test method.

4.4 Reference Method

"Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS." USEPA. October 1994.

Standard Operating Procedures
For the Analysis & Reporting of 1613 Drinking Waters

Issue date: 04/21/09
Revision: 10

SGS North America Inc.
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Updated By	042209 Date
Name, Laboratory Manager	4/23/09 Date
Jeannie Milholland Name, QA/QC Manager	4-22-2009 Date

Purpose

To describe the processes used in operating the HRGC/HRMS system, as well as the procedures followed in the generation, interpretation and review of laboratory data for Method 1613 drinking water samples.

Summary

This SOP details how to analyze and report drinking water samples by EPA Method 1613. HRGC/HRMS is used to detect and quantitate TCDD. Samples arrive at the MS lab having been extracted and fractionated using procedures in Section 3. Analyses are grouped into 12-hour sequences that include analyses of samples and standards mixtures. Upon completion of the sequence, the analyst reviews the data associated with both standards and samples in order to confirm the validity of the sequence and to determine any potential need for re-analysis or re-extraction. The analyst generates quantitation reports and chromatograms using sophisticated software. These reports are used to generate forms that summarize the results of the analysis.

4.1 Operation of HRGC/HRMS

4.1.1 Equipment

- HP6890 GC, Micromass Autospec Ultima high resolution mass spectrometer, vortex mixer, 10-100 uL pipette

4.1.2 Procedure

- Recall the GC temperature/pressure/flow program.
- Column: DB-5MS, 60 m, id 0.25 mm, 0.25 μm.
- Recall the MS experiment (see Table 1).
- Perform any necessary maintenance.
- Tune the MS resolution to 100 ppm at 5% height.
- Acquire location data to calibrate the MS and print a copy of MS resolution.
- Inject the GC resolution mix. This injection is used to verify that there is less than or equal to 25% peak to valley for the two close eluters of 2,3,7,8-TCDD. Print a copy of the GC resolution check. If the valleys are within specifications, proceed to calibrate or verify a previous calibration. If not, further investigation and/or maintenance may be required. Re-inject this solution after maintenance to check for improvement.
- Now that the GC/MS resolution has been verified, a series of five initial calibration standards may be injected and reviewed for method requirements. If an initial calibration already exists, a CS3 may be analyzed to verify continuing calibration. For North Carolina drinking water compliance, a daily reporting limit check sample (5 pg/L or less) must also be analyzed to verify instrument performance. The CS1 (0.25 pg/uL) may be used for this purpose. If the curve, the CS3 and the reporting limit check pass method requirements, sample analysis may begin.
- Reconstitution of a sample is accomplished by adding nonane containing the injection standard (JS), capping the vial, and mixing well with a vortex mixer. The amount of standard to be added can be found in the spike profile document.
- Samples are injected under conditions identical to those used to establish calibration.
- A "back-end" print out of the MS resolution must be performed.
- The calibration data from a sequence is filed in a folder cabinet under the day it was analyzed and includes all GC/MS resolution checks, valley verification, front end CS3s, reporting limit verifications, run logs and injection prep logs.
- Each sample hardcopy should include the quant report, chromatogram, and report form.

Table 1: Mass Descriptors used for Selected Ion Recording HRMS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	316.9824	100	10
1	2	316.9824	(Lock)	10
1	3	319.8965	100	10
1	4	321.8936	100	10
1	5	327.8847	100	10
1	6	331.9368	100	10
1	7	333.9339	100	10

4.2 Data Generation, Interpretation and Review

SGS Environmental Services defines a batch of samples as no more than 20 samples processed within a 12-hour shift. One LMB, one OPR and one OPRD are processed per analytical batch, following the same procedures as the field samples. Generally, drinking water is replaced by deionized water. An invalid LMB or OPR/OPRD requires a re-extraction of the affected samples.

4.2.1 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. The use of high purity reagents and solvents helps minimize interference problems. All new materials used in the analysis shall be demonstrated to be free from interferences by running an initial reagent blank. Interferences co-extracted from samples will vary considerably from source to

source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the analytes. The elimination of interferences is essential. Cleanup steps are used to reduce or eliminate these interferences and thereby permit reliable determination of the analyte as close as possible to the specified sensitivity.

4.2.2 Quality Assurance/Quality control

On an annual schedule, the laboratory shall perform a drinking water Method Detection Limit study (MDL). The laboratory shall perform an MDL study for each extraction method utilized. All MDL studies will be conducted following the guidelines set forth in 40 CFR, Part 136, appendix B and must be lower than one-third the regulatory compliance level or one third the Minimum Levels (ML) set forth in Table 2 of the reference method. In addition, at the time of the MDL a continuing calibration will be analyzed at a concentration other than the regularly utilized CS3 to further demonstrate instrument performance.

4.2.3 Initial Calibrations

The percent relative standard deviation for the mean response factor from the unlabeled standard must not exceed +/- 20%. The percent relative standard deviation from the labeled standards (i. e. extraction standard and cleanup standard) must not exceed +/- 35%. The signal to noise ratio for all signals present must be $\geq$ 10. The ion abundance ratios must be between 0.65 and 0.89. SGS uses the concentrations in Table 2 to construct the initial calibration.

Table 2. Initial Calibration Concentrations

	Concentration (pg/µL)				
Analyte	CS-1	CS-2	CS-3	CS-4	CS-5
Unlabeled					
2378-TCDD	0.25	2	10	40	200
Extraction Standard					
^{13}C-2378-TCDD	100	100	100	100	100
Cleanup Standard					
^{37}Cl-2378-TCDD	0.25	2	10	40	200
Injection Standard					
^{13}C-1234-TCDD	100	100	100	100	100

A new initial calibration is required when the continuing calibration criteria below are not met. Routine maintenance may be performed to correct any failures. Any major maintenance to the analytical system such as slit cleaning, analyzer lens cleaning, magnet shifts, and detector disk changes warrant a new ICAL. At a minimum, a new initial calibration must be performed annually.

4.2.4 Continuing Calibrations

Check that all paperwork is present. A CCal package should contain the documentation listed below.

- Pass: Run log. HRMS Resolution Checks. GC performance for 2,3,7,8-TCDD. CCal quantitation page. CCal chromatogram. RL Ver. quantitation page. RL Ver. chromatogram. Injection preparation log.
- Fail: The analyst listed on the run log can provide any missing paperwork.

Review the Run log.

- Pass: Check that the 12 hour windows have not been exceeded between the front end Ccal and the last sample of the sequence.
- Fail: Re-analysis of affected samples.

Review the HRMS Resolution checks.

- Pass: Verify 100ppm width at 5% height for PFK mass 305 or higher. Compare the resolution check times to those on the run log to be sure they bracket each sequence.
- Fail: An assessment should be made to determine any data quality impact.

Review the GC Performance documentation.

- Pass: Check that the valley between 2,3,7,8-TCDD and its close eluters does not exceed 25%.
- Fail: If the GC performance valley is greater than 25% instrument maintenance may be required. When a valley fails all samples must be reinjected.

Review the CCal Quantitation and Chromatograms.

- Pass: Check that all ion ratios are in specification. Verify that all compounds are within the concentration limits set by the method (see Table 3) for all front end CCals. Verify that the daily reporting limit check sample meets all peak identification criteria.
- Fail: Routine instrument maintenance such as installing new injection port hardware, inner source cleaning, retuning, column clipping etc. will usually correct a calibration failure. If these measures do not work, a new ICal is needed.

Compound Name	CCAL (pg/µL)	Limits (pg/µL)	Compound Name	CCAL (pg/µL)	Limits (pg/µL)
2,3,7,8-TCDD	10	8.2 - 12.3	$^{13}C_{12}$-2,3,7,8-TCDD	100	85 – 117
			$^{37}Cl_4$-2,3,7,8-TCDD	10	8.3 - 12.1

Table 3. Continuing Calibration Limits

Review the Injection Prep log sheet.

- Pass: Check that all samples have been spiked with 2 ng injection standard. Verify that final volume is 20 uL. Be sure that any dilutions or other comments are noted.
- Fail: Calculations of sample concentrations should reflect any deviations from normal injection prep parameters.

4.2.5 Quality Control Work Groups

The following elements should be present in a complete work group file:

- LMB topsheet
- LMB chromatogram
- OPR/OPRD topsheet
- OPR/OPRD chromatograms
- Extraction log sheet
- Cleanup log sheet
- Cleanup observation forms
- Any additional information (ex. re-extract request sheet)

The following procedure should be used for reviewing a work group:

- Review the header information on the LMB and OPR/OPRD topsheets. Verify that the method and sample ID (LMB or OPR/OPRD) are correct.
- Review the footer information on the LMB and OPR topsheets. Verify that the following information is correct: SGS Work Group number, extraction date, analysis date, method, matrix, sample weight/volume, pH, work group number, sample datafile, retcheck datafile, beginning cal datafile and ICal datafile.
- Verify that no target analytes are present in the LMB above Minimum Levels. If target analytes are above this limit, the associated samples must have concentrations that exceed 10 times the LMB concentration for the specified analyte. Otherwise, samples must be re-extracted.
- Verify that extraction and cleanup standard recoveries are within method specifications (see Table 4) for the LMB and OPR/OPRD. Validate any failures based upon signal to noise and acceptable detection limits. If the lab validation fails a corrective action is required. Corrective actions may include re-extraction, re-cleanup, lower sample volume, extract dilution, etc.
- Verify that the recoveries in the OPR meet SGS's recovery limits, found in Table 5.

Analyte	Amount Spiked (pg/µL)	Limits %
$^{13}C_{12}$-2,3,7,8-TCDD	100	31 – 137
$^{37}Cl_4$-2,3,7,8-TCDD	10	42 - 164

Table 4. Labeled Standard Recovery Limits

Analyte	Amount Spiked (pg/µL)	Limit (pg/µL)	OPRD %D Limit
2378-TCDD	10	7.3-14.6	20
^{13}C-2378-TCDD	100	25-141	NA
^{37}Cl-2378-TCDD	10	3.7-15.8	NA

Table 5. OPR Recovery Limits

4.2.6 Data Review

4.2.6.1 Procedure

- Complete Data Review Checklist (Section 4, Appendix B)

4.2.7 Calculations

4.2.7.1 Target compound calculation

- TCDD (ppt) = $\frac{\text{(Sum Ion Abun. of analyte)(ES Amount)}}{\text{(Sum Ion Abun. of Int. Std)(RRF from ICal)(Amt. of Sample)}}$

The instrumentation software calculates the noise level. However, manual noise determination may be employed at the reviewer's discretion in order to more accurately report peaks of interest.

4.2.7.2 Extraction Standard Recovery Calculation

- % Recovery = $\frac{\text{(Sum Ion Abun. of ES)(JS Amount)}}{\text{(Sum Ion Abun. of JS)(ES RRF from ICal)(ES Amount)}}$

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard

4.2.8 Requests for Re-extraction

Review all supporting data, including spike profiles, extraction logs, clean-up logs, injection prep logs, observation forms, and the sample tracking forms in the folder. The project or work group folder may contain exceptions or changes to routine spiking procedures.

Check the sample for problems relating to analysis. These problems include response factors that may introduce quantitative errors, interference that could be diluted out, or any interference that causes de-tuning or chromatographic conditions that could lead to quantitative errors.

The Laboratory Supervisor or Director should be consulted when re-extraction is considered.

If re-extraction is necessary, complete the Re-Extraction Form, which indicates the sample id, re-extraction due date, and reason for re-extraction (ref. form DC18). Change the status of the sample to PR:NEED in the database.

4.3 Safety

Each employee must read, understand and follow the safety guidelines in the Chemical Hygiene Plan. For further safety precautions, please refer to the referenced test method.

4.4 Reference Method

"Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS." USEPA. October 1994.

Method Manual of Standard Operating Procedures
For the Determination of Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)

Method 1613

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SGS North America Inc.
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Revised By:

Jim Plassard — Date: 042309

Name, Dioxin Supervisor:

Jim Plassard — Date: 042309

Name, Laboratory Director:

Kendall Sutler — Date: 4/23/2009

Name, QA/QC Manager:

Jeannie Milholland — Date: 4-23-2009

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1 Identification of Test Method

Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. USEPA, October 1994.

2 Applicable Matrix or Matrices

This method is for Aqueous, Solids (typically Soils and Sediment), and Biological Tissue. Other matrices are addressed independently in separate SOPs.

3 Scope and Application

This method provides procedures for the detection and quantitative measurement of polychlorinated dibenzo-p-dioxins (tetra- through octa-chlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octa-chlorinated homologues; PCDFs). Concentrations are provided in solid environmental matrices at the part-per-trillion (ppt) level as picograms per gram (pg/g) and in aqueous environmental matrices to the part-per-quadrillion (ppq) level as pg/L. Specifications are also referenced for separate determination of 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF) if requested. Samples containing concentrations of specific congeneric analytes (PCDDs or PCDFs) that are greater than ten times the upper initial calibration limit must be analyzed by a protocol designed for such concentration levels as they may be beyond the scope of this method. The lab will make effort to utilize a smaller sample aliquot or dilute the extract to bring the sample concentration within the range of the instrumentation.

4 Detection Limits for Components to be Analyzed

The detection limits and quantification levels of this method are usually dependent on the level of interferences within a given matrix rather than instrumental limitations. The Reporting limits listed in TABLE 1 are based on an initial calibration point at or below the values given.

5 Summary of the Test Method

The analytical method calls for the samples to arrive at the Mass Spectrometry lab having been extracted and fractionated by analysts who are experienced with residue analysis. High-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) are used to analyze such purified sample extracts. The GC/MS portions of this method are for use only by analysts experienced with HRGC/HRMS or under the close supervision of such qualified persons. The laboratory will demonstrate the ability to generate acceptable results before beginning sample analysis. Sample analyses are grouped into 12-hour sequences, which include analysis of calibration standard mixtures and quality control samples. Upon completion of the sequence, the analyst reviews the data associated with both standards and samples in order to confirm the validity of the run and to determine any potential need for re-analysis or re-extraction. The analyst generates quantification reports and chromatograms using sophisticated software. These reports are used to generate forms that summarize the results of the analysis.

6 Definitions

See Document DC139 Appendix D for a full list of definitions.

7 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines possibly causing misinterpretation of chromatograms. The use of high purity reagents and solvents helps minimize interference problems. All new materials used in the analysis should be demonstrated to be free from interferences by running an initial reagent blank. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the analytes. The elimination of interferences is essential. If interferences are not apparently present (i.e. sample history), the full battery of cleanup possibilities may be unnecessary. Otherwise, cleanup steps are used to reduce or eliminate these interferences and thereby permit reliable determination of the analyte as close as possible to the specified sensitivity.

8 Safety

Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCDDs or PCDFs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed.

Each employee must read, understand and follow the safety guidelines in the Chemical Hygiene Plan.

For further safety precautions, please refer to the referenced test method.

9 Equipment and Supplies

A list of the equipment used by this method can be found in the Statement of Qualifications.

Also provided is a description of the procedures followed for the preparation of various materials used in analytical procedures at SGS.

9.1 Tridecane Purification

Percolate tridecane through a carbon-celite column to purify it with the following procedure.

- Procedure

  Place 2 grams of carbon-coated celite in a disposable 10mL glass pipette. The bottom of the pipette contains glass wool and sodium sulfate to insure no breakthrough of carbon particulate. Percolate the tridecane directly from the supplier's bottle onto the carbon column. Flow through the column should not exceed 0.5 mL/min. Collect the purified tridecane inside a clean and properly labeled container. Document the preparation in the Supplies Preparation Logbook.

9.2 Glassware Cleaning

All reusable glassware such as glass columns, flasks, SDS, etc. are washed with soap and tap water. Concentrators are rinsed with extraction solvent, washed with soapy water, followed by an acetone and DI water rinse. Reusable glassware does occasionally require baking. Glassware is then placed inside a muffle furnace operating at 454°C for 6hr. We do bake our glassware more often than suggested by the analytical method, but not more often than necessary. We also occasionally take it to its annealing

temperature to refresh its structure to a new state. Even with routine baking as necessitated by sample matrix, with occasional annealing we do not see any of the adverse effects that are commonly associated with routine baking (i.e. irreversibly adsorbed compounds).

- Procedure
  - Rinse the concentrators with extraction solvent.
  - Scrub all glassware vigorously with a brush and hot soapy water.
  - Rinse with copious amounts of tap water.
  - Rinse all glassware with acetone.
  - Rinse all glassware with DI water.
  - Wrap cleaned foil around openings to prevent the infiltration of dust and store the cleaned glassware in its designated area.

9.3 Acid- and Base-Coated Silica

Silica is coated with NaOH or H_2SO_4 to produce acid- and base-coated silica. The resulting packing materials are used to prepare the A/B columns utilized during the clean up of sample extracts.

- Procedure for Acidic silica; 30% w/w:
  - Weigh out into a clean amber jar 100.0 g of silica.
  - Into a separate beaker, weigh 44.0 g of concentrated H_2SO_4.
  - Add the acid drop-wise via a pipette to the silica. Add 1-2mL at a time to avoid clumping.
  - Cap the jar and shake vigorously after each acid addition to distribute the acid.
  - If necessary, break up any aggregates with a glass-stirring rod.
  - Continue in this manner until all of the acid has been added into the silica.
  - Label the jar appropriately and store accordingly.
  - Document the preparation in the Supplies Preparation Logbook.

    Note: If stored for a prolonged period, fill the jar headspace with nitrogen and cap.
- Procedure for Basic silica; 1.2% w/w:
  - Weigh out into a clean amber jar 100.0 g of silica.
  - Into a separate beaker, weigh 30.0 g of 1 N NaOH.
  - Add the base drop-wise via a pipette to the silica. Add 1-2 mL at a time to avoid clumping.
  - Cap the jar and shake vigorously after each acid addition to distribute the base.
  - If necessary, break up any aggregates with a glass-stirring rod.
  - Continue in this manner until all of the base has been added into the silica.
  - Label the jar appropriately and store accordingly
  - Document the preparation in the Supplies Preparation Logbook.

    Note: If stored for a prolonged period, fill the jar headspace with nitrogen and cap.

9.4 Back Extraction Buffer

- Summary

  Fractionating an extract against this buffer will help to neutralize any pH effects an acid/base fractionation may leave on the extract.
- Procedure

Weigh out 6.8 grams of potassium phosphate (KH_2PO_4). Add it to the bottle. Dissolve it with some de-ionized water. Add 30mL of 1.0 N sodium hydroxide (Na OH) to the bottle. Fill the bottle to approximately 1000mL and verify a pH between 7.0-7.2.

9.5 Carbon / Celite

- Summary

  A carbon/celite mixture is prepared for use in carbon cleanup of sample extracts.

- Procedure

  Weigh out 18 grams of carbon and 82 grams of celite. Grind the carbon and celite with a mortar and pestle until they are completely mixed. The carbon should not separate out of the mixture. Activate the mixture at 130°C for 12 hours. Label the jar appropriately and store accordingly. Document the preparation in the Supplies Preparation Logbook.

9.6 Potassium silicate

- Summary

  Potassium silicate is prepared for use in the anthropogenic isolation columns. KOH is dissolved in methanol and added to neutral silica. The potassium silicate is dried and activated before use.

- Procedure
  - Dissolve 56 g of high purity KOH in 300 mL methanol.
  - Add 100 g silica and a stirring bar.
  - Stir on hot plate at 60-70°C for 1-2 hours.
  - Decant the liquid and rinse twice with 100 mL methanol.
  - Rinse once with 100 mL methylene chloride.
  - Spread the potassium silicate on foil and dry for 2-4 hours in the supplies vacuum concentrator.
  - Activate for 18 hours at 200°C.
  - Add 42 g neutral silica and tumble for one hour.
  - Label the jar appropriately and store accordingly
  - Document the preparation in the Supplies Preparation Logbook.

9.7 Acid / Base Silica Columns

- Assemble the acid/base silica column in a 25 mL pipette as follows from bottom: glass wool, 1 g SiO_2, 4 g NaOH coated silica, 1 g SiO_2, 8 g H_2SO_4 coated silica, 2 g SiO_2, 4 g Na_2SO_4.

9.8 Florisil Cleanup Columns

The florisil column is prepared in a glass tube using 6g of florisil.

- Insert glass fiber filter into the tube fitting.
- Attach the fitting to the bottom of the tube.
- Add a 6 g layer of florisil. Tap the top of the column gently
- Attach the top fitting.
- Use immediately.

9.9 Alumina Cleanup Columns

9.9.1 To pack a gravity column

The alumina column is prepared in a 10 mL glass pipette (approximately 300 mm x 10.5 mm) using 6 g acidic alumina and 4 g sodium sulfate.

- Insert a glass wool plug into the bottom of the column.
- Add a 6 g layer of acidic alumina. Tap the top of the column gently
- Add a 4 g layer of anhydrous sodium sulfate to cover the alumina.

9.10 Anthropogenic isolation columns

Anthropogenic isolation columns are used in place of the regular acid/base column for tissue cleanup. They consist of neutral and acidic silica, potassium silicate, and salt.

- Procedure

  Assemble the column as follows from bottom: PCU plug, 2 g neutral silica, 2 g potassium silicate, 2 g salt, 10 g acidic silica, 2 g salt, PCU plug.

9.11 Lot Testing

9.11.1 Procedure

- Upon receipt of the supply, it is logged into the Supplies logbook. If the lot received has already been tested and approved for use, no further action is necessary. If the lot has not been tested, it is scheduled for analysis.

9.11.1.1 Sample Bottles/Jars

- Rinse the bottle with three 10 mL rinses of DCM and transfer to a 60 mL vial.
- Concentrate the extract, add the appropriate amount of extraction standard, and transfer to a GC vial.
- Concentrate the extract to dryness and add the appropriate amount of injection standard.
- Analyze and evaluate results using the appropriate method's LMB requirements.
- If the test does not meet requirements, corrective action is required.

10 Reagents and Standards

Media, solutions and reagents are prepared, used and stored according to the manufacturer's instructions or the following procedures.

10.1 Reagent Receipt

- Documentation for media purchased pre-prepared, ready-to-use shall include manufacturer, lot number, type and amount received, date of receipt, opened date, and expiration date.

  For the following reagents, SGS attaches a sticker that permits the addition of documentation not covered by the manufactures label. Laboratory reagents include: 1M NaOH solution, concentrated sulfuric acid (H_2SO_4), Silica gel, Florisil, Acidic Alumina, Celite, Activated carbon, potassium hydroxide (KOH), sodium chloride (NaCl), sodium sulfate (Na_2SO_4), Dichloromethane (CH_2Cl_2), Hexane (C_6H_{14}), Methanol (CH_3OH), Nonane (C_9H_{20}), Toluene ($C_6H_5CH_3$), Cyclohexane (C_6H_{12}), Acetone (CH_3C0CH_3), and Tridecane ($C_{13}H_{28}$).
- Documentation for media prepared in the laboratory shall include date of preparation, preparer's initials, type and amount prepared, manufacturer and lot number, and expiration date. All media prepared in the laboratory is documented in the Supplies Preparation Logbook.

10.2 Lot Testing

10.2.1 Procedure

Upon receipt of the supply, it is logged into the Supplies logbook. If the lot received has already been tested and approved for use, no further action is necessary. If the lot has not been tested, it is scheduled for analysis.

10.2.1.1 Solvents

- Add 40 mL solvent to a 60 mL vial.
- Concentrate the extract, add the appropriate amount of extraction standard, and transfer to a GC vial.
- Concentrate the extract to dryness and add the appropriate amount of injection standard.
- Analyze and evaluate results using the appropriate method's LMB requirements. Data may require adjusting for the amount of solvent used during the prep process.
- If the test does not meet requirements, corrective action is required.

  Alternatively, a LMB may be extracted and analyzed using the new solvent.

10.2.1.2 Reagents

- Extract and clean up a LMB using the new reagent.
- Analyze and evaluate results using the appropriate method's LMB requirements.
- If the test does not meet requirements, corrective action is required.

10.3 Standards Preparation

10.3.1 Stock Standard Solutions

10.3.1.1 High-Resolution Concentration Calibration Solutions

Five nonane solutions containing 17 unlabeled and 18 carbon-labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homologue-dependent, with the lowest values for the tetra-chlorinated dioxin and furan (0.25 pg/µL) and the highest values for the octachlorinated congeners (2000 pg/µL).

10.3.1.2 GC Column Performance Check Solution

This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The $^{13}C_{12}$-2,3,7,8-TCDD is also present. The laboratory is required to use nonane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/µL per congener.

10.3.1.3 Sample Fortification Solution (Extraction Standards)

This nonane solution contains the fifteen internal standards at the nominal concentrations that are listed in Table 3. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that $^{13}C_{12}$-OCDF is not present in the solution.)

10.3.1.4 Sample Fortification Solution (Cleanup Standards)

This nonane solution contains the 2,3,7,8-$^{37}Cl_4$-TCDD at the nominal concentrations that are listed in Table 3. The concentration of this compound is quantified by the injection standards as a measure of cleanup efficiency.

10.3.1.5 Recovery Standard Solution (Injection standards)

This nonane solution contains two recovery standards, $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD, at a nominal concentration of 100 pg/µL per compound. 10 to 50 µL

of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

10.3.1.6 Matrix Spike Fortification Solution

Solution used to prepare the MS-MSD and OPR-OPRD samples. It contains **all** unlabeled analytes listed in Table 3 at concentrations corresponding to the CS-3.

10.4 Working Standard Solutions

Generally, reference materials are obtained from a commercial source either as a single component in solution or as a mixture containing a number of components in solution. A "Certificate of Analysis" normally accompanies the reference material. Definitions of the various standard solutions, details on the preparation, traceability and labeling of the working standard solutions are given below.

- Procedure

  All entries and calculation details are made with the help of the LIMS Standards Preparation Program. Call the Standards Preparation Program. Follow the directives and menu options as required. When all information entry is completed, print the Summary page. In the space provided on the Summary page, write the page number. Insert the Summary page inside the Standards Preparation Logbook (e.g., S-12). When available, attach the supplier's "Certificate of Analysis" correctly labeled to facilitate traceability. Print the waterproof label containing:

  - Solution identification number
  - Nominal concentration
  - Expiration date
  - Apply label on the vials and with the help of a magic marker, place a mark indicating the meniscus.
  - Keep all PCDD/F and PCB standard solutions at room temperature inside amber vials.

  Good practice: what is removed from the vial never returns to the vial.

  SGS uses, when available, standards from different manufacturers or a second lot from the same manufacturer, to calculate and verify all calibration standards. If second lots aren't available, SGS prepares a second solution/dilution to use until a second lot can be purchased. SGS has used standard reference material and performance evaluation standards to verify calibration.

  On the nomenclature used to describe the various labeled standard solutions: Each of the "dioxin" methods has its own way to name the labeled standards used throughout the procedure. SGS has adopted a single nomenclature that reflects the function of by the standards.

  Commercial standards come in different solvents; hexane, iso-octane, nonane, just to name a few. SGS practice is to use a solvent compatible with the manufacturer's choice. However, nonane is preferred and will be used whenever possible because of its favorable properties.

10.4.1 Lot Testing

Most standards purchased by SGS arrive having already been tested and certified by the manufacturer. Records of these analyses are maintained in the standards logbook. If the standard has not previously been certified, it should be analyzed upon receipt. Concentrations should fall within ±10% of the stated values, with no contamination present above LMB limits.

10.5 Expiration Dates for Standards

Primary Standard Solutions (ampoule from commercial source): Use the manufacturer's date. If a stability study is in progress, use 5 years.

Stock Standard Solutions (obtained from dilution/mixing Primary Standard Solutions): Up to 5 years, not to exceed the Primary Standard expiration date.

Working Standard Solutions (from Stock/Primary and used during sample processing): Up to 1 year, not to exceed the Stock Standard expiration date.

All solutions prepared by SGS with a nominal concentration of less than 0.1 ng/uL are assigned a 30-day expiration time.

Standards will be removed from use and disposed on the expiration date.

11 Sample Collection, Preservation, Shipment, and Storage

11.1 Sample collection

- Sample collection personnel should, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. (If the sample is clearly not homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.)
- Grab and composite samples must be collected in amber glass containers. Caps shall be lined with fluoropolymer. If amber bottles are not available, samples shall be protected from light (i.e. wrapped in foil).
- If residual chlorine is present in aqueous samples, the sampling protocol should include the addition of 80mg of sodium thiosulfate per liter of water. Upon receipt into the laboratory, samples should be checked for residual chlorine using AquaChek test strips. Samples containing residual chlorine should be preserved in the laboratory with 80 mg of sodium thiosulfate per liter.
- Conventional sampling practices must be followed. The bottle must not be pre-washed with sample before collection. Sampling equipment must be free of potential sources of contamination.
- Note: The lab does offer grinding or blending of fish samples - If not otherwise specified in a project plan, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested. If so requested, the above whole fish requirement is superseded.

11.2 Sample Storage and Holding times

Samples that arrive at the laboratory are subject to the Sample Acceptance policy. Samples received by the laboratory must meet certain criteria in order for the associated analytical data to meet method criteria. Samples must be properly documented and shipped in appropriate containers at specified temperatures. Holding times must be met and the amount of sample provided must be sufficient for the required extraction procedures. The laboratory will attempt to contact the client and resolve any issues with sample acceptance. Samples that are immediately rejected are ones with

improper preservation, damage, or show signs of contamination. Clients will also be contacted when samples arrive above 6°C. Samples that arrive at <10°C are considered acceptable by the laboratory.

All samples must be shipped on ice in the dark, and should be delivered as soon as possible (i.e. express carrier). Tissue samples should be frozen prior to shipping.

Once received by the lab, all samples, except tissue samples, must be stored at 4°C in the dark (tissue samples must be stored at <-10°C in the dark), and should be extracted within 365 days and completely analyzed within 45 days of extraction.

12 Quality Control

SGS defines a batch of samples as no more than 20 samples processed with in a 12-hour shift. One laboratory method blank (LMB), one On-going Precision and Recovery spike (OPR), and one OPRD are processed in every extraction batch (workgroup), following the same procedures used for the field samples. Other Batch QC samples such as Matrix Spikes and Matrix Spike Duplicates (MS/MSD) are extracted as well. These may apply to more than one workgroup as long as there are no more than 20 associated samples and the workgroups are less than 30 days apart (QC Batch, Sample Delivery Group). Client specified samples are used for MS/MSD whenever possible. Generally, for the LMB and OPR samples, soil is replaced by salt (Na_2SO_4), effluent by de-ionized water and biological tissues by vegetable oil. An invalid LMB or OPR requires a re-extraction of the affected samples.

12.1 Quality Control Work Groups

The following elements should be present in a complete work group file:

- LMB results summary
- LMB totals sheets
- LMB chromatograms (11 pages)
- OPR results summary
- OPR chromatograms
- OPRD results summary
- OPRD chromatograms
- Dry weight sheet (where applicable)
- Extraction log sheet
- Cleanup log sheet/observation forms
- Any additional information (ex. re-extract request sheet)

The following procedure should be used for reviewing a work group:

- Review the header information on the LMB summary sheets. Verify that the method and client sample ID (LMB or OPR) are correct.
- Review the footer information on the LMB and OPR summary sheets. Verify that the following information is correct: extraction date, analysis date, method, matrix, sample weight/volume, percent solids/lipids, pH, work group number, sample data file, RETCON data file, beginning cal data file, and ICAL data file.
- Verify that no target analytes are present in the LMB above the lower method calibration limit (LMCL). If target analytes are above this limit, the associated samples must have concentrations that exceed 10 times the LMB concentration for the specified analyte or the analyte must not

be detected in the associated sample. Otherwise, samples must be re-extracted.

DoD Blank Requirements:

- No analyte concentrations in the LMB greater than the minimum level (Method 1613 Table 2), or greater than one-third the regulatory compliance level, whichever is greater.

- Review the totals data for the LMB. Be sure that any ghosting peaks are removed from the totals concentrations and the associated detection limits are elevated to reflect the subtracted peaks.
- Verify that extraction standards are within method specifications listed in TABLE 8 for the LMB and OPR.
- Verify that unlabeled recoveries in the OPR meet the recoveries listed in TABLE 9. All target analytes are evaluated in the OPR.

12.2 Method Detection Limit Study and IPR Schedule

On an annual schedule, the laboratory shall perform a Method Detection Limit study (MDLs) utilizing each preparatory/analytical method combination. MDLs are to be calculated for each 2,3,7,8-substituted congener. All MDL studies will be conducted following the guidelines set forth in 40 CFR, Part 136, appendix B. An MDL verification sample will be analyzed concurrently at an estimated 2 to 3 times the expected calculated MDL value for use with USACE clients. In addition, at the time of the MDL a continuing calibration will be analyzed at a concentration other than the normally utilized CS3 to further demonstrate instrument performance.

Each analyst is required to successfully complete an Initial Demonstration of Precision and Accuracy (IPR) before working on samples. An IPR is conducted for each matrix and extraction technique utilized. IPRs are repeated with each relevant change to a technique.

13 Calibration and Standardization

13.1 Initial Calibrations

On an annual schedule, SGS uses the concentrations in Table 3 to construct the initial calibration. The percent relative standard deviations for the mean response factors from the seventeen unlabeled standards must not exceed +/- 20%. The percent relative standard deviations from the labeled standards (i. e. extraction standards, cleanup standards and sampling standards) must not exceed +/- 35%. The signal to noise ratio for all signals present must be $\geq$ 10. The ion abundance ratios must be within specified control limits (see Table 2). The concentration of the lowest calibration point is a modification of the referenced method. It is lower than required.

A new initial calibration is required when the continuing calibration criteria below are not met. Routine maintenance may be performed to correct any failures. Any major maintenance to the analytical system such as slit cleaning, analyzer lens cleaning, magnet shifts, and detector disk changes warrant a new ICAL. At a minimum, a new initial calibration must be performed annually.

13.2 Continuing Calibrations

13.2.1 Check that all paperwork is present

A CCal package should contain the documentation listed below.

- Pass: Run log. HRMS Resolution Checks. WDM retention time sheet. WDM chromatograms. GC performance for 2,3,7,8-TCDD. CCal quantitation page. CCal chromatograms. Injection preparation log
- Fail: The analyst listed on the run log can provide any missing paperwork.

13.2.2 Review the Run log.

- Pass: Check that the 12-hour windows have not been exceeded between the front end CCAL and the last sample of the sequence.
- Fail: Re-analysis of samples.

13.2.3 Review the HRMS Resolution checks.

- Pass: Verify 100ppm width at 5% height for PFK mass 318 or higher. Compare the resolution check times to those on the run log to be sure they bracket each sequence.
- Fail: Should a back-end resolution check fail, an assessment should be made to determine any data quality impact.

13.2.4 Review the Window Defining Mix and GC Performance Documentation.

- Pass: Check that the sample numbers on the WDM sheets match those on the runlog. Check that the retention times are correct for the WDM chromatograms.
- Check that the valley between 2,3,7,8-TCDD and its close eluters does not exceed 25%.
- Fail: Any missing peaks in the window-defining sample should be re-identified with a survey scan. Determine proper switching times. These must be entered into the HRMS ion function descriptors before analysis may resume. If the GC performance valley is greater than 25% instrument maintenance may be required. When a valley fails, all samples must be re-injected.

13.2.5 Review the CCal Quantitation and Chromatograms.

- Pass: All ion ratios must be within the control limits given in Table 2. Relative retention times between the native compounds and the labeled compounds must be within the limits in Table 10. Signal-to-noise for all peaks must be greater than 10. All concentrations must be within the concentration limits set by the method (see Table 7).
- Fail: Any samples associated with a failing CCAL will be scheduled for re-analysis. If an analyst is present, the sequence will be stopped to remedy the situation. Routine instrument maintenance such as installing new injection port hardware, inner source cleaning, retuning, column clipping etc. will usually correct a calibration failure. If these measures do not work, then the laboratory will demonstrate performance after corrective action with either two consecutive acceptable CCALs or a new initial calibration.

13.2.6 Review the Injection Prep log sheet.

- Pass: Check that all samples have been spiked with 2ng injection standard. Verify that final volume is 20µL. Be sure that any dilutions or other comments are noted
- Fail: Calculations of sample concentrations should reflect any deviations from normal injection prep parameters.

14 Procedures

14.1 Sample Preparation and Conditioning

14.1.1 Homogenization

- Prior to processing tissue samples, it must be determined as to the exact tissue that is to be analyzed. Analytical requests include whole fish, whole fish less skin, fillets, specific organs or various portions of the above. Once identified, the appropriate tissue must be frozen.
- When practical, samples are ground and homogenized while frozen.
- If specific portions must be dissected from the whole specimen the remaining tissue can be refrozen. When the entire sample, usually fish, requires analysis, reduce the sample down to manageable means using a butcher knife.

  Note: Great care must be exercised when reducing a frozen fish with a knife. Cut-resistant gloves must be worn to protect the hands as well as to hold the fish in place.
- When analyzing specific portions, such as a fillet, dissect the required portion under stringent safety guidelines to protect the analyst from injury as well as to ensure the integrity of the sample.
- Once reduced in size, process the tissue through a meat grinder and collect into a suitable container.
- To ensure complete homogeneity process the ground sample two more times.

  Note: Further reduction can be achieved by processing a portion of the specimen with a commercially available tissue macerator. However, continuous reprocessing of the sample through a meat grinder will often achieve the desired results.
- Record all observations.

14.1.2 Grinding

- After donning the appropriate personal protective equipment (PPE), usually nitrile gloves, open the sample in the hood.
- Remove any large objects such as rocks.
- If necessary pass the sample through a series of sieves to help facilitate the partitioning of the various particle sizes.
- Particles requiring grinding can in most instances be reduced by means of a mortar and pestle.
- In some cases reducing the temperature of the sample will aide in the grinding process.
- Some matrices such as certain papers, grains and amorphous solids need to be reduced using a Wiley Mill or other suitable mechanism.
- After sufficient reduction in the gross particle size, recombine and mix the various fractions of the sample.
- Record all observations.

14.1.3 Determination of Particle Size

- Spread a piece of clean aluminum foil in an appropriately ventilated fume hood.
- After donning the appropriate PPE, usually nitrile gloves, open the sample in the hood.

- With the aide of forceps spread a representative portion of sample on the aluminum foil.
- Estimate the size of the particles in the sample.
- If the estimate determines that the average particle size is greater than 1 mm, then the sample must be ground before extraction.
- Record all observations.

14.1.4 Percent Solids

- Solid Materials (by Modified ASTM D2216)
  - Label each weigh boat required with the unique sample ID number.
  - Thoroughly homogenize the sample, and grind the sample if necessary.
  - Weigh out 5-20 g of sample into the weigh boat.
  - Record the tare and sample weights taken and make note of any unique characteristics.
  - Note the time and date then dry the sample in an 110°C ± 5°C oven overnight.
  - After at least one hour, the sample may be weighted back in order to get a result, as calculated below, that can be used to approximate the minimum extraction amount. If this is performed, note the time and date and record the appropriate data. Then place the sample back in the oven
  - Remove the sample and allow to cool in a dessicator.
  - Note the time and date then reweigh the sample and record the dried weight.
  - Calculate the % solids as follows:

$$\%solids = \frac{weight\ of\ sample\ after\ drying}{weight\ of\ sample\ before\ drying} \times 100\%$$

  - Continue to re-weigh the sample, waiting at least one hour between weightings, until its mass changes by less than 1%. Use the final dried weight in the calculation of percent solids provided for sample reporting.
- Aqueous Materials
  - Desiccate and weigh a glass fiber filter to three significant figures.
  - Filter 10.0 ± 0.02 mL of well-mixed sample through the filter.
  - Dry the filter a minimum of 12 H at 110°C + 5°C, and cool in a dessicator.
  - Calculate percent solids by dividing the weight of solids trapped by 10, then times 100 for percent.

14.1.5 Sample Filtration

For aqueous samples containing visible particles but <1% solids, the sample should be filtered, then the filter extracted by SDS (Sec. 14.2.2), and the filtrate extracted by CLLE (Sec. 14.2.3).

Procedure:

- Assemble a Buchner funnel to a clean filtration flask.
- Tare the sample bottle for gravimetric volume determination.
- Apply a vacuum and pour the sample through a glass-fiber filter in the Buchner funnel.

- Rinse the bottle twice with approximately 5 mL DI water and pour onto the filter.
- Reweigh the bottle and record the volume.
- Remove the filter and proceed to extract using SDS.
- Remove the filtration flask and proceed to extract the filtrate by CLLE.

14.1.6 Percent Lipids

- Extract the sample via the appropriate method.
- Concentrate the sample extract to "dryness" inside a tared container.
- Calculate the lipids content to the nearest three significant figures:

$$\%L = \frac{R}{W} \times 100$$

where:
R = weight of dry residue in grams
W = weight of sample

14.1.7 Sub Sampling

Do not Split/Archive an inhomogeneous extract.

14.1.7.1 Sub Sampling Solid Samples

- Stirring the entire sample in its container is sufficient for wet samples. Dry samples should be emptied into a beaker or onto some foil for homogenization.
- Care should be taken when non-indigenous soils are handled. One must place foil down over the workspace to catch any spills. This foil and any items used in contact with the soil must be packed and properly labeled with purple stickers to insure proper disposal.

14.1.7.2 Sub Sampling Aqueous Samples

- Shake sample to homogenize. Particulates must be incorporated and evenly distributed. A powered shaker may be used.

14.2 Extraction

The primary objective of sample processing is to quantitatively remove the analytes of interest from the matrix into an appropriate solvent, and then to remove the solvent, leaving a dried extract that can then be reconstituted prior to analysis. Samples are first conditioned using homogenization for biological samples and grinding for solids with a particle size greater than 1 mm. If appropriate, a percent solids or percent lipids determination will be made during sample conditioning. The samples are then aliquotted and fortified with extraction standard. The samples are extracted using a procedure depending on the matrix, i.e. Soxhlet-Dean Stark (SDS) or Continuous Liquid-Liquid Extraction (CLLE) only. If necessary, extracts may be split after extraction, with a portion of the extract archived for future use. The resulting extracts are concentrated using heated nitrogen blow down (Turbovap).

14.2.1 General Extraction Procedures

14.2.1.1 Sample Fortification (Spiking)

In each case, add the appropriate amount of standards for the method in use. A 'witness' must be present. Note: the composition and amount of standards used may be subject to change based on method requirements and individual client needs. Refer to the spike profile (Controlled Document number DC120) and project folder for any changes. If the sample to be spiked is depleted or if only a limited volume remains, the

witness must verify the ID of the sample prior to spiking. The extractionist is responsible for identifying such samples to the witness.

14.2.1.2 Splitting/Archiving Extracts

Label the graduated and centrifuge tubes as required. Using a Pasteur pipette and a glass wool plug, filter the tridecane residue obtained after concentration directly to the graduated or calibrated tube (e.g., 3.5- and 7-mL marks). Add approximately 1 mL of hexane to the sample container, swirl the solvent and transfer to the Pasteur pipette. Add another 1 mL hexane inside the RB flask, swirl, and wash the RB flask walls using the pipette. Transfer the rinse to the Pasteur pipette. Collect all rinses directly in the graduated tube. Make sure the volume reaches the 7-mL mark. Transfer 3.5 mL to the centrifuge tube or any container suitable for the fractionation. Seal the graduated tube using a Teflon™-lined cap and store the sample inside the refrigerator. Notes:

- To prevent sample spills, always secure the sample tube and the Pasteur pipette to a rod using easily adjustable clamps.
- In the presence of particulates, it is necessary to rinse the particulates with toluene, and repeat the concentration and solvent exchange steps.
- **Do not Split/Archive an inhomogeneous extract.**

14.2.1.3 Extract Concentration (Turbovap)

- Set the water bath to the appropriate temperature for the solvent used and allow the bath to come to temperature (approximately 45°C for hexane and methylene chloride, 65°C for toluene).
- Rinse the nitrogen needles with CH_2Cl_2 to prevent any carryover.
- Place the 40- or 60-mL sample vials into the TurboVap. (Note: Be sure the correct vial rack is used.)
- Set the nitrogen at 1-2 psi, and set the time to allow the samples to evaporate down 5-10 mL. This will help prevent cross contamination. Once the samples have evaporated enough that solvent will not splash out of the vials, set the nitrogen at 10 psi and set the time to allow the sample to evaporate to near dryness. The TurboVap will beep to signal that time is up. Additional time may be added if samples are not completely evaporated.

Once the samples have evaporated, remove the vials and rinse the nitrogen needles with toluene.

14.2.2 Soxhlet/Dean-Stark

Samples containing >1% solids should be treated as solids for extraction purposes. An aliquot sufficient to provide 10 grams of dry solids should be used. If necessary, excess water may be decanted, or the sample aliquot may be filtered through a glass-fiber filter. The aqueous portion may be discarded.

Procedure:

- Remove samples from designated storage area and allow them to come to room temperature.
- Assemble the SDS without the condenser.
- Add 2-4 Teflon™ chips inside the 500-mL round-bottom flask.
- Add 300 mL of toluene through the DS section.
- Assemble the condenser.
- Turn on the water recirculator, which is set to 15°C.

- Turn on the heating mantle to position "65" for toluene.
- Allow the reflux to go on for at least three hours.
- Turn off the heating element, and allow the unit to cool down.
- Transfer the toluene to solvent waste.
- Add 2-4 fresh Teflon™ chips.
- Add 300 mL of toluene and 0.5 mL of purified tridecane.
- Adapt the thimble holder and insert the thimble containing a ten gram dry weight equivalent of the sample mixed with sodium sulfate.
- Spike the sample with the extraction standards as listed on the spike profile (Controlled Document number DC120) and allow to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked**.
- Spike the appropriate samples with matrix spike as listed on the spike profile (Controlled Document number DC120) and allow to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked**.
- Assemble the DS and condenser components.
- Cover the opened extremity of the condenser with foil.
- Turn on the water recirculator, then the heater.
- Insulate the "vapor pathway".
- Allow the extraction to continue for 12 to 18 H.
- During the extraction, verify that the solvent cycles normally.
- Drain the water as appropriate to prevent water from returning to the boiling flask.
- At the end of the reflux cycle time, proceed with the sample concentration as described below.
- **Do not turn off the heater yet.**
- Drain the toluene present in the DS side arm directly to solvent waste.
- Continue to drain the toluene until approximately 20 mL of toluene remains inside the RB flask.
- The toluene inside the thimble holder is removed and disposed.
- Turn off the heater when the toluene reaches the 20-mL mark. **Do not go to "near dryness".**
- Remove the heating mantle and allow the flask to cool down.
- Rotovapping may be necessary in some cases to expedite solvent evaporation.
- Transfer sample to 60 mL vial for Turbovap.

14.2.3 Continuous Liquid-Liquid Extraction

- Remove samples from designated storage area.
- Check and record the pH of the sample using dedicated pH strips. Pipet a small volume of water from the sample jar onto the strip and check against pH color chart. Do not dip the strip into the jar. If the pH of the sample is greater than 9 then adjust to within a pH of 7-9 using a disposable pipette to dispense drops of 5:1 H_2SO_4. Stir and recheck.
- Remove the sample from storage and spike with extraction standards as listed on the spike profile (Controlled Document number DC120).
- If the sample contains visible particles but is <1% solids, the sample should be filtered prior to extraction. See Section 14.1.5.

Pre-Extraction:

- Make sure the individual water supply valves on the CLLE rack are closed.

- Turn on the main pump valve to "extraction." This valve is located inside the cabinet.
- Open the water return valve, located on the extraction rack near the tank.
- Add several boiling chips to each concentrator.
- Attach the 1000 mL extraction body, drying adapter and concentrator to the CLLE rack.
- Attach the water supply and return lines to the concentrator, and open the valves.
- Turn on the pump power switch on the side of the cabinet to "extraction."
- Turn on the water heater and allow the temperature to come up to 160°C.
- Make sure the chiller is running at the proper temperature (10°C).
- Add 250 mL of dichloromethane to each extraction body.
- Add 1000 mL of deionized water to each extraction body.
- Attach the condenser to the extraction body and open the stopcock.
- Allow the apparatus to pre-extract for four hours.
- When the pre-extraction is complete, close the stopcock and allow the extract to concentrate down to approximately 10 mL.
- Turn off the water heater.
- Turn off the power to the water pump.
- Close the main return valve.
- Switch the main pump valve to "drain."
- Turn on the power to the "drain" pump.
- Crack open the individual water return valves to allow air in, and water to drain out.
- Run the "drain" pump until the water has completely drained.
- Turn off the power to the "drain" pump.
- Close the individual water supply and return valves.
- Properly dispose of the DI water and DCM from the pre-extraction procedure.

Extraction of samples:

- Reassemble the extraction body and concentrator on the rack.
- Add 250 mL of dichloromethane to each extraction body.
- Place sample jar on the balance and zero the scale.
- Add the entire contents of the sample jar to a 1000 mL extraction body. Rinse the jar with the additional 50 mL of dichloromethane needed for the 1000 mL extraction, and add the rinse DCM to the extraction body as well.
- Reweigh the sample jar on the zeroed scale and record the weight of the displaced sample volume.
- Attach the condenser to the extraction body and open the stopcock.
- Allow the samples to extract for 24 hours.
- When the extraction is complete, close the stopcock and allow the extract to concentrate down to approximately 1 mL.
- The solvent that returns to the concentrator is dried during extraction by passing thru a salt chamber.
- Turn off the water heater.
- Turn off the power to the water pump.
- Close the main return valve.
- Switch the main pump valve to "drain."
- Turn on the power to the "drain" pump.

- Crack open the individual water return valves to allow air in, and water to drain out.
- Run the "drain" pump until the water has completely drained.
- Turn off the power to the "drain" pump.
- Close the individual water supply and return valves.
- Turn off the chiller.
- Transfer the extract to a 60mL vial containing the cleanup standards and 100uL tridecane.
- Properly dispose of the remaining sample water and dichloromethane.
- Concentrate the extract to near dryness.

14.2.4 Waste Dilution (For high level samples)

- Weigh out 1 gram of sample into a 60mL vial.
- Dilute with toluene to achieve the dilution level required (20ml, 50ml, etc.)
- Shake the sample for 30 minutes and allow to settle. Remove the supernatant. Properly dispose of the remaining sample.
- Aliquot the desired amount of sample extract from the reserved supernatant. Prepare QC samples with the same amount of solvent.
- Spike the samples with the appropriate amount of extraction standard as listed in the spike profile (Controlled Document number DC120).
- Add tridecane and concentrate to remove the toluene.
- Add 5mL of hexanes to the vial, making sure the sample is dissolved in the solvent.
- Proceed directly to PCU cleanup.

Note: If the matrix is known to be exceedingly difficult, an extra dilution may be performed by taking a second aliquot of a smaller amount.

14.3 Cleanup

Concentrated extracts are subjected to cleanup procedures in order to remove potential interferences. The cleanup procedures involve column chromatography using different packing materials as appropriate. The cleanup procedures result in small volumes of extracts which are then reduced further for analysis. Please refer to TABLE 6 for guidance as to which cleanups are typically performed for each matrix. Additional cleanup procedures are listed here and are used in certain cases such as, when sample history indicates a need for them, or when a re-extract request indicates that interferences were present and that additional cleanup steps may be required to remove them from the re-extracted sample extract.

14.3.1 Conventional

This procedure describes a conventional, non-automated, all gravity fed, three column cleanup procedure that can be used to purify extracts.

- Assemble the multi-bed acid/base silica column in a 25 mL pipette as follows from bottom: glass wool, 1 g SiO_2, 4 g NaOH coated silica, 1 g SiO_2, 8 g H_2SO_4 coated silica, 2 g SiO_2, 4 g Na_2SO_4.
- Wash column with 50 mL hexane.
- Load sample and two 1-mL rinses of hexane.
- Elute with 100 mL hexane.
- The eluate is concentrated to near dryness and is now ready for the alumina column.
- The alumina column is prepared in a 10 mL pipette using 6 g acidic alumina and 4 g sodium sulfate.
- Load sample and two hexane rinses onto alumina column.

- Wash with 2%.
- Elute with DCM to collect dioxins/furans.
- Turbovap to near dryness.
- Follow the Carbon Cleanup column cleanup procedure contained in this section.
- Concentrate the extract in toluene to near dryness.

14.3.2 PCU-F

This procedure describes an automated (Paradigm Clean-up Unit) version of a 1613 cleanup utilizing an acid/base coated silica column and a florisil column.

- Assemble the acid/base silica column.
- Assemble the florisil column.
  Note: For tissue samples (e.g., fish) use the anthropogenic isolation column.
- Assemble waste collection setup.
- Wet columns with hexane.
- Fill the 2% line of the machine.
- Flush the center fitting of the machine.
- Load sample plus two vial rinses.
- Elute the acid base column with hexane.
- Wash florisil with 2%.
- Position the extract collection vial.
- Elute sample with 100% DCM.
  Note: To perform method, start PCU software, load method clen.2000.txt, and hit start. Concentrate the extracts when the method is complete.

14.3.3 Florisil Column Cleanup

This procedure describes an automated version of an extract cleanup utilizing only a florisil column.

- Assemble the bottom column as follows from bottom: filter, florisil.
- Assemble the top column as follows from bottom: filter, sodium sulfate.
- Position waste collection assembly.
- Wet columns with 2%.
- Wet columns with hexane.
- Load sample plus two vial rinses.
- Elute florisil column with hexane.
- Wash with column with 2%.
- Position collection vial.
- Elute sample with 100% DCM.
  Note: To perform method, start PCU software, load method fonly3.txt, and hit start. Concentrate the extracts when the method is complete.

14.3.4 Carbon Cleanup

This procedure describes a conventional 1613 cleanup utilizing only a carbon column.

- Assemble the carbon column by placing a glass wool plug in the disposable pipette; add 5 mm of celite, 5 mm carbon/celite mixture, then another plug of glass wool.

- Secure the column in a clamp and place a "waste" jar underneath the column. The column should be positioned so that the celite end is up.
- Rinse the column with 4 mL of toluene. Allow the solvent to run completely through the column. Continue to rinse the column with 2 mL of a tertiary mixture (75% Methylene Chloride:20% Methanol:5% Toluene), 4 mL of a binary mixture (50% Methylene Chloride:50% Hexane), and 2 ml methylene chloride. Be sure to let the solvent run completely through the column between each rinse, but do not let the column go dry.
- Load the sample onto the column with three 1-2 mL rinses of methylene chloride.
- Rinse the column with 10 mL binary, then 5 mL tertiary.
- Flip the column over so that the celite end is down. Remove the waste jar and position a collection vial underneath the column.
- Elute the sample off the column with 20mL toluene.

14.4 Analysis

14.4.1 Operation of HRGC/HRMS

- Recall the GC temperature/pressure/flow program.
- Recall the MS experiment (see Table 4).
- Perform any necessary maintenance.
- By using a PFK molecular leak, tune the instrument to meet the minimum required resolution of 10,000 (10 percent valley) at m/z 304.9824. This is accomplished by tuning mass 331.9792 to above 100ppm resolving power with an accelerating voltage of 7200 volts.
- Acquire location data to calibrate the MS and print a copy of each function's MS resolution report.
- Verify that the measured mass of 380.9760 is within 5ppm of the published exact mass (the mass on the report must be between 380.9741 and 380.9779).
- Verify that the 100ppm resolving power requirement is met by the 304.9824 ion.
- Inject the window defining/GC resolution/continuing calibration mix (RETCON). Evaluate descriptor-switching times for accuracy. If any window defining peaks have shifted outside the descriptor windows, adjust the switching times before injecting any samples. This injection is also used to verify that there is less than or equal to 25% peak to valley for the two close eluters of 2,3,7,8-TCDD. Print a copy of the GC resolution check. If the valleys are within specifications, proceed to calibrate or verify a previous calibration. If not, further investigation and/or maintenance may be required. Re-inject this solution after maintenance to check for improvement.
- Now that the GC/MS resolution and descriptor switching times have been verified, a series of five initial calibration standards may be injected and reviewed for method requirements, and then a RETCON must be analyzed to verify the initial calibration is valid. If an initial calibration already exists, only RETCON must be analyzed to verify the initial calibration is still valid. If the RETCON passes method requirements, sample analysis may begin.
- Reconstitution of a sample is accomplished by adding nonane containing the injection standards (JS), capping the vial, and mixing well with a

vortex mixer. The amount of standard to be added can be found in the spike profile (Controlled Document number DC120).

- Prior to analysis, sample extracts are stored in the hood.
- Samples are injected under conditions identical to those used to establish calibration.
- A "back-end" print out of the MS resolution must also be performed and validated.
- The calibration data from a sequence is filed in a folder cabinet under the day it was analyzed and includes the all GC/MS resolution checks, window verification, valley verification, RETCONs, runlogs and window defining mix (WDM) retention time sheets.
- Each sample hardcopy should include the quant report, totals pages, deviations, chromatograms, and report forms.
- GC Column: DB-5MS, 60 m, id 0.25 mm, 0.25 µm.

14.4.2 Manual Integrations

14.4.2.1 Purpose

To describe the procedures followed for manually integrating peaks on GC/MS data systems.

14.4.2.2 Summary

Under some conditions, the GC/MS data systems will not provide accurate integration of chromatographic peaks. When this occurs, the GC/MS operator will manually re-integrate the peak(s) in question, print the re-integration, initial and date the printout, and save the modified datafile. Both the original chromatogram and the re-integration are included in the data package

14.4.2.3 Procedure

The accuracy and precision of GC/MS data are dependent upon accurate integration of the chromatographic peaks produced during the analysis. Mass spectral data systems provide automated peak integration based upon certain parameters relating to peak height, peak width, peak slope, and other variables. These parameters can be set to provide accurate integration for the majority of peaks present in an analysis; however, no set of parameters will provide accurate integration for all peaks. It is therefore imperative that the analyst reviews all automated peak integrations and manually re-integrates any that are not accurate. When manual re-integration is required, the following procedure will be used:

- The analyst will use professional judgment in determining when peak re-integration is necessary. Under no circumstances should a manual integration be performed solely for the purpose of meeting quality control requirements.
- When re-integration is performed, the analyst will provide the original integration as well as a printout indicating the retention time and integration baselines of re-integrated peaks.
- The operator will initial and date the printout.
- The operator will re-print the quantitation and totals reports, which include the areas of the re-integrated peaks, as well as flags indicating modified peaks.
- The corrected peak areas (or heights, if applicable) will be used in all calculations of analyte concentration.

- For DoD projects, the rationale for manual integration will also be noted on the printout. In addition, the case narrative for the project shall identify the samples and analytes for which manual integration was necessary.

15 Calculations

15.1 Relative Response Factor of Analyte

To calculate the RF of an analyte in an initial calibration point or calibration verification:

$$RF_{ukn} = \frac{\left(A_{ukn}^{ion1} + A_{ukn}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} x \frac{Q_{ES}}{Q_{ukn}}$$

where:

- A_{ukn} = Area of analyte ions monitored.
- A_{ES} = Area of extraction standard ions monitored.
- Q_{ukn} = Concentration of analyte
- Q_{ES} = Concentration of extraction standard

15.2 Relative Response Factor of Extraction/Cleanup Standard

To calculate the RF of an extraction standard in an initial calibration point or a calibration verification:

$$RF_{ES} = \frac{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)}{\left(A_{JS}^{ion1} + A_{JS}^{ion2}\right)} x \frac{Q_{JS}}{Q_{ES}}$$

where:

- A_{ES} = Area of extraction standard ions monitored.
- A_{JS} = Area of injection standard ions monitored.
- Q_{ES} = Concentration of extraction standard.
- Q_{JS} = Concentration of injection standard standard.

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard.

15.3 Average of ICAL RFs for a compound

Calculate the average of the ICAL RFs, as follows:

$$A = \frac{\sum_{i=1}^{n} X}{n}$$

where:

n = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

15.4 ICAL Percent Relative Standard Deviation (%RSD)

First, calculate the standard deviation (s) of the RFs, as follows:

$$s = \sqrt[2]{\frac{\sum_{i=1}^{n} (X - A)}{n-1}}$$

where:

n = number of calibration levels
X_i; i=1 to n, are the compounds RF values for each calibration point
A = average of the RFs from above

Then calculate the %RSD as follows:

$$\%RSD = \frac{s}{A} \times 100$$

where:
s = the standard deviation of the RFs in the curve
A = Average of the RFs in the curve

15.5 Relative Percent Difference

For sample to duplicate comparisons, calculate the Percent Difference (%D) as follows:

$$\%D = \frac{|V_1 - V_2|}{V_1 + V_2 / 2} \times 100$$

where:
V_1 and V_2 = the two values to be compared (i.e. sample and sample dup)

15.6 Percent Difference

For CCAL to ICAL comparisons, calculate the Percent Difference (%D) as follows:

$$\%D = \frac{\exp - acc}{acc} \times 100$$

where:
exp = experimental value (i.e. CCAL)
acc = accepted value (i.e. ICAL)

15.7 Target Compound Concentration Calculations

Calculate the concentration of an analyte in a sample as follows:

$$\left[PCDD/PCDF_{ppt}\right] = \frac{\left(A_{unk}^{ion1} + A_{unk}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{W_{unk} \times \overline{RF}}$$

where:
A_{unk} and A_{ES} = the integrated area for each ion monitored.
Q_{ES} = the amount of extraction standard added to the sample in nanograms
W_{unk} = the initial sample aliquot size, in liters for waters and in grams for solids.
RF = Average RF from the ICAL for the compound

Calculate the adjusted Reporting Limit in a sample as follows:

$$RL_{adj.} = RL_{\exp.} \times \frac{W_{std.}}{W_{actual}} \times \left[\frac{100}{\%S}\right]$$

where:
RL_{exp} = the RL listed in TABLE 1
W_{std} = The standard amount extracted (i.e. 10g for solids, 1000mL for waters.)
W_{actual} = The amount of sample extracted.
[ ] = Needed only to correct for samples reported on a dry weight basis.
%S = Percent solids

Once the 17 2,3,7,8-substituted isomers concentrations have been determined, calculate the Toxicity Equivalency Quotient (TEQ) and its reporting limit $DL_{(TEQ)}$ as follows:

$$TEQ = \sum_{i=1}^{17} [S_i] \times (TEF_i)$$

where:

i = 1 to 17, i represents each one of the 17 2,3,7,8-substituted isomers.

S_i = the concentration of the isomer if detected or the RL if the isomer was not detected. TEQ calculations for USACE clients use the full value of the greater of the EDL/MDL(or check concentration)/EMPC if the isomer was not detected.

TEF_i = Toxic equivalency factor (TEF), each of the 17 2,3,7,8-substituted isomers has an associated TEF which scales its toxicity to 2,3,7,8-TCDD. (Several groups of TEFs exist, SGS will routinely apply the 1998 WHO TEFs for mammals and humans and the 1989 international TEFs.)

$$DL_{(TEQ)} = \sum_{i=1}^{17} [DL(S_i)] \times (TEF_i)$$

where:

i = 1 to 17, i represents each one of the 17 2,3,7,8-substituted isomers.

$DL(S_{i)}$ = the concentration of the RL. $DL_{(TEQ)}$ calculations for USACE clients use the full value of the greater of the EDL/MDL(or check concentration)/EMPC.

15.8 Extraction/Cleanup Standard Amount Recovered Calculation

Calculate the concentration of an extraction standard in a sample as follows:

$$[ES_{ng}] = \frac{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)}{\left(A_{JS}^{ion1} + A_{JS}^{ion2}\right)} \times \frac{Q_{JS}}{\overline{RF}}$$

where:

A_{ES} and A_{JS} = the integrated area for each ion monitored.

Q_{JS} = the amount of injection standard added to the sample in nanograms

RF = Average RF from the ICAL for the compound

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard.

15.9 Percent Recovery

Calculate the percent recovery of an extraction/cleanup standard as follows:

$$\%R = \frac{R_{ng}}{S_{ng}} \times 100$$

where:

R_{ng} = the amount of extraction standard recovered in nanograms.

S_{ng} = the amount of extraction standard spiked in nanograms.

16 Method Performance

To describe the procedures used to achieve, maintain, and prove by documentation a defined analytical quality. The establishment of a system of control charts is applied in this context to track and establish warning and control limits. The objective of these control charts is to reduce errors to required limits. This can help assure that the results have a high probability of being of acceptable quality. Control charts are constructed for OPR recovery. Please see the SOP for Control Charts for more information. Data for the LMB provide for a graphical analysis of laboratory background. LMB values above this background can then be identified and considered for investigation. Many persistent organic pollutants are ubiquitously present and are difficult to completely eliminate. A higher than normal background can result from:

- contamination of container for sampling or during sample storage

- contamination of reagents, supplies, glassware or laboratory equipment
- carryover and cross-contamination

17 Pollution Prevention

17.1 Solvent Spills

- Immediately alert others in the affected area and notify the appropriate supervisor. If necessary, evacuate the area.
- Attend to any person(s) who may be contaminated. Contaminated clothing must be removed immediately and any affected skin flushed with water.
- Don PPE (personal protective equipment) as appropriate to the spill. MSDS's are located in both labs and reference the appropriate gloves and clothing to be used for handling each chemical.
- Within each spill cart are spill socks/booms and spill pillows. Use the socks/booms to contain the spill and prevent further spread of the solvent. Spill pillows can be used to absorb the remaining solvent.
- Used sorbant materials are to be placed in the provided hazardous waste material bags and disposed of accordingly.
- If a volatile solvent is spilled, ventilate the area as much as possible; i.e. open hoods, open rear doors, and if necessary, use portable fans.

17.2 Acid and Caustic Spills

- Attend to any person(s) who may be contaminated. Contaminated clothing must be removed immediately and any affected skin flushed with water.
- Don PPE (personal protective equipment) as appropriate to the spill.
- Contain the spill if required using spill socks or spill pillows.
- With in each spill kit are acid and caustic neutralizer chemicals. Additional acid neutralizer is also stored in the shipping/receiving area.
- Distribute the neutralizers evenly over the spilled liquid. Using the included scoop, work from the outside of the spill towards the center. This technique prevents splashing and any further spread of the liquids.
- When the spilled materials have been absorbed, use the brush and scoop to place materials in an appropriately labeled hazardous waste material bag.
- Neutralized acid and caustic waste can be disposed into the regular trash.
- Decontaminate surfaces involved using mild soap and water, as appropriate.

17.3 Non-Indigenous Soil Spill

- Locate several containers, such as 4 oz. jars, that will be used to package the spill.
- Label the jars with the purple stickers that indicate that the contents are non-indigenous.
- Anything that comes into contact with the soil must then be stored in the waste jars.
- Use paper napkins, aluminum foil, and the like to clean up small spills.
- If a spill warrants the use of a wisk broom and dustpan then they must be labeled with the sticker to ensure proper disposal.

18 Data assessment and Acceptance Criteria

18.1 Target Peak Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

18.1.1 A peak must be at the Correct Retention time as follows:

- For 2,3,7,8-substituted congeners which have an isotopically-labeled extraction standard present in the sample extract, the retention time at maximum peak

height of the sample components must be within -1 to +3 seconds of the isotopically-labeled standard.

- For non-2,3,7,8-substituted compounds, the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

  Note: Samples with high levels of interferences can suffer retention time shifts. The rule for totals peaks still applies but one must ensure that the first and last eluter is still within the MS function.

18.1.2 Monitored ion maximization requirement is as follows:

- The ion current responses for both ions used for quantitative purposes must reach maximum simultaneously (± 2 seconds).

18.1.3 Signal–to–Noise ratio requirements are as follows:

- All ion current intensities must be > 2.5 times noise level for positive identification of an unlabeled PCDD/PCDF compound or a group of coeluting isomers.

18.1.4 Ion ratio requirements are as follows:

- The integrated ion currents for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned.

18.1.5 Polychlorinated diphenyl ether interferences

In addition to the above criteria, the positive identification of a GC peak as a PCDF can only be made if no signal having a S/N > 2.5 is detected at the same retention time (± 2 seconds) in the corresponding polychlorinated diphenyl ether channel.

Note: These peaks are presented with the flag, "DPE" indicating their potential source.

18.2 Data Review

18.2.1 Initial Review

- Summary form generation and organization of Ical, Ccal, Samples and QC data is conducted by the analyst.
- Analysts review data upon generation and elicit peer review.
- Data is then released to a secondary review level with the analyst's signature.

18.2.2 Final Review

- Assemble project samples and QC data
- Review client information and documentation.
- Complete Data Review Checklist (Section 4, Appendix A) and sign reports.
- Samples are released to clients with the data reviewer's signature after the completed package is reviewed by a QA Manager or Lab Director.

18.3 Data Acceptance Criteria

See TABLE 5.

19 Corrective actions for handling out-of-control data

19.1 Requests for Re-extraction

Post-analysis is an early place to discover the need for re-extraction, so it is important to review all supporting data. This includes spike profiles, extraction logs, clean-up logs, injection prep logs, observation forms, and the sample tracking forms in the folder. The project or work group folder may contain exceptions or changes to our routine spiking procedures.

One should also check the sample for problems relating to analysis. These problems include response factors that may introduce quantitative errors, interference that could be diluted out (yellow extracts), or any interference that causes de-tuning or chromatographic conditions that could lead to quantitative errors.

Many things can cause the need for a re-extraction. Extraction or clean-up recovery problems not detailed in the related paperwork indicate the need to talk to the person(s) involved. This type of communication can solve difficult problems and help to ensure that the re-extraction is planned well.

In planning a re-extraction the involvement of a technical reviewer is invaluable. Reviewers notice trends and problems that may call the need for re-extracting a batch of samples. They can give insights as to whether re-extraction will even help or if a different extraction approach should be considered.

Our QA/QC officer should be consulted when re-extraction is considered so that the impact on other samples, or potentially a batch can be assessed.

The analyst will schedule the re-extraction(s) by generating a form indicating the information necessary for the lab to do the re-extraction. Some info includes the sample id, re-extraction due date, and reason for re-extraction (ref. form DC18). The samples are marked "REX" in the LIMS.

When the extractionist receives the form, the samples are scheduled in as other samples are; they can tell that this is a re-extraction by the REX indicator in the report column.

20 Contingencies for handling out-of-control or unacceptable data

When data are produced that are out-of-control or unacceptable, the client is contacted and informed of the situation. A resolution is agreed upon and carried out. This can involve the request for backup sample, re-sampling to acquire more sample, providing the data qualified and narrated to explain the problems encountered. If a corrective action is needed to solve the problem, it is completed before the samples are re-analyzed.

21 Waste Management

Wastes from various processes within the laboratory must be separated based on type (solid, solvent, waste oil, etc.) and then disposed of properly. Non-hazardous items such as solvent-rinsed, air-dried glass vials may be disposed of in the trash. Solvents are stored in drums and disposed of through a waste disposal company. Other solid waste is disposed of through a waste disposal company. Samples suspected of containing dioxins are kept segregated from other waste streams and are disposed of through a hazardous waste disposal company.

Individuals working in the lab should be familiar with the properties and hazards of the materials with which they work. In the event of a chemical spill, it is the responsibility of the individual(s) for prompt and proper clean up of the spill. Before beginning work with

chemicals, all employees should be familiar with the proper techniques for spill clean up, and the appropriate clean-up materials should be readily available.

21.1 Disposal of used laboratory supplies

21.1.1 Prep area waste

21.1.1.1 Paradigm Clean-up Unit (PCU)

- Solvent waste generated during the clean up of samples is collected in a 5 gallon stainless steel receptacle. This carboy is then emptied into the solvent waste drums on a periodic schedule or as sample workload mandates.
- Gloves, pipettes, paper towels and other disposable lab products go in the trash.
- Solvent-rinsed sample vials and collection vials are air dried and disposed of in the trash.
- Used media is collected in a central container and dried in the vacuum hood. When the solvent saturated media is completely dried, it is disposed of in the trash.
- Solvent used for hardware cleaning is dumped into the solvent waste drum.
- Empty solvent bottles are air dried in the vacuum hood and then disposed of in the trash.
- Vacuum pump oil is collected into a dedicated waste oil drum.

21.1.1.2 Soxhlet Dean-Stark

- All waste solvent is collected and then disposed of in the solvent waste drum.
- Sample containing thimbles are collected in a grounded metal trash container. The container is air dried on a periodic basis and dumped in the trash dumpster.

21.1.1.3 Continuous, Soxhlet Dean-Stark (SDS) and Roto-Vap

- All waste solvent is collected and then disposed of in the solvent waste drum.

21.1.1.4 Glassware Cleaning

- All dishwater is poured down the sink. Copious amounts of water are flushed down the sink along with the dishwater.

21.1.1.5 Turbovap

- Solvent saturated paper towels are air-dried in the vacuum hood and then disposed of in the trash dumpster.

Note: Waste Industries, Inc provides all trash and recycling services.

21.1.2 Analytical instrument area waste

21.1.2.1 Solvent Waste

- Nonane waste from the HRGC-HRMS autosamplers is collected and disposed of in the solvent waste drum.

21.1.2.2 Sample extract vials

- All vials are collected and archived. After the holding times have expired, extract vials are ground in the Dioxin Only Solids waste drum.

21.1.2.3 Standard Vials

- All standard vials are opened and the contents allowed to evaporate in the vacuum hood. Dried vials are collected and crushed in the Dioxin Only Solids waste drum.

Note: ECOFLO Inc. provides all waste incineration services.

21.2 Waste Removal

21.2.1 Sample Tracking

All analyzed samples are removed from the primary storage cooler 45 days after the final report has been delivered to the client. This time may vary based on specific client contract requests. Pulled samples are maintained on storage shelves an additional 30 days prior to disposal. Sample ID's are recorded into a waste disposal logbook as samples are physically disposed of into the appropriate 55-gallon drum. A separate waste log is maintained for both solid and aqueous samples.

21.2.2 Solvent Waste

All solvents are collected in specifically labeled 55-gallon drums. Each drum is also labeled with start and end dates.

21.2.3 Solid Waste

All solid samples are ground into 55 gallon drums labeled for dioxin solids only.

21.2.4 Hazardous Waste

All dioxin/furan samples are segregated and disposed of via incineration through a hazardous waste disposal company.

21.2.5 Aqueous Waste

All aqueous samples are disposed of into polypropylene lined 55-gallon drums labeled for dioxin aqueous samples only.

21.2.6 Non-Indigenous Waste

Having been identified at login and labeled with brightly colored "non-standard waste" stickers, non-indigenous solid samples are returned to the client. Non-indigenous aqueous samples are collected into the dioxin aqueous waste drums.

When a sufficient number of drums have accumulated in six months or less, a vendor is contacted for removal and disposal.

22 References

Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. USEPA, October 1994.

Shell for Analytical Chemistry Requirements, U.S. Army Corps of Engineers, EM 200-1-3, Appendix I, 1 Feb 01

23 Appendices and Tables

TABLE 1:

Analyte List with Reporting Limits per Matrix.

Analyte	Solid/Tissues (pg/g)	Aqueous (pg/L)	CAS Number*
2378-TCDD	1	10	1746-01-6
12378-PeCDD	5	50	40321-76-4
123478-HxCDD	5	50	39227-28-6
123678-HxCDD	5	50	57653-85-7
123789-HxCDD	5	50	19408-74-3
1234678-HpCDD	5	50	35822-39-4
OCDD	10	100	3268-87-9
2378-TCDF	1	10	51207-31-9
12378-PeCDF	5	50	57117-41-6
23478-PeCDF	5	50	57117-31-4
123478-HxCDF	5	50	70648-26-9
123678-HxCDF	5	50	57117-44-9
234678-HxCDF	5	50	60851-34-5
123789-HxCDF	5	50	72918-21-9
1234678-HpCDF	5	50	67562-39-4
1234789-HpCDF	5	50	55673-89-7
OCDF	10	100	39001-02-0

* = Chemical Abstract Service Registry Number

TABLE 2:

Theoretical Ion Abundance Ratios and Their Control Limits

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6[a]	0.51	0.43	0.59
7	1.04	0.88	1.20
7[b]	0.44	0.37	0.51
8	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF
[b] Used only for ^{13}C-HpCDF

TABLE 3:

Initial Calibration Concentrations

Analyte	Concentration (pg/μL) CS-1	CS-2	CS-3	CS-4	CS-5
Unlabeled					
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
Extraction Standards					
^{13}C-2378-TCDD	100	100	100	100	100
^{13}C-2378-TCDF	100	100	100	100	100
^{13}C-12378-PeCDD	100	100	100	100	100
^{13}C-12378-PeCDF	100	100	100	100	100
^{13}C-23478-PeCDF	100	100	100	100	100
^{13}C-123678-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-123678-HxCDF	100	100	100	100	100
^{13}C-123789-HxCDF	100	100	100	100	100
^{13}C-234678-HxCDF	100	100	100	100	100
^{13}C-1234678-HpCDD	100	100	100	100	100
^{13}C-1234678-HpCDF	100	100	100	100	100
^{13}C-1234789-HpCDF	100	100	100	100	100
^{13}C-OCDD	200	200	200	200	200
Cleanup Standards					
^{37}Cl-2378-TCDD	0.25	2	10	40	200
Injection Standards					
^{13}C-1234-TCDD	100	100	100	100	100
^{13}C-123789-HxCDD	100	100	100	100	100

TABLE 4:

Mass Descriptors used for Selected Ion Recording HRMS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	50	10
1	2	305.8987	50	5
1	3	315.9419	50	5
1	4	304.9824	50	5
1	5	304.9824	(Lock)	5
1	6	317.9389	50	5
1	7	319.8965	50	5
1	8	321.8936	50	5
1	9	327.8847	50	5
1	10	331.9368	50	5
1	11	333.9339	50	5
1	12	375.8364	50	5
2	1	339.8597	50	10
2	2	341.8568	50	5
2	3	351.9	50	5
2	4	353.897	50	5
2	5	355.8546	50	5
2	6	357.8517	50	5
2	7	366.9792	50	5
2	8	366.9792	(Lock)	5
2	9	367.8949	50	5
2	10	369.8919	50	5
2	11	409.7974	50	5
3	1	373.8207	50	10
3	2	375.8178	50	5
3	3	380.976	50	5
3	4	380.976	(Lock)	5
3	5	383.8639	50	5
3	6	385.861	50	5
3	7	389.8156	50	5
3	8	391.8127	50	5
3	9	401.8559	50	5
3	10	403.853	50	5
3	11	445.7555	50	5
4	1	407.7818	50	10
4	2	409.7788	50	5
4	3	417.8253	50	5
4	4	419.822	50	5
4	5	423.7767	50	5
4	6	425.7737	50	5
4	7	430.9728	50	5
4	8	430.9728	(Lock)	5
4	9	435.8169	50	5
4	10	437.814	50	5
4	11	479.7165	50	5
5	1	441.7427	50	10
5	2	443.7398	50	5
5	3	454.9728	50	5
5	4	454.9728	(Lock)	5
5	5	457.7377	50	5
5	6	459.7348	50	5
5	7	469.778	50	5
5	8	471.775	50	5
5	9	513.6775	50	5

TABLE 5:

SGS's Quality Control Requirements for Method 1613			
QC Check	**Frequency**	**Limits**	**Corrective Action**
Method Blank	One per extraction batch.	PCDD/F< SOP RL or <10% of level in sample. (PCDD/F< ½ LMCL for Dod)	Affected samples must be re-extracted.
Initial Calibration (ICAL)	As needed to maintain acceptable CCAL, after major maintenance, or at a minimum one per year.	20% RPD for native and 35% RPD for labeled species.	An acceptable ICAL must be established and verified with an independent source (CCAL) before sample reporting may begin.
Mass Resolution	Before and after each 12 hour analytical sequence.	Mass resolution must be at 10,000 as estimated from a printout of a PFK peak.	MS maintenance.
Descriptor Defining Isomers	At the beginning of each 12h analytical sequence.	First and last eluters must be present within the switching times.	Perform a survey scan to identify the correct switching times.
GC Performance Isomers	At the beginning of each 12h analytical sequence.	Must be <25% valley.	GC maintenance.
OPR (LCS)	One per extraction batch.	See Table 9	Re-extract batch if low and detected. Evaluate data quality for high and non-detected.
OPRD (LCSD)	One per extraction batch.	See Table 9, ≤ 20%RSD.	If needed and fails, re-extract batch.
Extraction Standards (ES)	Every sample must receive ES before extraction.	See Table 8.	Evaluate data quality, and if needed re-extract sample.
Continuing Calibration (CCAL)	At the beginning of sample analysis every 12h.	See Table 7.	Acceptable CCAL must be established before sample analysis may begin.
Lab Sample Duplicate (DUP)	Per client request.	<20%Difference.	Flag failures.
Matrix spike/duplicate (MS/MSD)	One pair per method, per matrix, per extraction technique, per 30 days, per 20 samples.	±25% recovery, 20% RPD.	Flag failures, report OPRD.

TABLE 6:
Cleanup Flowchart

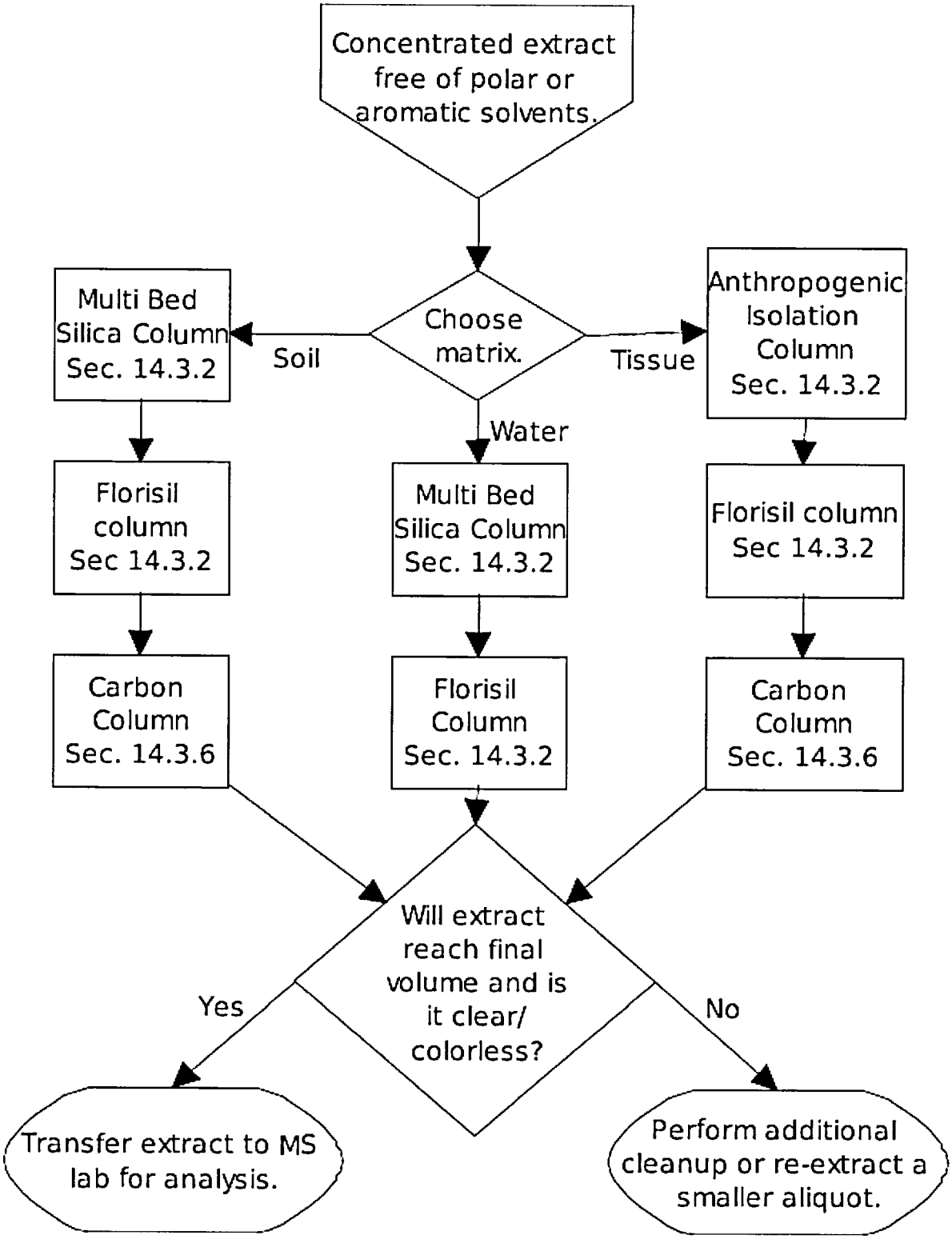

TABLE 7:

Continuing Calibration Limits					
Compound Name	**CCAL (pg/µL)**	**Limits (pg/µL)**	**Compound Name**	**CCAL (pg/µL)**	**Limits (pg/µL)**
2,3,7,8-TCDD	10	7.8 - 12.9	$^{13}C_{12}$-2,3,7,8-TCDD	100	82 - 121
1,2,3,7,8-PeCDD	50	39 - 65	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	62 - 160
1,2,3,4,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	85 - 117
1,2,3,6,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	85 - 118
1,2,3,7,8,9-HxCDD	50	41 - 61	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	72 - 138
1,2,3,4,6,7,8-HpCDD	50	43 - 58	$^{13}C_{12}$-OCDD	200	96 - 415
OCDD	100	79 - 126	$^{13}C_{12}$-2,3,7,8-TCDF	100	71 - 140
2,3,7,8-TCDF	10	8.4 - 12	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	76 - 130
1,2,3,7,8-PeCDF	50	41 - 60	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	77 - 130
2,3,4,7,8-PeCDF	50	41 - 61	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	76 - 131
1,2,3,4,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	70 - 143
1,2,3,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	74 - 135
2,3,4,6,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	73 - 137
1,2,3,7,8,9-HxCDF	50	44 - 57	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	78 - 129
1,2,3,4,6,7,8-HpCDF	50	45 - 55	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	77 - 129
1,2,3,4,7,8,9-HpCDF	50	43 - 58	$^{37}Cl_4$-2,3,7,8-TCDD	10	7.9 - 12.7
OCDF	100	63 - 159			

TABLE 8:

Labeled Standard Recovery Limits for Samples and Blanks		
Compound Name	**Amount Spiked (pg/µL)**	**Limits %**
$^{13}C_{12}$-2,3,7,8-TCDD	100	25 - 164
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25 - 181
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32 - 141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28 - 130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23 - 140
$^{13}C_{12}$-OCDD	200	17 - 157
$^{13}C_{12}$-2,3,7,8-TCDF	100	24 - 169
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24 - 185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21 - 178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26 - 152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26 - 123
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	29 - 147
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	28 - 136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28 - 143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26 - 138
$^{37}Cl_4$-2,3,7,8-TCDD	10	35 - 197

TABLE 9:

OPR Recovery Limits		
Analyte	**Amount Spiked (pg/µL)**	**Limit (pg/µL)**
2378-TCDD	10	6.7-15.8
12378-PeCDD	50	35-71
123478-HxCDD	50	35-82
123678-HxCDD	50	38-67
123789-HxCDD	50	32-81
1234678-HpCDD	50	35-70
OCDD	100	78-144
2378-TCDF	10	7.5-15.8
12378-PeCDF	50	40-67
23478-PeCDF	50	34-80
123478-HxCDF	50	36-67
123678-HxCDF	50	42-65
123789-HxCDF	50	39-65
234678-HxCDF	50	35-78
1234678-HpCDF	50	41-61
1234789-HpCDF	50	39-69
OCDF	100	63-170
^{13}C-2378-TCDD	100	20-175
^{13}C-12378-PeCDD	100	21-227
^{13}C-123478-HxCDD	100	21-193
^{13}C-123678-HxCDD	100	25-163
^{13}C-1234678-HpCDD	100	26-166
^{13}C-OCDD	200	26-397
^{13}C-2378-TCDF	100	22-152
^{13}C-12378-PeCDF	100	21-192
^{13}C-23478-PeCDF	100	13-328
^{13}C-123478-HxCDF	100	19-202
^{13}C-123678-HxCDF	100	21-159
^{13}C-123789-HxCDF	100	17-205
^{13}C-234678-HxCDF	100	22-176
^{13}C-1234678-HpCDF	100	21-158
^{13}C-1234789-HpCDF	100	20-186
^{37}Cl-2378-TCDD	10	3.1-19.1

Table 10. Relative Retention Times

Compound	Retention Time Reference	Relative Retention Time
2,3,7,8-TCDD	13C12-2,3,7,8-TCDD	0.999-1.002
1,2,3,7,8-PeCDD	13C12-1,2,3,7,8-PeCDD	0.999-1.002
1,2,3,4,7,8-HxCDD	13C12-1,2,3,4,7,8-HxCDD	0.999-1.001
1,2,3,6,7,8-HxCDD	13C12-1,2,3,6,7,8-HxCDD	0.998-1.004
1,2,3,7,8,9-HxCDD	13C12-1,2,3,6,7,8-HxCDD	1.000-1.019
1,2,3,4,6,7,8-HpCDD	13C12-1,2,3,4,6,7,8-HpCDD	0.999-1.001
OCDD	13C12-OCDD	0.999-1.001
2,3,7,8-TCDF	13C12-2,3,7,8-TCDF	0.999-1.003
1,2,3,7,8-PeCDF	13C12-1,2,3,7,8-PeCDF	0.999-1.002
2,3,4,7,8-PeCDF	13C12-2,3,4,7,8-PeCDF	0.999-1.002
1,2,3,4,7,8-HxCDF	13C12-1,2,3,4,7,8-HxCDF	0.999-1.001
1,2,3,6,7,8-HxCDF	13C12-1,2,3,6,7,8-HxCDF	0.997-1.005
2,3,4,6,7,8-HxCDF	13C12-2,3,4,6,7,8-HxCDF	0.999-1.001
1,2,3,7,8,9-HxCDF	13C12-1,2,3,7,8,9-HxCDF	0.999-1.001
1,2,3,4,6,7,8-HpCDF	13C12-1,2,3,4,6,7,8-HpCDF	0.999-1.001
1,2,3,4,7,8,9-HpCDF	13C12-1,2,3,4,7,8,9-HpCDF	0.999-1.001
OCDF	13C12-OCDD	0.999-1.008
13C12-2,3,7,8-TCDD	13C12-1,2,3,4-TCDD	0.976-1.043
13C12-1,2,3,7,8-PeCDD	13C12-1,2,3,4-TCDD	1.000-1.567
13C12-1,2,3,4,7,8-HxCDD	13C12-1,2,3,7,8,9-HxCDD	0.977-1.000
13C12-1,2,3,6,7,8-HxCDD	13C12-1,2,3,7,8,9-HxCDD	0.981-1.003
13C12-1,2,3,4,6,7,8-HpCDD	13C12-1,2,3,7,8,9-HxCDD	1.086-1.110
13C12-OCDD	13C12-1,2,3,7,8,9-HxCDD	1.032-1.311
13C12-2,3,7,8-TCDF	13C12-1,2,3,4-TCDD	0.923-1.103
13C12-1,2,3,7,8-PeCDF	13C12-1,2,3,4-TCDD	1.000-1.425
13C12-2,3,4,7,8-PeCDF	13C12-1,2,3,4-TCDD	1.011-1.526
13C12-1,2,3,4,7,8-HxCDF	13C12-1,2,3,7,8,9-HxCDD	0.944-0.970
13C12-1,2,3,6,7,8-HxCDF	13C12-1,2,3,7,8,9-HxCDD	0.949-0.975
13C12-2,3,4,6,7,8-HxCDF	13C12-1,2,3,7,8,9-HxCDD	0.959-1.021
13C12-1,2,3,7,8,9-HxCDF	13C12-1,2,3,7,8,9-HxCDD	0.97-1.047
13C12-1,2,3,4,6,7,8-HpCDF	13C12-1,2,3,7,8,9-HxCDD	1.043-1.085
13C12-1,2,3,4,7,8,9-HpCDF	13C12-1,2,3,7,8,9-HxCDD	1.057-1.151
37Cl4-2,3,7,8-TCDD	13C12-1,2,3,4-TCDD	0.989-1.052

Standard Operating Procedures
For the Login and Storage of Samples

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SGS Environmental Services
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Revised By:

Greg Dickinson — 12-23-09
Greg Dickinson — Date

Name, Technical Director:

Greg Dickinson — 12-23-09
Greg Dickinson — Date

Name, QA Manager:

Jeannie Milholland — 12-23-09
Jeannie Milholland — Date

(Official copies of final documents will contain all three signatures.)

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1 Identification Of Test Method

This SOP applies to all methods used in this lab and describes procedures used to record sample receipt and requested analysis information.

2 Applicable Matrix or Matrices

Please refer to the individual test methods for applicable matrices. SGS does not accept human blood or tissue.

3 Scope and Application

All samples received at SGS are logged into the Laboratory Information Management System (LIMS). The LIMS is used to direct the laboratory to perform requested testing within method holding times or due dates.

4 Detection Limits for Components to be Analyzed

Please refer to the individual test methods for detection limit information.

5 Summary of the Test Method

Samples arrive in the receiving area, usually packed on ice in coolers. The coolers are inspected for damage then opened. The sample temperature is checked with a thermometer and recorded on the Chain of Custody (COC). Sample identifications on the bottle labels are compared to the chain of custody. Tests requested are compared to bottles received. Preserved samples are pH tested to verify preservation. Sample information is entered into the LIMS and each sample assigned unique laboratory identification. The bottles are labeled with the identifier and stored. A record of the log-in is printed and placed in a folder with all documentation. The log-in is then reviewed by a project manager. Once folder review is complete, the samples are ready for the lab. Their status in the LIMS is updated to allow the lab to begin work. The folders are stored in bins in the data reporting section of the laboratory.

6 Definitions

LIMS: Laboratory Information Management System.
See Document DC139 Appendix D for a full list of definitions.

7 Interferences

- Minimize time that sample containers are out of refrigeration. Every effort is made to keep samples chilled.
- Place samples in the correct refrigerator. Volatile samples, semi-volatile samples, Ultratrace samples, and metal samples are stored in different locations.
- Storage blanks are prepared and analyzed on a prescribed basis to ensure that cross-contamination does not occur.

8 Safety

- Safety glasses, gloves, and laboratory coat are required when working in sample management.
- Carefully check for any broken glass when opening a cooler. Place any cooler with broken glass in a fume hood or outside as a sample of unknown content may be open.

Transfer the damaged sample to a new, clean container if practical. Contact project management to decide appropriate action.
- Wear Kevlar gloves to remove contents if any broken glass is observed.
- Place cooler in a fume hood or outside if any odors are detected.

9 Equipment and Supplies

- Digital thermometer
- PH strips
- Free chlorine strips
- Syringe
- Sample bottles and vials
- Paper towels
- Office supplies
- Sample receipt checklist (SRC)

10 Reagents and Standards

- Deionized water
- Preserving solutions
  - HCl
  - HNO_3
  - H_2SO_4
  - Sodium Thiosulfate, 99.99^{+}%, powder.

11 Sample Collection, Preservation, Shipment, and Storage

Sample size, container type, and preservative for each parameter are tabulated in section 23.

12 Quality Control

- The digital thermometer requires quarterly calibration against a NIST traceable thermometer.
- Samples must arrive in the laboratory on ice at 4±2°C or on ice with a downward temperature trend for local samples that have been collected the day of receipt.
- Any discrepancies must be noted on the sample receipt checklist (SRC).
- Project management must review all projects after log-in and before placing the samples into the laboratory. All discrepancies are resolved with the client by a project manager and noted in the project folder.
- Storage blanks must meet method blank requirements for each method analyzed.

13 Calibration and Standardization

13.1 Digital thermometer Calibration

- The Digital Thermometer calibration is checked at ice point temperature (0.1 °C), a temperature close to sample refrigerator temperature(2-6 °C), and room temperature (20 °C).
- Place the NIST reference thermometer in a vessel of water and allow to equilibrate at the measurement temperature.
- Take 10 replicate measurements each of the digital against the NIST.
- The replicate data collected must have an RSD of less than or equal to 5% for each level checked. If the RSD fails for any one type of container then a corrective

action is required and the test repeated for all containers successfully before proceeding.

- Average the results of each container type and calculate the offsets. NOTE:(where the NIST value does not change over the course of the measurement sequence, use the overall average, otherwise, would have to calculate offsets for each measurement and average.)
- The maximum allowable offset is ±1°C.
- Record this data and label the thermometer with the applied offsets.
- The thermometers are calibrated once per quarter.

14 Procedures

14.1 Login and Storage

14.1.1 Equipment

- Digital thermometer.

14.1.2 Supplies

- Folders, folder labels, and sample labels.

14.1.3 Dioxin Procedure

- Upon delivery to the lab all containers will be checked for damage that may have affected the integrity of their contents.
- Containers, usually coolers, should be opened and contents examined for damage.
- Measure the temperature as described in Section 14.2 and record on the chain-of-custody (COC). Sign and date/time the COC.
- Remove and examine contents. Verify that all information on the sample bottle labels match the COC.
- For 1613, 8290, and 8280 water samples: Check for residual (free) chlorine using test strips. Samples with results equal to or above 0.5 mg/L (ppm) will require the addition of 80mg of sodium thiosufate per liter. If residual chlorine is present, note the improper preservation on the SRC.
- Inspect the paperwork and samples for a USDA Plant Protection and Quarantine (PPQ) form 550. These labels can be further recognized by the words "Soil Samples Restricted Entry". Please note on the SRC that the soils are non-indigenous. These samples require special labeling.
- If air samples are present in the shipment, contact a member of management for a verification of sample identification. (Method 23, 29, etc.) See Section 14.6 regarding air sample Login.
- pH will be measured and adjusted prior to extraction if needed.
- Fill out the Sample Receipt Checklist (SRC) and note any discrepancies.

Enter information from COC into the LIMS

- Upon entry of client's identification number, the LIMS will automatically assign a sequential project ID that is unique to the client.
- The LIMS automatically assigns a five digit laboratory ID.
- Client provided information such as sample ID and date of collection are entered from the COC.

Enter information specific to the LIMS. (See Table 2 for more information.)

- QC level
- Report Format
- Analysis
- Location
- Due date
- Any discrepancies are entered into the SRC.

Sample labeling (for more information, see section 14.4)

- Labels containing the lab ID, project ID, and storage location are printed from the LIMS.
- Larger containers such as 4 oz. soil jars and 1L amber jars receive one label on the bottle and a second label on the cap.
- Water resistant adhesive labels are used.
- All samples are logged-in and labeled prior to beginning work on the samples.

Sample storage

- After labeling, the samples are stored in the appropriate refrigerator or freezer.
- Waters and soils are stored in refrigerator W2.
- Tissues are stored in freezer F1 and F2.
- Samples known to contain high levels of target analytes will be segregated from other samples whenever possible.

Project folder preparation

- Label a folder with the lab ID
- List analysis required on the front of the folder.
- Place all documents received with the samples in the folder.
- Place the shipping label in the folder
- Place the SRC in the folder.
- Folder is reviewed by project management.

14.1.4 Soil & Water Procedure

See SGS's Sample Acceptance Policy, below.
Upon delivery, the cooler is checked for custody seal, opened, checked for damage, and condition of sample containers checked.

- If broken containers are observed or any odor is detected from the cooler, place the cooler in a fume hood or outside.
- Wear glass resistant Kevlar gloves if any broken glass is observed.

Sample temperature is checked with the following priorities.

- Read trip thermometer if present.
- Take temperature with digital thermometer from temperature blank if present.
- Take temperature of the cooler interior if a temperature blank is not provided.

Record temperature on the COC, sign and date with time received.
Remove and examine sample contents.

- Compare bottle labels to the COC entries. Note discrepancies on the SRC.
- Compare volumes and preservative types to analyses requested.
- Note discrepancies on the SRC.
- Verify sample is within holding time for requested analysis.
- If air samples are present in the shipment, contact a member of management for a verification of sample identification. (Method 23, 29, etc.) See Section 14.6 regarding air sample login.

Check pH preserved samples.
- Use short range pH strips.
- Use a clean disposable pipette to transfer a drop of sample to pH paper. Do not immerse paper into the sample.
- Check color change and note if sample not properly preserved.
- If preservative is added then record amount and lot# on the SRC.

Enter information from COC into the LIMS
- Upon entry of client's identification number, the LIMS will automatically assign a sequential project ID that is unique to the client.
- The LIMS automatically assigns a five digit laboratory ID.
- Client provided information such as sample ID and date of collection are entered from the COC.

Enter information specific to the LIMS. (See Table 2 for more information.)
- QC level
- Report Format
- Analysis
- Location
- Due date
- Any discrepancies are entered into the SRC.

Sample labeling (for more information, see section 14.4)
- Labels containing the lab ID, COC #, project ID, Client ID, Analysis, Matrix, Sample date and time, Disposal date and storage location are printed from the LIMS.
- All plastic bottles and VOC vials receive one label.
- Larger containers such as 4 oz. soil jars and 1L amber jars receive one label on the bottle and a second label on the cap.
- Water resistant adhesive labels are used.
- All samples are logged-in and labeled prior to beginning work on the samples.

Check for non-indigenous samples.
- Inspect the paperwork and samples for a USDA Plant Protection and Quarantine (PPQ) form 550. These labels can be further recognized by the words "Soil Samples Restricted Entry". Please note on the SRC that the soils are non-indigenous. These samples require special labeling; please see the sample labeling SOP for further instruction.

Project folder preparation
- Label a folder with the lab ID
- List analysis required on the front of the folder.
- Place all documents received with the samples in the folder.
- Place the shipping label in the folder
- Place the SRC in the folder.
- Folder is reviewed by project management.
- Project manager assigns a due date to the folder.
- Folder is placed in appropriate file in data review area.

14.2 Temperature Measurement of Sample Containers

14.2.1 Equipment

- Digital thermometer

14.2.2 Procedure

14.2.2.1 Use of the Digital thermometer

- Verify that the quarterly calibration is valid. If not use the procedure in section 13.
- Turn on the unit and wait for a stable reading.
- Never take a temperature from inside a sample bottle or jar.

14.2.2.2 Priority of temperature procedures

- Read the trip thermometer if present.
- Take temperature from temp blank (if present).
- Take temperature from cooler using the digital thermometer.

14.3 Temperature Monitoring of Storage Units

14.3.1 Equipment/Supplies

- Walk-in coolers
- Freezers
- Refrigerators
- Logbook

14.3.2 Procedure

- All units contain thermometers that are traceable to NIST for accuracy.
- Each morning, before heavy use of these units, the temperature is recorded in the logbook.
- Refrigerators must be maintained at 4±2°C.
- Freezers must be kept between -10 and -20°C. Thermostats are set at -20°C.
- For units containing DoD samples, temperature will be monitored every day, including weekends and holidays.
- Whenever these limits are not met, the failure is noted in the logbook. An attempt to correct the thermostat is made. A repeated failure warrants moving the samples to another unit and placing a service call to a vendor to repair the unit.

14.4 Sample Labeling

Sample labeling consists of applying water/organic solvent proof labels on the glass wall of the sample container (e.g., vials, centrifuge or other fraction-collection tube) and the bottle and bottle's cap for incoming samples. The labels must be durable and utilize indelible ink. The label contains information such as the SGS project/sample number and the clients sample identification. The SGS Project number is automatically generated by the LIMS system. All required sample containers will be pre-labeled at the start of any step.

14.4.1 Procedure

- Upon entry of the client's name into the LIMS, a new project number will be issued by the LIMS. This number is sequential and specific to the project (G100-1).
- Each individual sample container is assigned a unique laboratory sample identification number automatically by the LIMS (G100-1-3C).
- Samples identified as non-indigenous require an additional label. These large purple labels help ensure that the samples are unmistakably identified as needing special disposal procedures. These procedures are outlined in the disposal SOP.

14.4.2 Printing

- Once the information is entered into the LIMS (log-in is complete), a series of labels are printed which will be used to label the in-coming sample bottle and cap.
- Additional labels will be printed at the time of sample extraction for use throughout the remainder of the laboratory processes. The labels must be waterproof and of appropriate size to accommodate the various sample containers. Additionally, the use of highlighted labels for QC samples such as the OPR is recommended.

14.5 Internal Chain of Custody

- Samples requiring an Internal Chain of Custody (ICOC) as specified per client will have ICOCs generated at the time of folder review. Each ICOC shall list the sample ID, number of containers received, and storage location as well as any pertinent information from the submitted COC.
- To begin the chain the login personnel will sign the ICOC. The samples, along with their respective ICOCs, will be released to a designated person, the sample custodian, from the extraction department.
- Each time the sample or its resulting extract is removed from storage, the ICOC is signed and dated. The analyst will note which container is removed and for what purpose. Only the sample custodian and the GC/MS analyst shall have access to the controlled extract storage.
- After sixty days (or longer if negotiated by the client) the sample is designated for disposal and the ICOC is transferred to waste stream tracking. The extract is maintained in extract storage for one year. The ICOC records are filed with the data package.

14.6 Air Sample Login

Careful attention and an extra layer of management review at the bench level is given to air samples because of their irreplaceable nature. Air samples consist of multi matrix, multi fraction components that can require a varied combination of sample fractions as required by the client.
Management and log-in personnel review paperwork, COC, SOW, task order's, etc. pertaining to the project. They identify and organize all sample fractions before log-in begins. Once the samples are logged in and labeled, a secondary review of the sample labels by management takes place. Any discrepancies are corrected.
Common examples of air project login are given below. Keep in mind that the practice of ordering the field fractions that are to make up an analytical fraction is key. If any other procedure is required, detail them in project notes. Then verbally inform the project manager and prep personnel.

14.6.1 Method 23 Example

Field fractions to be combined are listed in order before an analytical test fraction is entered in to the LIMS. For example, if the front and back halves are to be analyzed for dioxins separately, the login should proceed as follows:

-01 Prep of filter
-02 Prep of solvent rinse of probe
-03 Dioxin (SW8290) analysis front half

-04 Prep of trap
-05 Prep of back half solvent rinses
-06 Prep of impinger contents

-07 Prep of impinger rinses
-08 Dioxin (SW8290) analysis of back half

14.7 Method 29 Example

Field fractions to be combined are listed in order before an analytical test fraction is entered in to the LIMS. For example, if the Hg only impingers are to be analyzed separately, the login should proceed as follows:

-01 Prep of filter (Field Container 1)
-02 Prep of acetone rinse (Field Container 2)
-03 Prep of acid probe rinse (Field Container 3)
-04 Analysis - Metals on front half
-05 Analysis - Hg on front half

-06 Prep of back half impingers (Field Container 4)
-07 Analysis - Metals on back half
-08 Analysis - Hg on back half

-09 Prep of pre-Hg empty impinger (Field Container 5A)
-10 Analysis - Hg on pre-Hg empty impinger

-11 Prep of $KMnO_4$ impingers (Field Container 5B)
-12 Analysis - Hg on $KMnO_4$ impinger

-13 Prep of $KMnO_4$ impinger HCl rinse (Field Container 5C)
-14 Analysis - Hg on $KMnO_4$ impinger rinse

This example does not include the prep and analysis of field rinse blanks.

14.8 Storage blanks

14.8.1 Equipment/Supplies

- Walk-in cooler/refrigerator
- Sample bottles/vials
- Sample labels

14.8.2 Procedure

- Storage blanks are prepared and analyzed at a prescribed frequency.
    - Dioxins/PCBs: bi-weekly
    - Volatiles: bi-weekly
- The sample is "collected" into the appropriate sample container, using an analyte-free matrix such as deionized water or sodium sulfate.
- The sample is logged into the LIMS for the appropriate analytical test, and labeled as such.
- The sample is stored in the appropriate refrigerator/freezer for two weeks.
- The sample is prepared and analyzed following the standard operating procedures used for all laboratory samples.
- Each storage blank must meet its method's LMB requirements, or corrective action is required.

15 Calculations

Not applicable

16 Method Performance

Please refer to the individual test methods for method performance information.

17 Pollution Prevention

Please refer to the individual test methods for pollution prevention procedures.

18 Data assessment and Acceptance Criteria

18.1 Sample Acceptance Policy

Samples received by the laboratory must meet certain criteria in order for the associated analytical data to meet method criteria. Samples must be properly documented and shipped in appropriate containers at specified temperatures. Holding times must be met and the amount of sample provided must be sufficient for the required extraction procedures.

18.1.1 Procedure

The validity and defensibility of analytical data is highly dependent upon proper delivery of samples to the laboratory. It may be impossible for the laboratory to perform the required analyses if samples are not properly documented and shipped in a timely fashion using appropriate containers and shipping materials. The following criteria are used to evaluate the suitability of samples prior to their acceptance for analysis:

a) Documentation: All samples must be accompanied by chain-of-custody forms indicating unique sample identification, date and time of sampling, location of collection, collector's name, any preservatives used, sample type (e.g. soil, water, etc.), requested analyses and any special remarks concerning the samples.
b) Chain-of-custody forms must accompany the samples and cannot be sent separately.
c) Sample Labeling: Each sample must be given a unique identifier that allows for cross-referencing with the chain-of-custody information. Sample labels must be durable enough to remain attached to the containers and utilize indelible ink to remain legible during shipping.
d) Sample Containers: Samples must be shipped in containers appropriate for the required analyses. Liquid and solid samples collected for organics analyses should be shipped in glass containers. Other matrices may require special containers (e.g. air samples). Any questions regarding sample containers must be resolved prior to collecting the samples. Containers should be packaged in such a way as to preclude breakage or leaking during shipping.
e) Damage: Samples should be inspected for signs of damage. *Discrepancy Procedure: Damaged containers must be safely repackaged and quarantined. Record the damage found and action taken. Report container damage to client services.*
f) Contamination: The shipment should be inspected for signs of contamination. Discrepancy Procedure: Safely separate the samples and

remove any further means of contamination. Record the condition of the samples and action taken. Report potential contamination to client services.

g) Preservation: It is generally required that samples be chilled to < 4° ±2° C and kept at that temperature during shipping. Therefore, samples should be placed in insulated coolers with ice to keep them at or near this temperature until delivery at the laboratory. Samples that arrive above 6°C and are not on ice will be rejected and the client will be contacted for further instructions, such as re-sampling if for compliance monitoring. *Discrepancy Procedure: Inadequate preservation must be reported to client services.*

h) Low Concentration Soil Sample for Volatiles Analysis: Soil samples that contain carbonate minerals may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small, any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that contain only de-ionized water. Any effervescence must be noted by field personnel. This sampling procedure must be documented on the COC due to the storage requirements.

i) Holding Times: In order to provide accurate, defensible data, analytical methods strictly limit the amount of time that may elapse between collection and extraction of samples. Data taken from the analysis of samples that have exceeded the required holding times is generally considered invalid and is not legally defensible. Therefore, it is imperative that samples be sent to the laboratory as quickly as possible after collection. Ideally, the samples will be sent via overnight courier on the same day as collection. When this is not possible, the samples must arrive at the laboratory with enough time left to allow for proper login procedures before the holding time elapses. Samples received with few days left in the holding time may not be accepted by the laboratory.

j) Sample Volume: Sufficient sample volume must be sent for all required tests. Each method specifies the required sample volume for each applicable matrix. Questions regarding required sample volumes must be resolved prior to sampling.

k) Client Notification: When sample login is completed, a summary of the samples received and entered into the LIMS is generated. This can be faxed to the client upon request. This fax will include client sample ids, lab ids, dates, analyses requested and status summaries in short form.

The laboratory will attempt to contact the client and resolve any issues with sample acceptance. Samples that are immediately rejected are ones with improper preservation, damage, or show signs of contamination. In other cases, it may be possible to continue with analysis, although the analysis may be compromised. For instance, samples received with insufficient volume may be analyzed with the resulting data reflecting increased detection limits due to the insufficient volume, and minor discrepancies in sample documentation may be easily resolved. In such cases, the Technical Director/Project Manager will contact the client and describe the problem and any potential effects on the data. The client may then determine if the resulting data is likely

to meet their needs. In other instances (e.g. samples received without labels or in broken containers), it may be impossible for the lab to proceed with analysis. In all cases, the Technical Director/Project Manager will make the final decision regarding the laboratory's acceptance of samples.

19 Corrective actions for handling out-of-control data

Not Applicable.

20 Contingencies for handling out-of-control or unacceptable data

Not Applicable.

21 Waste Management

Not Applicable.

22 References

EPA's National Environmental Laboratory Accreditation Conference Standards – 2002, Chapter Five.

23 Appendices and Tables

TABLE 1: Container, Preservation and Holding Time Requirements.

Analysis	*Matrix*	*Container*	*Preservative*	*Hold Time*
Alkalinity	Water	125mL plastic	------	14 days
BOD	Water	1000mL plastic	------	48 hours
COD	Water	125mL plastic	H2SO4	28 days
Chloride	Water	125mL plastic	------	28 days
Cyanide	Water	500mL plastic	NaOH	14 days
Analysis	***Matrix***	***Container***	***Preservative***	***Hold Time***
CO2	Water	500mL plastic	------	24 hours
Fluoride	Water	500mL plastic	------	28 days
Hardness	Water	125mL plastic	HNO3	6 months
MBAS	Water	500mL plastic	------	48 hours
Ammonia Nitrogen	Water	500mL plastic	H2SO4	28 days
TKN	Water	500mL plastic	H2SO4	28 days
Nitrate	Water	125mL plastic	------	48 hours
Nitrite	Water	125mL plastic	------	48 hours
Total Nitrogen NO2+NO3-N	Water	125mL plastic	H2SO4	28 days
Oil & Grease	Water	1 Liter Amber	H2SO4	28 days
TRPH by 418.1	Water	1 Liter Amber	HCL	14 days
PH	Water	125mL plastic	------	Immediate
Phenol	Water	1 Liter Amber	H2SO4	28 days
Ortho Phosphorus	Water	125mL plastic	------	48 hours
Phosphorus	Water	125mL plastic	H2SO4	28 days
Specific Conductivity	Water	125mL plastic	------	28 days

Sulfate	Water	125mL plastic	------	28 days
Sulfide	Water	125mL plastic	NaOH/ZnAc	7 days
TOC	Water	250 mL Amber	HCl	28 days
TOX	Water	1 Liter Amber	H2SO4	28 days
TSS	Water	500mL plastic	------	14 days
TDS	Water	500mL plastic	------	14 days
Turbidity	Water	125mL plastic	------	48 hours
Metals	Water	125mL plastic	HNO3	6 months
Metals + Hg	Water	500mL plastic	HNO3	Hg-28 days
Cr +6	Water	500mL plastic	------	24 hours
Ferrous Fe	Water	500mL plastic	------	6 months
Bacteria	Water	125mL plastic	Sodium Thiosulfate	6 h or 24 h

Volatiles 601, 602, 6210D, 6230D, 8010, 8020, 8021, 8260 502.2, 524.2, 624	Water	3 40ml VOA vials	HCl	14 days unpres-7days

Semi Volatiles 8270, 625, 610, 8310	Water	1 Liter Amber	------	ex-7days/40d

8080, 8081, 8082	Water	1 Liter Amber	------	ex-7days/40d
8150, 8151		1 Liter Amber	------	ex-7days/40d

Gasoline, 5030	Water	2 40ml VOA vials	HCL	14 days
Diesel, 3510	Water	1 Liter Amber	HCL	ex-7days/40d
Varsol	Water	1 Liter Amber	HCL	ex-7days/40d
EPH	Water	1 Liter Amber	HCL	ex-14days/40d
Analysis	***Matrix***	***Container***	***Preservative***	***Hold Time***
VPH	Water	2 40ml VOA vials	HCL	14 days

EDB, 504.1	Water	3 40ml VOA vial	HCL	14 days
Ethylene Glycol	Water	2 40ml VOA vial	------	
Formaldehyde	Water	1 Liter Amber	------	ex-72 hr

8260/5035	Soil	Encores; 3 5g		ex-48 hr
8260/5035	Soil	2 40ml VOA vial; 1 40ml VOA vial	Sodium Bisulfate; MEOH	14 days
VPH	Soil	Encores; 2 5g	------	ex-48 hr
VPH	Soil	2 40ml VOA vial	MEOH	14 days
5035/8015 GRO	Soil	2 40ml VOA vial	MEOH	14 days

Table 2: Sample Containers, Preservation, and Hold Times for Dioxin/Furan & PCB Analyses

Method	Matrix	Extraction Holding Times	Analysis Holding Times	Container[1]	Preservation
8280	Aqueous/Solid	30 days	45 Days from Collection	Amber Glass Bottle/Jar	4°C[2]
8290	Aqueous/Solid Tissue	30 Days	45 Days from Collection	AGB/AGJ	4°C (T: -20C)[2], Dark
1613	Aqueous/Solid Tissue	1 Year	45 Days from Extraction	AGB/AGJ	A: 0-4°C[2] S/T:<-10°C
613	Aqueous	7 Days	40 Days from Extraction	AGB	4°C, Dark
23	MM5 Train	30 Days	45 Days from Extraction	Train/AGB	0-4°C; Dark
TO9	PUF	7 Days	40 Days from Extraction	Aluminum Foil	<4°C
1668	Aqueous/Solid	1 Year	1 Year from Extraction	AGB/AGJ	A/S: 0-4°C[2] T:<-10°C
DLM02.0 CBC01.0	Aqueous/Solid/ Tissue	10 Days VTSR[3]	30 Days from Extraction	AGB/AGJ	A/S: 4±2°C[2] T:<-10°C

[1]Minimum Sample Amount: Aqueous, 1L; Solid, 100g; Tissue, 100g.
[2]If residual chlorine is present in aqueous samples, add 80 mg sodium thiosulfate per liter of water.
[3]Tissue holding time is 24 hours from thawing.

TABLE 3: Storage Locations

Prep	Analysis	Loc
EX	504.1	R7
	601	R7
	602	R7
EX	608	W1
	624	R7
EX	625	W1
EX	1613	W2
EX	1668	W2
	6210D	R7
	6230D	R7
	8010	R7

Prep	Analysis	Loc
	8020	R7
	8021	R7
EX	8081	W1
EX	8082	W1
EX	8141	W1
EX	8151	W1
	8260	R7
EX	8270	W1
EX	8280	W2
EX	8290	W2
EX	8310	W1
EX	8315	W1
EX	5030	W1
EX	3510	W1
EX	3550	W1
EX	1664A	Sub
EX	9071	Sub
EX	EPH	W1
	VPH-0	R7
	VPH-2	R5
	VPH-3	R5
	Metals	L1
	Mercury (Hg)	L1
	% Solids	W1
	pH	L1
	Chloride	L1
	Nitrate	L1
	Nitrite	Sub
	Sulfate	L1

APPENDIX D

EXCERPTS FROM THE ROUNDS 1 THROUGH 10 INTERIM OFFSHORE MONITORING PROGRAM REPORT FOR OPERABLE UNIT 4, PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE, FEBRUARY 2010

TABLE 1-2

MODIFICATIONS TO THE INTERIM OFFSHORE ROUNDS 1 THROUGH 10 INTERIM OFFSHORE MONITORING PROGRAM REPORT PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE

Report	Round	Modification	Rationale/Comments	Modification Implemented
Baseline Interim Offshore Monitoring Report For Operable Unit 4 (TtNUS, July 2002)	Round 1-4	Discontinue analysis of sediment samples for AVS/SEM.	AVS/SEM data were not needed for future evaluations.	Round 6
		Discontinue juvenile lobster sampling.	Chemical concentrations in juvenile lobsters were similar between monitoring and reference stations. Mussels are a better indicator of contaminant concentrations.	
		Conduct future sampling rounds in late summer.	No significant differences in data collected during spring or summer. Round 5 samples were collected in summer before report was approved.	
Rounds 1 through 7 Interim Offshore Monitoring Program Report (TtNUS, November 2004)	Round 1-7	Collect sediment samples only at MS-05, MS-08, MS-09, MS-10, MS-13, and MS-14 during Rounds 8 and 9.	These locations had increasing concentrations that exceed IRGs or are predicted to exceed the IRGs within the next 5 years.	Round 8
		Conduct additional scrutiny only at MS-01, MS-03, MS-04, MS-05, MS-08, MS-09, MS-11, and MS-12. PAHs and metals were identified as target analytes.	Locations exceed IRGs and are predicted to continue to exceed the IRGs within the next 5 years.	
		Discontinued mussels collection until the next 5-year sampling event.	BSAFs/BAFs are not expected to change. Area is closed to shellfishing, so human health risks do not need to be re-evaluated.	
		Discontinue analysis of sediment and mussel samples for alkylated PAHs.	Alkylated PAHs have not been identified as COCs and are not used to make recommendations for additional sampling.	
		Perform dioxin/furan analysis only at MS-08, MS-09, and the reference stations for Rounds 8 and 9, but perform dioxin/furan analysis for MS-07 through MS-12 and the reference stations during Round 10.	Risks from dioxins/furans to human and ecological receptors are low. Samples will be collected in future rounds due to increase in dioxin/furans concentrations during Round 7.	
		Conduct further evaluation of 4,4'-DDT as part of the additional scrutiny for MS-01 and MS-08. (Additional scrutiny for 4,4'-DDT was not conducted at MS-08 because the sediment with the elevated 4,4'-DDT detection was excavated as part of the OU3 remedial activities in 2004.)	Elevated 4,4'-DDT level at MS-01.	

TABLE 1-2

MODIFICATIONS TO THE INTERIM OFFSHORE
ROUNDS 1 THROUGH 10 INTERIM OFFSHORE MONITORING PROGRAM REPORT
PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE

Report	Round	Modification	Rationale/Comments	Modification Implemented
Additional Scrutiny Quality Assurance Project Plan for Operable Unit 4, Appendix B: Technical Memorandum Comparison of Analytical Methods (TtNUS, August 2005)	Round 1-7	Use USEPA analytical methods for organic chemical analysis of sediment as opposed to NOAA methods.	Similar results by both methods. Greater number of laboratories capable of analyzing using USEPA methods, which results in cost saving.	Round 8 / Additional Scrutiny
		Use USEPA analytical methods for organic chemical and metal analyses of mussel samples as opposed to NOAA methods.	Similar results expected by both methods. Greater number of laboratory capable of analyzing using USEPA methods results in cost saving.	
		Remove select pesticides (chloropyrifos, pentachloroanisole, pentachlorobenzene, 1,2,3,4-tetrachlorobenzene, and 1,2,4,5-tetrachlorobenzene) from analytical parameter list.	At least two different analytical methods needed using USEPA methods for all parameters. The eliminated pesticides were not identified as COCs, do not have IRGs, and are not used in decision-making.	
Phase II Additional Scrutiny Quality Assurance Project Plan, Appendix A (TtNUS, September 2007)	Round 8 / Additional Scrutiny	Move sample locations at MS-05 and MS-09.	Obtain better spatial coverage of chemical concentrations in sediment.	Round 9 / Phase II Additional Scrutiny
		Collect sediment at two additional locations at MS-05 for analysis of copper and lead for Round 9.	Ensure that size of area near MS-05, Loc. 1 with elevated concentrations of metals is not increasing.	
		Eliminate sampling at reference stations (Navy agreed to collect sediment samples at one reference station for Round 9 after receiving comments from the regulators).	No increasing or decreasing trends in concentrations noted.	
		Discontinue monitoring at MS-10, MS-13, and MS-14.	Data do not indicate any impacts from IRP sites, and concentrations of PAHs are generally low and do not appear to be increasing.	

TABLE 1-2

MODIFICATIONS TO THE INTERIM OFFSHORE
ROUNDS 1 THROUGH 10 INTERIM OFFSHORE MONITORING PROGRAM REPORT
PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE

Report	Round	Modification	Rationale/Comments	Modification Implemented
Technical Memorandum for Round 10 (TtNUS, September 2008)	Prior to Round 10	Eliminate sampling at reference stations.	Overall, chemical concentrations at reference stations were consistent across all rounds at a particular station, with no increasing or decreasing trends in concentrations.	Round 10[1]
			The reference data are not currently being used for decision making because chemical concentrations are less than IRGs.	
			The data set is adequate for making decisions after Round 10.	
		Only collect sediment samples from MS-05, MS-08, and MS-09 during Round 10.	Sediment samples not needed at other monitoring stations for various reasons as discussed in the Technical Memorandum.	
		Discontinue use of NOAA methods for analysis of metals in sediment. Use USEPA Method 6020B for analysis of metals in future offshore sampling events. During an October 8, 2008, conference call, it was suggested that the Navy collect additional sediment samples from MS-05, MS-08, and MS-09 during Round 10 for analysis of metals by NOAA and USEPA methods to strengthen the data set.	Greater number of laboratories capable of analyzing using USEPA methods, which results in cost saving.	
		Discontinue mussel sampling at all monitoring and reference stations.	Bioaccumulation factors do not need to be recalculated.	

1 - Some of these modifications were recommended for future rounds, but based on regulator comments, it was agreed that the modifications would be applied to Round 10 only.
AVS - Acid volatile sulfides
BAF - Biota accumulation factor
BSAF - Biota sediment accumulation factor
COCs - Chemicals of concern
IRGs - Interim remediation goals
IRP - Installation Restoration Program
NOAA - National Oceanic and Atmospheric Administration
PAHs - Polycyclic aromatic hydrocarbons
SEM - Simimultaneously extracted metals
USEPA - United States Environmental Protection Agency

TABLE 6-1

SUMMARY OF RECOMMENDATIONS FOR INTERIM OFFSHORE MONITORING ROUNDS 1 THROUGH 10 INTERIM OFFSHORE MONITORING PROGRAM REPORT PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE

AOC/Monitoring Activity	Monitoring/Reference Station	IRP Site/OU	Recommendation
Back Channel	MS-01	Site 34 (OU9)	• Conduct interim offshore monitoring during five-year sampling events (next scheduled for 2013) until a final remedy for OU4 is implemented. • Analyze samples for PAHs.
	MS-02	None	• Discontinue interim offshore monitoring.
	MS-03 and MS-04	Site 32 (OU7)	• Conduct interim offshore monitoring during five-year sampling events (next scheduled for 2013) until a final remedy for OU4 is implemented. • Analyze samples for PAHs and copper.
Jamaica Cove	MS-05	OU3	• Conduct interim offshore monitoring bi-annually (every 2 years) and at five-year sampling events until a final remedy for OU4 is implemented. • Analyze bi-annual samples for copper, lead, and nickel. • Analyze five-year samples for PAHs and metals.
	MS-06		• Discontinue interim offshore monitoring.
Clark Cove	MS-07	OU3	• Conduct interim offshore monitoring during five-year sampling events (next scheduled for 2013) until a final remedy for OU4 is implemented. • Analyze samples for PAHs, 4,4'-DDT, dioxins/furans, PCBs, and metals.
	MS-08 and MS-09		• Conduct interim offshore monitoring bi-annually (every 2 years) and at five-year sampling events until a final remedy for OU4 is implemented. • Analyze bi-annual samples for PAHs, 4,4'-DDT, copper, lead, nickel, and dioxins/furans. • Analyze five-year samples for PAHs, 4,4'-DDT, metals, PCBs, and dioxins/furans.
Sullivan Point	MS-10	None	• Discontinue interim offshore monitoring.
DRMO Storage Yard	MS-11	Sites 6 and 29 (OU2)	• Conduct interim offshore monitoring during five-year sampling events (next scheduled for 2013) until a final remedy for OU4 is implemented. • Analyze samples for copper, lead, and nickel.

TABLE 6-1

SUMMARY OF RECOMMENDATIONS FOR INTERIM OFFSHORE MONITORING
ROUNDS 1 THROUGH 10 INTERIM OFFSHORE MONITORING PROGRAM REPORT
PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE

AOC/Monitoring Activity	Monitoring/Reference Station	IRP Site/OU	Recommendation
Dry Docks	MS-12	Sites 5 and 10	• Conduct interim offshore monitoring during five-year sampling events (next scheduled for 2013) until a final remedy for OU4 is implemented. o Analyze samples for PAHs and lead. • Collect additional sediment samples near AS12-SD12 bi-annually (every 2 years) and at five-year sampling events until a final remedy for OU4 is implemented. o Analyze samples for lead.
	MS-13 and MS-14	Sites 5 and 31	• Discontinue interim offshore monitoring.
None	All reference stations	None	• Do not collect sediment samples as part of the OU4 interim offshore monitoring program.
Analytical Methods for Metals	None	None	• Analyze future sediment samples using standard USEPA analytical methods (USEPA Method 6010B/6020 for all metals except mercury (and USEPA Method 7471A for mercury). • Use regression equations to compare results from USEPA and NOAA analytical methods and to convert IRGs for copper and nickel, when necessary.

AOC – Area of Concern
IRG – Interim Remediation Goal
IRP – Installation Restoration Program
NOAA – National Oceanic and Atmospheric Administration
OU – Operable Unit
PAHs – Polycyclic aromatic hydrocarbons
USEPA – United States Environmental Protection Agency